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EFEITO DO DESGASTE COM INSTRUMENTOS DIAMANTADOS E DA DEGRADAÇÃO A BAIXAS TEMPERATURAS NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP

DISSERTAÇÃO DE MESTRADO

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Santa Maria, RS, Brasil

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EFEITO DO DESGASTE COM INSTRUMENTOS DIAMANTADOS E DA DEGRADAÇÃO A BAIXAS TEMPERATURAS NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP

Gabriel Kalil Rocha Pereira

Dissertação 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 grau de **Mestre em Ciências Odontológicas com ênfase em Prótese Dentária**

Orientador: Prof. Dr. Luiz Felipe Valandro

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A Comissão Examinadora, abaixo assinada,
aprova a Dissertação de Mestrado

EFEITO D DESGASTE COM INSTRUMENTOS DIAMANTADOS E DA DEGRADAÇÃO A BAIXAS TEMPERATURAS NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP

elaborada por

Gabriel Kalil Rocha Pereira

como requisito parcial para obtenção do grau de

Mestre em Ciências Odontológicas com ênfase em Prótese Dentária

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“É muito melhor lançar-se em busca de conquistas grandiosas,
mesmo expondo-se ao fracasso,
do que alinhar-se com os pobres de espírito,
que nem gozam muito nem sofrem muito,
porque vivem numa penumbra cinzenta,
onde não conhecