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COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP APÓS DESGASTE COM BROCA DIAMANTADA E DEGRADAÇÃO HIDROTÉRMICA

Santa Maria, RS
2016

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Dissertação de mestrado apresentada ao Curso de Mestrado do Programa de Pós-graduação em Ciências Odontológicas, Área de Concentração em Odontologia, Ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do título de **Mestre em Ciências Odontológicas**.

Orientador: Prof. Dr. Luiz Felipe Valandro

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Luís Felipe Guilardi

COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP APÓS DESGASTE COM BROCA DIAMANTADA E DIFERENTES ESTRATÉGIAS DE ENVELHECIMENTO

Dissertação de mestrado apresentada ao Curso de Mestrado do Programa de Pós-graduação em Ciências Odontológicas, Área de Concentração em Odontologia, Ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do título de **Mestre em Ciências Odontológicas**.

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Santa Maria, RS
2016

DEDICO ESTE TRABALHO

Com muito carinho e amor,

aos meus pais Paulo e Marialva e aos meus irmãos Paulo Henrique e Bernardo pelo apoio constante,

aos familiares, amigos e colegas por fazerem parte da minha caminhada.

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RESUMO

COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP APÓS DESGASTE COM BROCA DIAMANTADA E DEGRADAÇÃO HIDROTÉRMICA

AUTOR: Luís Felipe Guilardi
ORIENTADOR: Luiz Felipe Valandro

Este estudo avaliou o efeito do desgaste com brocas diamantadas de diferentes granulações (Grossa – G e Extrafina – FF), associado ou não ao envelhecimento em autoclave (LTD) na micromorfologia superficial, na transformação de fase (tetragonal para monoclínica, *t→m*), e nas propriedades mecânicas de uma zircônia policristalina parcialmente estabilizada por óxido de ítrio. Foram confeccionados 180 discos (15,0 Ø x 1,2 mm de espessura) conforme a ISO 6872-2008, que foram divididos em 6 grupos (n=30) de acordo com dois fatores em estudo: desgaste da superfície (controle C - sem desgaste; desgaste com broca G ou FF); e envelhecimento em autoclave (C^{LTD}, G^{LTD} FF^{LTD}); Os desgastes foram executados por um operador treinado sob refrigeração constante; e a LTD em autoclave a 134ºC, sob 2 bar de pressão, por 20 horas. Os resultados indicam que o envelhecimento em autoclave associado ou não ao desgaste não foram prejudiciais às propriedades mecânicas da zircônia. Entretanto cabe ressaltar que estes estímulos promoveram grande aumento de fase monoclínica, o que pode ser um indício de degradação do material. A análise de Weibull (Módulo de Weibull) demonstrou que os tratamentos avaliados (desgaste, envelhecimento em autoclave) não promoveram efeitos deletérios, não resultando em maior variabilidade da resistência mecânica. Logo, não há indícios de que os diferentes tratamentos realizados impactem negativamente na resistência à flexão biaxial da Y-TZP. Adicionalmente, nota-se que as superfícies desgastadas com brocas diamantadas têm uma menor susceptibilidade à LTD (menor aumento de fase *m*).

Palavras-chave: Desgaste. Tratamento de Superfície. Degradação Hidrotérmica. Análise de Weibull. Cerâmica Y-TZP. Zircônia Policristalina Parcialmente Estabilizada por Óxido de Ítrio.

ABSTRACT

MECHANICAL BEHAVIOR OF A Y-TZP CERAMIC AFTER GRINDING WITH DIAMOND BUR AND HYDROTHERMAL DEGRADATION

AUTHOR: Luís Felipe Guilardi
ADVISER: Luiz Felipe Valandro

This study aimed to determine the effects of grinding and low temperature aging on the surface micromorphology (roughness measurement, Scanning Electron Microscopy and Atomic Force Microscopy), phase transformation (t→m), biaxial flexural strength and structural reliability (Weibull analysis) of an yttrium-stabilized tetragonal zirconia polycrystalline ceramic. One hundred and eighty ceramic discs (15.0 × 1.2 ± 0.2 mm, VITA In-Ceram YZ) were prepared and randomly assigned into six groups according to 2 factors ($n = 30$): ‘grinding’ (Ctrl – without treatment, as-sintered; Xfine – grinding with extra fine diamond bur - 30 μm; Coarse – grinding by coarse diamond bur - 151 μm), and ‘aging’ (without or with aging: CtrlLTD; XfineLTD; CoarseLTD). Grinding was performed in an oscillatory motion with a contra-angle handpiece under constant water-cooling. Low temperature degradation (LTD) was simulated in an autoclave at 134 °C, under 2 bar pressure, for 20 h. The roughness (Ra and Rz parameters) significantly increased after grinding in accordance with bur grit-size (Coarse > Xfine > Ctrl), and aging promoted distinct effects (Ctrl = CtrlLTD; Xfine > XfineLTD; Coarse = CoarseLTD). Grinding increased the m-phase, and aging led to an increase in the m-phase in all groups. However, different susceptibilities to LTD were observed. Weibull analysis showed a significant increase in the characteristic strength after grinding (Coarse = Xfine > Ctrl), while aging did not lead to any deleterious impact. Neither grinding nor aging resulted in any deleterious impact (no statistical decrease in the Weibull moduli) on material reliability. Thus, neither grinding nor aging led to a deleterious effect on the mechanical properties of the evaluated Y-TZP ceramic although a high m-phase content and roughness were observed.

Keywords: Grinding, Hydrothermal degradation, Y-TZP Ceramics, Dental materials, Surface treatments, Yttrium-stabilized zirconium oxide

SUMÁRIO

1. INTRODUÇÃO

Diante da crescente demanda por materiais mais resistentes e estéticos para emprego em prótese parcial fixa, a zircônia policristalina tetragonal parcialmente estabilizada por óxido de ítrio (Y-TZP) têm merecido destaque em decorrência de sua boa estabilidade química e dimensional, resistência mecânica e tenacidade superiores, além de módulo de Young na mesma ordem de grandeza das ligas de aço inoxidável (CAMPOSILVAN; FLAMANT; ANGLADA, 2015; CHEVALIER; GREMILLARD; DEVILLE, 2007; CURTIS; WRIGHT; FLEMING, 2006; BAN et al., 2008; BLATZ; SADAN; KERN, 2003; DENRY; KELLY, 2008; PICONI; MACCAURO, 1999; VAGKOPOULOU, 2009).

A zircônia é um material polimórfico que apresenta três formas cristalográficas, as quais mudam de acordo com a temperatura em que o material se encontra e em pressão atmosférica ambiente: em temperatura ambiente até 1170°C a fase é monoclínica (*m*), entre 1170°C e 2370°C a estrutura assume a forma tetragonal (*t*), e acima de 2370°C até seu ponto de fusão (2680°C) a forma estável é a cúbica (*c*) (PICONI; MACCAURO, 1999).

A transformação da fase *t* para *m* durante o resfriamento é acompanhada por um aumento discreto em seu volume (3-5%), sendo o suficiente para causar uma falha catastrófica (HANNINK; KELLY; MUDDLE, 2000; PICONI; MACCAURO, 1999). Por este motivo óxidos “estabilizadores ou dopantes” (CaO, MgO, CeO_2, Y_2O_3) são adicionados a sua composição, permitindo que a conformação tetragonal se mantenha estável em temperatura ambiente. Dentre estes, o óxido de ítrio (Y_2O_3) têm sido a principal opção de escolha nas áreas biomédicas (PICONI; MACCAURO, 1999).

Apesar da adição de óxidos estabilizadores, a zircônia continua apresentando metaestabilidade, e desta forma, permanece susceptível à transformação de fase (t→m). Essa mudança de fase é desencadeada quando este material é submetido à aplicação de diferentes estímulos: mecânicos (i.e. desgaste, polimento, jateamento), físicos (i.e. ciclagem mecânica), e químicos (i.e. ação de água e temperatura – denominado degradação hidrotérmica ou degradação em baixas temperaturas (LTD), variação de pH). (CHEVALIER; GREMILLARD; DEVILLE, 2007; COTES et al., 2014; EGILMEZ et al., 2014; INOKOSHI et al., 2015; KOBAYASHI; KUWAJIMA; MASAKI, 1981; LUCAS et al., 2015; PAPANAGIOTOU et al., 2006; SATO; SHIMADA 1985; TURP et al., 2012).

A metaestabilidade apresentada pelas cerâmicas Y-TZP é desejável pois o aumento de volume superficial causado pela mudança t→m leva à concentração de tensões compressivas na superfície da cerâmica, o que atua promovendo o fechamento de defeitos superficiais (trincas e micro-trincas), ou seja, atua dificultando a propagação de trincas. Este mecanismo foi denominado mecanismo de tenacificação por transformação (*transformation toughening mechanism*) e é considerado o responsável pelas elevadas resistência e tenacidade à fratura das cerâmicas Y-TZP (HANNINK; KELLY; MUDDLE, 2000).

Desta forma, existem alguns fatores determinantes para que a Y-TZP seja suscetível à transformação (metaestável): tamanho do grão cristalino (quanto maior o grão, maior a susceptibilidade à transformação); densidade do material (menor densidade leva a maior facilidade de ação da umidade e desta forma maior susceptibilidade à transformação) e a quantidade de óxido estabilizador (quanto maior a quantidade do dopante, mais estável o material, e consequentemente menor a resposta através do mecanismo de tenacificação) (CHEVALIER; GREMILLARD; DEVILLE, 2007; LI; WATANABE, 1998). O percentual de óxido de ítrio deve ser fracionado adequadamente para evitar a transformação (t→m) durante o resfriamento no ciclo de sinterização mas, ao mesmo tempo, permitir a metaestabilidade da fase tetragonal. Uma boa combinação entre alta resistência à flexão (maior que 1000 MPa) e moderada tenacidade à fratura (6-10 MPa.m1/2) pode ser alcançada com a adição de 3 mol% de óxido de ítrio (3Y-TZP). (BAN et al., 2008; CAMPOSILVAN; FLAMANT; ANGLADA, 2015; CHEVALIER; GREMILLARD; DEVILLE, 2007; CURTIS; WRIGHT; FLEMING, 2006; KAMADA; YOSHIDA; ATSUTA, 1998)

Entretanto, apesar de a Y-TZP ter se mostrado primeiramente um material com propriedades excelentes, a comunidade científica se tornou cautelosa em relação ao seu uso devido ao episódio Prozyr em 2001, onde milhares de próteses de quadril feitas com Y-TZP falharam prematuramente devido à sua alta susceptibilidade à degradação causada por uma mudança no processo de sinterização do material que o deixou mais propenso ao envelhecimento (transformação da fase t para m). (CHEVALIER; GREMILLARD; DEVILLE, 2007).

Kobayashi e colaboradores, 1981, demonstraram que quando a zircônia é exposta ao longo do tempo em ambientes úmidos e em baixas temperaturas (150° – 400 °C) esta sofre um processo espontâneo de transformação de fase t→m denominado *low-temperature degradation* (LTD). (KOBAYASHI; KUWAJIMA; MASAKI, 1981) Esse processo é utilizado para simular o envelhecimento da zircônia Y-TZP de forma acelerada. Em condições orais (próteses dentárias) e corporais (próteses de quadril) às quais a zircônia Y-TZP está exposta, a transformação de fase ocorre muito lentamente, mas se houver mudança nos componentes que controlam a metaestabilidade da fase tetragonal (tamanho do grão, quantidade de estabilizador e processo de sinterização) essa transformação pode acontecer de maneira acelerada levando à falha precoce do material (i.e. episódio Prozyr 2001). (CHEVALIER; GREMILLARD; DEVILLE, 2007).

Ainda não existe um consenso sobre o mecanismo de desenvolvimento da LTD. Independente de qual seja esse mecanismo, está bem estabelecido que a transformação (t→m) inicia nos grãos superficiais e depois segue para o interior da amostra, provocando uma alteração topográfica da superfície, micro trincas e perda de grãos com subsequente aumento da rugosidade do material, isso possibilita a penetração da água na sub superfície propagando a transformação para o interior do material; levando ao desenvolvimento de trincas maiores. (LUGHI; SERGO, 2010) A consequência desse mecanismo é a diminuição da resistência, tenacidade e densidade das estruturas de Y-TZP (BAN et al., 2008; HIRANO, 1992; LUGHI; SERGO, 2010). Na presença de calor e umidade este processo é acelerado, e uma das formas mais extensivamente descritas de simular os efeitos do envelhecimento de cerâmicas Y-TZP em um ambiente *in vitro*, é o envelhecimento em autoclave (CHEVALIER; GREMILLARD; DEVILLE, 2007; PEREIRA et al., 2015).

De acordo com Chevalier (2007) em temperatura ambiente e em condições do corpo humano (37 °C e umidade), a LTD ocorre muito devagar atingindo apenas uma camada superficial de poucos micrômetros. No entanto, clinicamente as restaurações estão expostas a diferentes estímulos (mastigação, ação da água e baixas temperaturas, biofilme, mudança no pH) e por isso constitui um ambiente ideal para que a LTD ocorra. Isso torna relevante a associação de diferentes métodos de envelhecimento *in vitro* que busquem simular essas condições para tentarmos entender melhor o comportamento da Y-TZP quando submetida ao ambiente oral a longo prazo. (CHEVALIER; GREMILLARD; DEVILLE, 2007).

Porém, os estudos têm avaliado a LTD apenas através de fatores isolados: armazenamento em água destilada, armazenamento em soluções ácidas, ciclagem em autoclave, ciclagem mecânica, armazenamento em câmaras de vapor d’água. (CHEVALIER; GREMILLARD; DEVILLE, 2007; COTES et al., 2014; EGILMEZ et al., 2014; INOKOSHI et al., 2015; LUCAS et al., 2015; TURP et al., 2012). Até o presente momento, apenas Cotes et al (2014) avaliaram a LTD através da associação de diferentes estímulos (autoclave + ciclagem mecânica) associados. (COTES et al., 2014).

Além disso, embora o uso do sistema CAD/CAM reduza as necessidades de ajuste clínico e laboratorial da cerâmica devido a sua precisão (ABOUSHELIB; FEILZER; KLEVERLAAN, 2009), frequentemente são necessários ajustes/desgastes com pontas diamantadas na região da superfície interna e/ou externa dessas restaurações, visando um melhor ajuste da peça protética (assentamento no preparo protético, ajuste oclusal, do pôntico e conectores, etc.) (ABOUSHELIB; FEILZER; KLEVERLAAN, 2009; CHEVALIER; GREMILLARD; DEVILLE, 2007; GUAZZATO et al., 2005; KOSMAC et al., 1999).

Este procedimento é capaz de desencadear a transformação de fase t→m, mas ao mesmo tempo ele pode introduzir defeitos superficiais. Se estes defeitos introduzidos forem maiores que a camada de compressão criada, eles podem gerar zonas de concentração de tensão o que aumentaria o risco de uma fratura catastrófica precoce da peça. (GUAZZATO et al., 2005; KOSMAC et al., 1999) Mas quando esse defeito fica confinado dentro da camada compressiva, a literatura tem demonstrado que essa transformação superficial é benéfica e resulta no aumento da resistência do material através do mecanismo de tenacificação. (KAMADA; YOSHIDA; ATSUTA, 1998).

Entretanto, até o presente momento existem poucos estudos na literatura que avaliem o comportamento mecânico, susceptibilidade à LTD e transformação de fase de cerâmicas Y-TZP, após desgaste com brocas diamantadas. Adicionalmente existem escassos estudos na literatura que avaliem o comportamento da Y-TZP frente a diferentes métodos de envelhecimento que simulem os estímulos aos quais este material será submetido diariamente no ambiente oral, principalmente levando em consideração situações de associação de estímulos.

Desta forma, esta dissertação de mestrado tem como objetivo avaliar o efeito do desgaste com brocas diamantadas e envelhecimento em autoclave no comportamento mecânico, topografia superficial, estabilidade estrutural (transformação de fase) e confiabilidade estrutural (análise de Weibull) de uma cerâmica Y-TZP.

Para efeitos de apresentação, esta Dissertação está apresentada na forma de um artigo científico, o qual será submetido à publicação no periódico *Journal of the Mechanical Behavior of Biomedical Materials*. ISSN: 1751-6161, Fator de Impacto: 2.876, Qualis: A2. As normas para publicação estão descritas no ANEXO A.

ARTIGO

SURFACE MICRO-MORPHOLOGY, PHASE TRANSFORMATION, AND MECHANICAL RELIABILITY OF GROUND AND AGED MONOLITHIC ZIRCONIA CERAMICS.

2. ARTIGO - Surface micro-morphology, phase transformation, and mechanical reliability of ground and aged monolithic zirconia ceramics.

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Running title: Mechanical behavior after grinding and aging of a Y-TZP ceramic.

ABSTRACT

This study aimed to determine the effects of grinding and low temperature aging on the surface micromorphology (roughness measurement, Scanning Electron Microscopy and Atomic Force Microscopy), phase transformation (t→m), biaxial flexural strength and structural reliability (Weibull analysis) of an yttrium-stabilized tetragonal zirconia polycrystalline ceramic. One hundred and eighty ceramic discs (15.0 × 1.2 ± 0.2 mm, VITA In-Ceram YZ) were prepared and randomly assigned into six groups according to 2 factors ($n = 30$): 'grinding' (Ctrl – without treatment, as-sintered; Xfine – grinding with extra fine diamond bur - 30 µm; Coarse – grinding by coarse diamond bur - 151 µm), and 'aging' (without or with aging: Ctrl^{LTD}; $\text{Xfine}^{\text{LTD}}$; $\text{Coarse}^{\text{LTD}}$). Grinding was performed in an oscillatory motion with a contra-angle handpiece under constant water-cooling. Low temperature degradation (LTD) was simulated in an autoclave at 134 °C, under 2 bar pressure, for 20 h. The roughness (R_a and R_z parameters) significantly increased after grinding in accordance with bur grit-size (Coarse > Xfine > Ctrl), and aging promoted distinct effects ($\text{Ctrl} = \text{Ctrl}^{\text{LTD}}$; $\text{Xfine} > \text{Xfine}^{\text{LTD}}$; $\text{Coarse} = \text{Coarse}^{\text{LTD}}$). Grinding increased the m-phase, and aging led to an increase in the m-phase in all groups. However, different susceptibilities to LTD were observed. Weibull analysis showed a significant increase in the characteristic strength after grinding (Coarse = Xfine > Ctrl), while aging did not lead to any deleterious impact. Neither grinding nor aging resulted in any deleterious impact (no statistical decrease in the Weibull moduli) on material reliability. Thus, neither grinding nor aging led to a deleterious effect on the mechanical properties of the evaluated Y-TZP ceramic although a high m-phase content and roughness were observed.

Keywords: Grinding, Hydrothermal degradation, Y-TZP Ceramics, Dental materials, Surface treatments, Yttrium-stabilized zirconium oxide.

2.1. INTRODUCTION

Over recent years, the use of Y-TZP (yttrium-stabilized tetragonal zirconia polycrystal) for restorative dentistry has increased exponentially because of its excellent mechanical properties and enhanced optical properties (Zhang, 2014). The superior properties demonstrated by these materials have also led to an expansion of their applications, e.g., Y-TZP is now considered as an alternative for manufacturing single-unit monolithic full-contour restorations (Beuer et al., 2012; Nakamura et al 2015; Sabrah et al., 2013). Monolithic Y-TZP is a viable substitute for the common procedure of manufacturing frameworks in single- or multi-unit fixed dental prosthesis (FDPs) that are later veneered with glass porcelain (Denry and Kelly, 2014).

Y-TZP is a polycrystalline, metastable material. When submitted to mechanical (e.g., grinding, polishing, sandblasting), physical (e.g., oral mastication forces, mechanical cycling in the laboratory), and chemical (e.g., exposure to water, temperature changes, pH change) stimuli, or even when exposed to an oral biofilm, a transformation mechanism can be triggered in Y-TZP from the tetragonal (t) to the monoclinic phase (m) (Bordin et al., 2015; Cotes et al., 2014; Chevalier et al., 2007; Egilmez et al., 2014; Inokoshi et al., 2015; Lucas et al., 2015; Turp et al., 2012).

This mechanism leads to a volume increase (~3–4%) around superficial grains resulting in compression stress concentration around any existing superficial defects (cracks and micro cracks) and consequently hindering crack propagation. This protective mechanism is known as transformation toughening and is considered responsible for the superior mechanical properties of Y-TZP (Hannink et al., 2000; Piconi and Maccauro, 1999). The increase in the m-phase (after it reaches a critical value) may lead to an increased roughness, decreased density, and a potentially deleterious effect that is known as *low-temperature degradation,* on the mechanical properties (Kobayashi et al., 1981).

Pereira et al. (2016[a]) recently undertook a systematic review of the effects of grinding on Y-TZP ceramics. For these materials, the parameters used during grinding (e.g., the grinding tool, presence of cooling, speed, grit-size, control of movement) define the final mechanical and surface characteristic properties, as has been previously suggested (Iseri et al., 2010; Jing et al., 2014; Yin et al., 2003). Based on this knowledge, it was expected that changing the direction of the grinding movement would also be a major factor in defining the final mechanical behavior of this ceramic, directly impacting the introduction of superficial defects.

From a clinical perspective, although there have been significant advances in the precision of CAD/CAM (Computer Aided Design / Computer Aided Machining) milling systems, a final adjustment of the prostheses is still necessary to improve adaptation, occlusion, and to ensure an adequate emergence profile (Aboushelib et al., 2009; Iseri et al., 2012; Pereira et al., 2015[b]; Pereira et al., 2016[b]; Pereira et al., 2016[c]; Preis et al., 2015). In addition, in many scenarios, the clinician has to perform these adjustments directly in the mouth. This restricts the use of instruments and control of grinding movements, usually leading to a more hazardous procedure.

Another major aspect of the mechanical behavior of Y-TZP (as stated by Chevalier et al., 2007) is that susceptibility of the material to the t-m phase transformation depends on density, stabilizer content, grain size, processing characteristics (e.g., homogeneity, manufacturing procedure, and preparation) and the presence of residual stresses. The response of the Y-TZP when submitted to stimuli is therefore material dependent (i.e., any change in the material characteristics could lead to a distinct response).

To the authors' knowledge, only two studies (Michida et al., 2015; Subasi et al., 2014) have evaluated the effect of grinding on VITA In-Ceram YZ ceramics. These studies noted a significant decrease in strength after grinding. It should be highlighted that these authors did not consider different grit-sizes (the first used a fine grit-size of 30 µm, and the second used a coarse grit-size of 110 µm). There were also other differences in the grinding protocols (presence/absence of cooling, the pressure, and the movement of the grinding tool). Recently, Pereira et al. (2015[b]; 2016[c]) evaluated the mechanical behavior of two different brands of Y-TZP ceramics using a highly standardized, stringent protocol for grinding. They concluded that the use of a high-torque multiplier contra-angle, coupled with a low speed motor with constant cooling, did not affect the mechanical properties of the materials.

VITA In-Ceram YZ ceramic is a Y-TZP ceramic recommended for a framework of FDPs and monolithic restorations and has been extensively used globally. Hence, a fundamental study involving a complete characterization of the effect of different grinding grit sizes using a promising grinding tool (as demonstrated by Pereira et al., 2015[b]; 2016[c]), in a protocol that simulates the movements executed in a clinical scenario is important.

This *in vitro* study aimed to evaluate the effects of grinding (with diamond burs of different grit sizes under an oscillatory movement) and low temperature aging on the surface micromorphology (roughness measurements, scanning electron microscopy (SEM) and atomic force microscopy (AFM) micrographs), phase transformation (t→m), biaxial flexural strength, and structural reliability (Weibull parameters) of yttrium-stabilized tetragonal zirconia polycrystal ceramics.

2.2. MATERIALS AND METHODS

Specimens' preparation

Disc-shaped specimens (n = 204) were manufactured according to ISO 6872-2008. Pre-sintered blocks of Y-TZP zirconia (VITA In-ceram YZ for inLab 40/19, Zahnfabrik, Bad-Säckingen, Germany) were shaped into cylinders using 600–1200 grit SiC papers (3M, St. Paul, Minnesota USA) under constant water-cooling. Slices of 18 mm (Ø) × 1.6 mm (thickness) were obtained using a precision saw (ISOMET 1000, Buehler, Lake Bluff, IL, USA). To remove any surface irregularities introduced by processing and to standardize the surfaces, the specimens were fine ground with 1200 grit SiC paper and cleaned in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ind. e Com. Equip. Med. Odonto. LTDA, Ribeirao Preto, Sao Paulo, Brazil) containing 78% isopropyl alcohol, for 10 minutes.

After that, the specimens were sintered (VITA furnace Zyrcomat 6000 MS, Vita Zahnfabrik, Germany) according to the manufacturer's instructions (heat rate 1: 60 °C/min up to 700 °C; heat rate 2: 60 °C/min up to 1300 °C; heat rate 3: 40 °C/min up to 1530 °C; dwell time: 25 min, followed by slow cooling by opening the furnace at temperatures below 400 °C). The discs with final dimensions of approximately 15 mm × 1.2 mm (±0.2 mm) were then randomly assigned into six groups (Table 1).

Surface treatments

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

Grinding: One of the major restrictions during grinding in a clinical environment is the difficulty in standardizing the direction of movement. In this study, it is assumed that under in vitro (laboratory) conditions, oscillatory movements of the grinding tool would mimic the grinding movement more precisely, thus introducing more superficial defects in all possible directions and significantly affecting the mechanical properties.

Based on this concept, a series of grinding pilot studies were undertaken, followed by SEM and roughness analysis, until a reproducible procedure was achieved. After that, a single trained operator ground one side of each specimen using a coarse or an extra-fine diamond bur (3101G - grit size 151 µm, and 3101FF - grit size 30 µm; KG Sorensen, Cotia, Sao Paulo, Brazil) with a contra-angle handpiece (T2 REVO R170 up to 170,000 rpm, Sirona, Bensheim, Germany) coupled to a low speed motor (Kavo Dental, Biberach, Germany), under constant water cooling (~30 mL/min). After each treatment, the diamond bur was replaced by a new one (1 bur/specimen) (Pereira et al., 2015[b]; 2016[c]).

To ensure stability during grinding, the specimens were fixed in a metal base with double-sided tape (3M do Brazil, Sumare, Sao Paulo, Brazil). The specimens were marked with a permanent marker pen (Pilot, Sao Paulo, Sao Paulo, Brazil) to standardize the abrasion thickness and ensure that the entire surface of the specimen was treated. Grinding was performed in an oscillatory motion under gentle pressure (finger pressure) until the mark made by the pen was eliminated.

Hydrothermal Aging

Low-temperature degradation was simulated in an autoclave (Sercon HS1-0300 n11560389/1) at 134 °C, under 2 bar (0.2 MPa) of pressure, for 20 h (Pereira et al., 2015[a]).

Phase transformation analysis

Quantitative analysis of the phase transformation was conducted ($n = 2$) to determine the relative amount of the m-phase present in each of the evaluated conditions. The analysis was performed using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected in the 2θ range of 25 to 35 degrees (where θ is the angle of reflection), at a step interval of 1 s, and step size of 0.03 degrees/step. The monoclinic phase fraction (X_M) was calculated using the Garvie and Nicholson (1972) method:

$$X_M = \frac{(-111)_M + (+111)_M}{(-111)_M + (+111)_M + (111)_T} \quad \text{Eq. (1)}$$

where $(-111)_M$ and $(+111)_M$ represent the monoclinic peaks (peaks around 28 degrees and 31 degrees), and $(111)_T$ indicates the intensity of the respective tetragonal peak (peak around 30 degrees). The volumetric fraction (F_m) of the m-phase was calculated according to Toraya et al. (1984):

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

Surface topography and roughness analysis

The surface topography patterns were qualitatively and quantitatively analyzed using a surface roughness tester ($n = 30$; Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan), a scanning electron microscope (SEM) ($n = 1$, JSM-6360, JEOL, Tokyo, Japan) and an atomic force microscope (AFM) ($n = 1$, Agilent Technologies 5500, Chandler, Arizona, USA).

The arithmetic mean of three surface roughness measurements was calculated for each specimen, based on the parameters of ISO 1997 (R_a - arithmetic mean of the absolute roughness values of the peaks and valleys measured from a medium plane (µm), and R_z - average distance between the five highest peaks and five lowest valleys found in the standard (µm)) with a cut-off wavelengths ($n = 5$), $\lambda c = 0.8$ mm and $\lambda s = 2.5$ µm. This analysis was performed on all specimens undergoing the biaxial flexural strength test and was aimed at evaluating any correlation between roughness and strength.

Two additional specimens were manufactured for each evaluated condition. One was sputter coated with a gold-palladium alloy and examined under a SEM to obtain images at 20, 500, 1000 and 5000x magnification. The second was analyzed by an AFM using a non-contact methodology with specific probes in an area of 20 × 20 µm (PPP-NCL probes, nanosensors, force constant = 48 N/m). The AFM images were processed with a specific computer software Gwyddion (Gwyddion™ version 2.40, GNU, Free Software Foundation, Boston, Massachusetts, USA).

Biaxial flexure test (piston-on-three-balls test)

The biaxial flexural strength (n = 30) was measured with a piston-on-three balls test using a universal mechanical testing machine (EMIC DL 2000, Sao Jose dos Pinhais, Brazil), according to ISO 6872-2008. Each specimen was positioned with the treated surface facing down (tensile stress zone) on three steel balls (diameter of 3.2 mm, positioned 120° apart on a support circle with a diameter of 10 mm). Before testing, an adhesive tape was fixed on the compression side of the discs to distribute the contact pressure. This also helped provide better contact between the piston and the disc (Wachtman et al., 1972) and avoided spreading the fragments (Quinn, 2007). Another film of a non-rigid material (cellophane, 2.50 µm) was placed between the supporting balls and the specimen (tensile surface), and for better distribution of the contact pressure (ISO 6872-2008). The set was immersed in water and a flat, circular tungsten piston (Ø = 1.6 mm) was used to apply an increasing load (1 mm/min) until catastrophic failure occurred. The biaxial flexural strength (in MPa) was calculated according to ISO 6872-2008:

$$\sigma = -0.2387 \cdot \frac{P(X-Y)}{b^2} \qquad \text{Eq. (3)}$$

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 by:

$$X = (1+\nu)ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{(1-\nu)}{2}\right]\left(\frac{r_3}{r_3}\right)^2 \qquad \text{Eq. (4)}$$

$$Y = (1+\nu)\left[1 + ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-\nu)\left(\frac{r_1}{r_3}\right)^2, \qquad \text{Eq. (5)}$$

where ν is Poisson's ratio (ν = 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).

Data analysis

Statistical analysis was carried out with Minitab 16 and Statistix 8.0. A descriptive analysis of the roughness and the strength data was carried out to obtain the mean and the standard deviation values. Normality (Shapiro-Wilk) and homoscedasticity tests were performed to evaluate the data distribution. As the roughness data (R_a and R_z) displayed a nonparametric distribution, a Kruskal-Wallis test, in addition to a post-hoc Dunn's test, was performed considering two factors (grinding and aging) and the interaction between them. A Pearson correlation test was performed to determine the linear correlation between the roughness data (R_a) and the biaxial flexural strength (MPa).

The variability of the flexural strength values was analyzed using the two-parameter Weibull distribution function (Weibull, 1951). This is a method to describe the variation of resistance by obtaining the Weibull modulus (m) and the characteristic strength (σ_c) with a confidence interval of 95%, as determined by a diagram according to DIN ENV 843-5 (2007):

$$\ln \ln \left(\frac{1}{1-\mathrm{F}}\right) = m \ln \sigma_c - m \ln \sigma_0 \qquad \text{Eq. (6)}$$

where F is the failure probability, σ_0 the initial strength, σ_c the characteristic strength, and *m* is the Weibull modulus. The characteristic strength is the strength at a failure probability of 63.2%. Weibull modulus is a measure of the distribution of the strength, and is a way of expressing the reliability of a material.

Qualitative fractographic analysis

Fractured specimens of all groups were cleaned in 78% isopropyl alcohol, for 10 min by ultrasonic immersion and then analyzed under a light microscope (Stereo Discovery V20, Carl Zeiss; Gottingen, Germany) to determine the location of the fracture origin. Representative specimens were selected and sputter coated with platinum. These samples were then analyzed by field emission scanning electron microscopy (FE-SEM Inspect F50, FEI; Hillsboro, Oregon, USA), to assess the effect of different aging protocols on the fracture origin mode. The fracture surfaces were qualitatively analyzed at 200, 1000 and 5000x magnification..

2.3 RESULTS

Qualitative surface analysis (SEM and AFM) showed that grinding with diamond burs (Xfine and Coarse) modified the topographic pattern. Oscillatory grinding was found to have introduced scratches that followed a principal direction with some lateral projections. Aging did not cause noticeable surface alteration for either as-sintered surfaces or ground surfaces (Figs. 1, 2 and 3). Grinding significantly increased the roughness values (R_a and R_z parameters) which were directly related to the grit-size of the bur (Coarse > Xfine > Ctrl). Aging only promoted a significant decrease for the Xfine group and did not alter the other conditions (Coarse = $\text{Coarse}^{\text{LTD}}$ > Xfine > $\text{Xfine}^{\text{LTD}}$ > Ctrl = Ctrl^{LTD}) (Table 2).

The fractographic analysis showed similar fracture patterns in all groups. A critical defect for each fracture event originated on the bottom surface of each specimen, at the tensile side of the biaxial flexural strength test. The failure characteristics are shown in Fig. 4.

The Pearson linear correlation coefficient between the roughness data (R_a) and the biaxial flexural strength (MPa) demonstrated a significantly weak negative correlation ($p < 0.05$) for the Ctrl group ($0.1 < (r) < 0.4$) and a significantly moderate negative correlation ($p < 0.005$) for the Xfine group ($0.4 < (r) < 0.6$), according to the Dancey & Reidy (2006) scale. No statistical difference was observed for the other conditions (Table 2).

X-ray diffraction showed that grinding and aging promoted an important increase in the m-phase content. Coarse burs appear to promote a higher m-phase transformation compared to Xfine burs. Grinding also affected the material's susceptibility to the t-m phase transformation during aging. A lower m-phase content was noted for the ground groups than for the Ctrl group after aging (before aging: Ctrl < Xfine < Coarse; after aging: $\text{Xfine}^{\text{LTD}}$ < $\text{Coarse}^{\text{LTD}}$ < Ctrl^{LTD}) (Table 2).

Grinding had no effect on the reliability (Weibull moduli) of the material, while low temperature aging increased it for the as-sintered condition (Ctrl). Grinding, irrespective of the grit-size, significantly increased the characteristic strength compared to the Ctrl group. Aging increased the characteristic strength for the as-sintered condition only (Ctrl < Ctrl^{LTD}; Xfine = $\text{Xfine}^{\text{LTD}}$; Coarse = $\text{Coarse}^{\text{LTD}}$) (Table 2).

2.4 DISCUSSION

From the findings of this study, we may note that, regardless of the grit-size, grinding (using a high torque contra-angle handpiece (T2 REVO R170 up to 170,000 rpm) coupled to a low speed motor, under constant water cooling (~30 mL/min), performed in an oscillatory motion and with gentle finger pressure) did not reduce the mechanical properties of the Y-TZP ceramic, although an increase in the roughness and the m-phase content was noticed. Aging did not impair the mechanical properties, although a wide m-phase transformation was observed for the aged samples.

The literature reports indicate that procedures to adjust the zirconia surface, such as grinding, can induce superficial modifications, damage, and phase transformation from the tetragonal (t) to the monoclinic (m) phase (Karakoca and Yilmaz, 2009; Maerten et al., 2013; Mochales et al., 2011; Pereira et al., 2016[a]).

From the SEM and AFM micrographs (Fig. 2), it can be seen that the oscillatory movement of the grinding tool generated oscillatory scratches that followed the grinding direction. The generated surface appears similar to that observed in previous studies at the microscopic scale (Fig. 1) (Pereira et al., 2015[b]; 2016[c]), a fact that may indicate that the defects introduced were similar. Those studies used a similar grinding protocol to the one used here (same grinding instrument, under the same cooling protocol, and with similar pressure). The only difference was that in previous studies, a more controlled set up was used (with a device that allowed movement in only one direction), whereas in this study a manual oscillatory motion was introduced. This simulates a clinical scenario better. It should also be noted that each study used Y-TZP ceramics from different manufacturers.

The roughness data collected in this study corroborates the literature data, which shows that an increase in the bur grit size leads to increased roughness (Curtis et al., 2006; Pereira et al., 2015[b]; 2016[c]). Flury et al. (2012) stated that the surface roughness might play a crucial role in the resistance of ceramics, and usually has a significant negative correlation with flexural strength (higher roughness with lower flexural strength).

Quinn (2007) however, explained that the presence of a roughness-strength correlation is only observed in some specific cases and is defined by the balance between the depths of the defects introduced by grinding, compared to existing surface flaws. When the depth of the introduced cracks is similar to that of the existing surface flaws, a correlation would not be expected. However, when the introduced cracks are deeper than the existing surface flaws, a stronger correlation is noticed. Additionally, the phase transformation toughening mechanism, which is triggered by grinding, may have a counter-balance effect, decreasing the influence of roughness and resulting in the absence of any correlation.

It is important to highlight that, contrary to what was expected, our results are in agreement with two other studies that evaluated different Y-TZP ceramics under similar conditions to those evaluated by us (Pereira et al., 2015[b]; 2016[c]). The literature states that Y-TZP responses to the stimuli are material dependent (altering the composition, stabilizer content, grain size and protocol of processing leads to alterations in the response) (Chevalier et al., 2007; Pereira et al., 2015[a]). This should mean that Y-TZP ceramics from different manufacturers should exhibit different susceptibility to phase transformation, and consequently distinct mechanical behavior. The findings in this study indicate that these Y-TZP ceramics behave similarly and that the grinding protocol seems to be more relevant than small differences between materials composition and processing. The effect of grinding and aging on these other Y-TZP ceramics could be extrapolated to some extent, from the results of this study.

One explanation for this fact could be that previous studies, as well as ours, evaluated grinding protocols that may be considered “gentle.” This does not necessarily mean the same outcome would be observed using a more severe grinding protocol. In grinding protocols under high speeds, high load forces and/or without proper cooling the surface temperature may rise, (Iseri et al., 2012; Kosmac et al., 2008; Pereira et al., 2016[a]; Swain and Hannink, 1989) achieving temperatures above the critical point at which the t-m phase transformation can occur. This leads to the inverse transformation (m-t) and increases the risk of introducing critical defects (Iseri et al., 2010; Jing et al., 2014; Pereira et al., 2016[a]; Yin et al., 2003). Studies that have evaluated this scenario commonly notice a decrease in the mechanical properties of Y-TZP (Curtis et al., 2006; Iseri et al., 2012; Kosmac et al., 1999; 2000; Kosmac and Dakskobler, 2007; Kosmac et al., 2008; Michida et al., 2015; Pereira et al., 2016[a]).

Some studies evaluated the effect of aging on ground Y-TZP ceramics (Amaral et al., 2013; Kim et al., 2010; Kosmac and Dakskobler, 2007; Kosmac et al., 2008; Pereira et al., 2015[b]; 2016[a]; 2016[b]; 2016[c]; Sato et al., 1996). Unfortunately, these studies have a wide variability in grinding and aging protocols that were used, and a clear interpretation is difficult. Pereira et al. (2015[a]) conducted a systematic review of aging in Y-TZP ceramics (only the as-sintered condition, without surface treatment). They noticed that the aging protocol is fundamental to the final outcome on the mechanical properties, and that a deleterious effect was only observed when more than 50% of the m-phase was present.

In another systematic review (Pereira et al., 2016[a]) it was noted that, although the authors state that there is insufficient aging data to draw final conclusions, the literature indicates that the grinding protocol plays an important role in this scenario, as it leads to residual stress concentration on the ceramic surface which decreases the susceptibility of the Y-TZP ceramic to new t-m phase transformations.

The lower susceptibility of ground groups to the t-m phase transformation was attributed in some studies to the introduction of compressive residual stresses on the Y-TZP surface (Chevalier et al., 2007). Muñoz-Tabares et al. (2011), however, attributed this fact to microstructural changes caused by grinding. The microstructural changes essentially consist of three well defined layers from the outer to the inner surface and can be described as follows: (1) a crystallized zone, just on the surface, where the grains have a diameter in the range of 10–20 nm; (2) a plastically deformed zone; (3) a zone in which a t-m phase transformation has taken place.

In this scenario, the resistance to hydrothermal degradation could be related to the existence of this very thin layer of tetragonal recrystallized nano-grains (10-20 nm), with sizes smaller than the critical size for transformation in a humid environment.

Even though the benefits of the transformation toughening mechanism have been stated, the scientific community is still concerned about the fact that a high t-m transformation rate may decrease mechanical stability over-time. As previously mentioned, grinding generates micro cracks in the transformed grain boundaries. These micro cracks can act as paths for water penetration into the material, increasing the susceptibility to LTD, and potentially jeopardizing the mechanical properties of Y-TZP ceramic under mechanical fatigue (Chevalier et al., 2007; Pereira et al., 2015[a]; 2016[a]). Currently, there are few studies that evaluate the fatigue behavior of Y-TZP ceramics after grinding (Kosmac and Dakskobler, 2007; Kosmac et al., 2008; Pereira et al., 2016[b]) and hence more studies need to be carried out to better understand this phenomenon.

In the oral environment, ceramic restorations are susceptible to fatigue failure, mainly due to the presence of cyclic masticatory forces and moisture (Zhang et al., 2013). Fatigue failure may be defined as the fracture of a material due to progressive brittle cracking under repeated cyclic stresses, with intensity below the material's monotonic strength (Wiskott et al., 1995). It could be considered as the limiting point of our investigation as no fatigue evaluation has been conducted.

Another important consideration is that our study evaluated a restricted scenario, since the increase in characteristic strength, in response to grinding, should not be considered as a positive endorsement of grinding in a clinical scenario. These results serve to highlight the potential of toughening in response to the phase transformation mechanism. As with all other important findings related to a clinical environment, additional studies are required to fully comprehend the impact of grinding on mechanical properties (fatigue) and oral biofilm formation.

2.5 CONCLUSIONS

- Aging and grinding did not reduce the characteristic strength of the tested Y-TZP ceramic; on the contrary, they led to an increase in strength, caused by the phase transformation toughening mechanism (increase in the m-phase content).
- Grinding significantly increased the zirconia surface roughness, and was directly related to the grit-size of the diamond bur.
- The percentage of the m-phase was higher after undergoing a low temperature aging process for all evaluated conditions. However, ground surfaces had lower m-phase content after aging when compared to as-sintered samples. It appears that grinding may decrease the effect of hydrothermal aging (susceptibility to t-m phase transformation).

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2.8 FIGURES AND TABLES

Figures

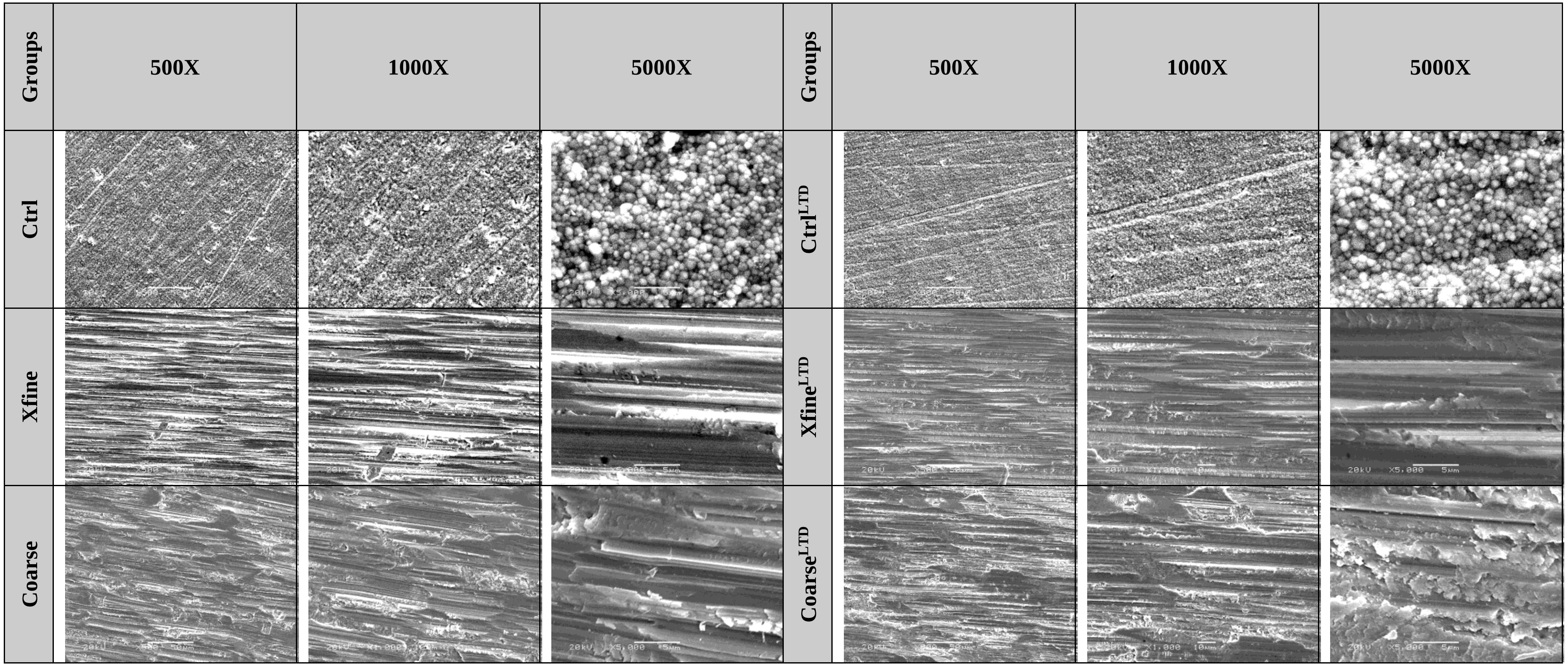

Figure 1- SEM images of zirconia surface of all study groups. Grinding with diamond burs (Xfine and Coarse) modified the topographic pattern of "as sintered" samples creating parallel scratches following a main direction, while aging did not cause a relevant alteration of this pattern.

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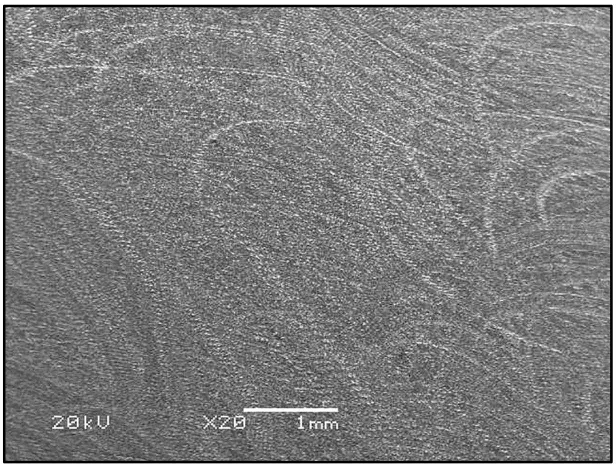
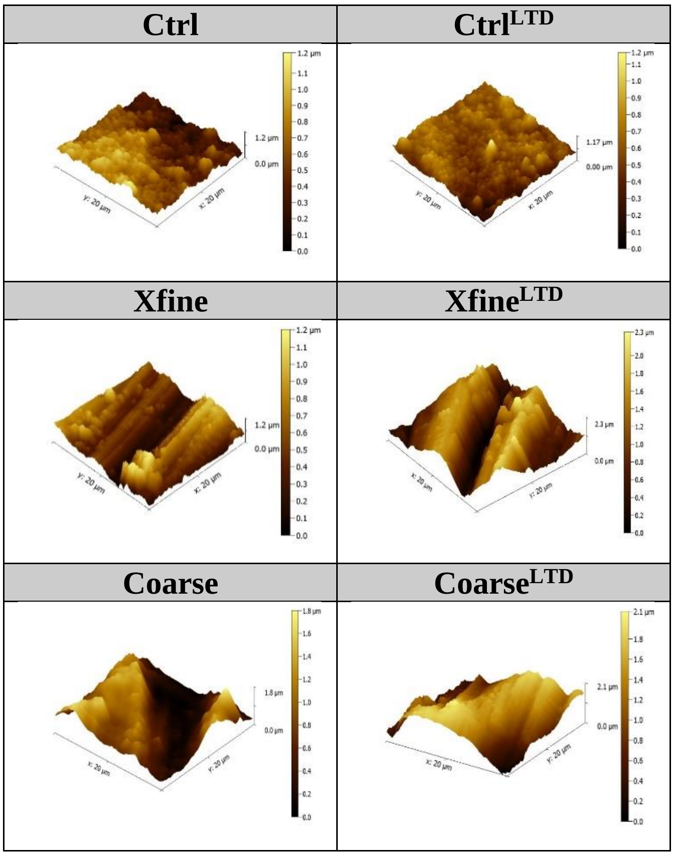


Figure 2 – Micrographic of zirconia sample of the Coarse group (20x magnification) showing the projections of scratches in all directions because of the oscillatory grinding.


Ctrl

Ctrl^{LTD}

Xfine

Xfine^{LTD}

Coarse

Coarse^{LTD}

Figure 3 - AFM images (20 µm X 20 µm) of tested groups. We observed the same pattern that the SEM test.

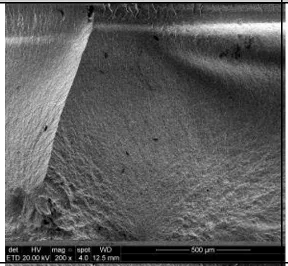
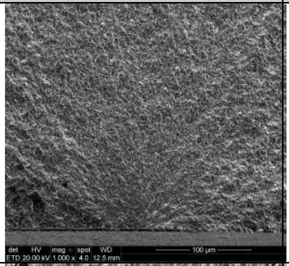
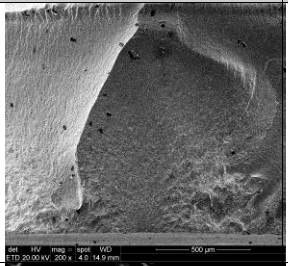
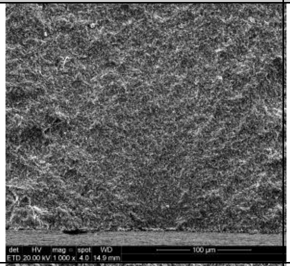
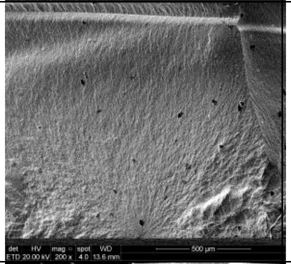
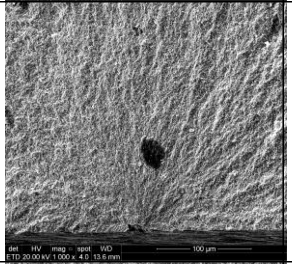
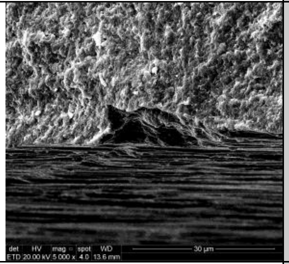
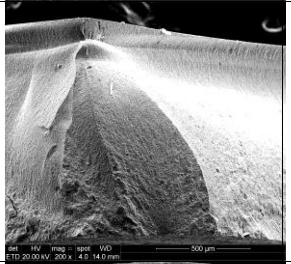
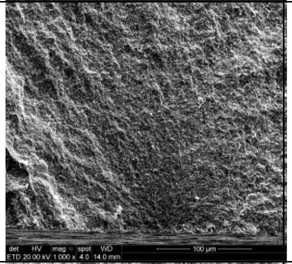
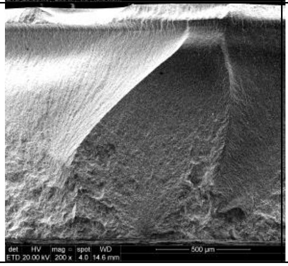
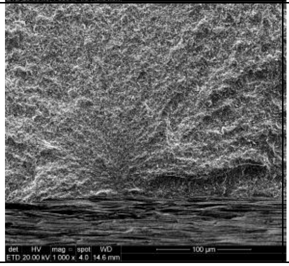
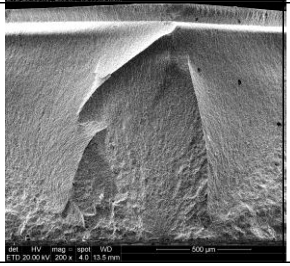
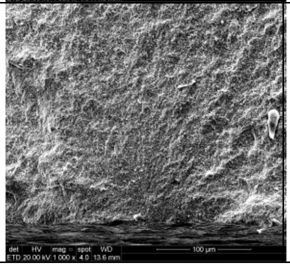
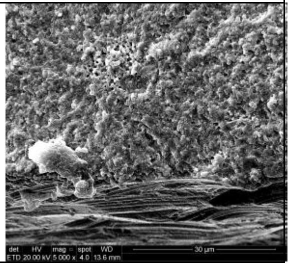
Groups	200x	1000X	5000X	Groups	200X	1000X	5000X
Ctrl			-	**Ctrl^{LTD}**			-
Xfine				**Xfine^{LTD}**			-
Coarse			-	**Coarse^{LTD}**			

Figure 4 – Fractographic analysis: Typical brittle fracture surfaces, which clearly shows that the initial crack nucleation and propagation region are located in the lower side of the micrographs, corresponding to the region of maximum tensile stress in the biaxial test.

Tables

Table 1 - Experimental Design

Groups	Study factors	
	Surface treatment	LTD
Ctrl	Control, as-sintered (without any additional treatment)	Without
Ctrl^{LTD}		With
Xfine	Grinding with extra-fine diamond bur (#3101FF - grit size 30 µm, KG Sorensen, Cotia, Brazil)	Without
Xfine^{LTD}		With
Coarse	Grinding with coarse diamond bur (3101G - grit size 151 µm, KG Sorensen, Cotia, Brazil)	Without
Coarse^{LTD}		With

Table 2 - X-ray Difractometry analysis (m-phase content); Roughness analysis (Ra and Rz parameters - µm) with mean and standard deviation (SD); Descriptive analysis of biaxial flexural strength data (mean and standard deviation - MPa); Pearson correlation between biaxial flexural strength and roughness Ra; and Weibull analysis data (Characteristic Strength - σ_c with 95% confidence interval and Weibull Moduli - *m* with 95% confidence interval).

GROUPS	XRD Analysis	Roughness Analysis		Descriptive Analysis of Strength	Pearson Linear Correlation (σ x Ra)	Weibull Analysis	
	m-phase (%)	Ra (SD)	Rz (SD)	Mean (SD)		*m* (95% CI)	σ_c (95% CI)
Ctrl	0.00%	0.20 (0.05)^{A}	1.70 (0.40)^{A}	889.63 (99.97)	-0.36 (p=0.05)*	10.8 (7.6-13.9)^{A}	932.1 (895.3-969.2)^{A}
Ctrl^{LTD}	63.12%	0.23 (0.09)^{A}	1.82 (0.56)^{A}	993.36 (51.46)	-0.06 (p=0.76)	21.4 (15.0-27.6)^{B}	1018.8 (998.3-1039.1)^{B}
Xfine	9.70%	0.82 (0.29)^{C}	5.09 (1.38)^{C}	1162.2 (171.03)	-0.51 (p=0.00)*	8.1 (5.7-10.4)^{A}	1231.4 (1167.0-1297.5)^{C}
Xfine^{LTD}	24.42%	0.69 (0.13)^{B}	4.33 (0.74)^{B}	1091.6 (106.44)	-0.20 (p=0.28)	11.2 (7.8-14.4)^{A}	1142.7 (1099.1-1186.7)^{C}
Coarse	13.07%	1.33 (0.14)^{D}	8.14 (1.01)^{D}	1084.4 (104.51)	-0.06 (p=0.77)	12.4 (8.7-15.9)^{AB}	1129.8 (1090.8-1169.1)^{C}
Coarse^{LTD}	40.94%	1.36 (0.15)^{D}	8.03 (0.94)^{D}	1105.8 (80.33)	-0.13 (p=0.48)	16.7 (11.7-21.5)^{AB}	1140.9 (1111.6-1170.2)^{C}

-same letters correspond to statistical similarity
-different letters correspond to statistical difference
***indicates that there is statistical difference**

3. CONCLUSÃO

Através deste trabalho pudemos concluir que tanto o desgaste com brocas diamantadas grossa e extra-fina quanto o envelhecimento em autoclave associado ou não ao desgaste da superfície do material, não foram deletérios às propriedades da Cerâmica In-Ceram YZ da VITA. Pelo contrário, esses tratamentos promoveram aumento da resistência do material devido ao mecanismo de tenacificação por transformação de fase (t->m). Outra observação foi que o desgaste aumentou consideravelmente a rugosidade do material, sendo diretamente relacionado ao tamanho do grão da broca utilizada.

O envelhecimento em autoclave foi o que mais aumentou o conteúdo de fase monoclínica em todos os grupos, mas esse aumento foi menor nos grupos desgastados se comparados ao grupo controle. Assim, parece que o desgaste reduz a susceptibilidade do material ao envelhecimento em autoclave.

Esse trabalho é muito importante para entendermos o processo de degradação que ocorre na cerâmica Y-TZP quando submetida ao desgaste clínico e a um ambiente úmido com baixas temperaturas. Porém, mais estudos que associam diferentes estímulos que ocorrem em boca (mastigação, umidade, temperaturas, variação de pH) são necessários para compreendermos melhor a degradação desse material a longo prazo no ambiente oral.

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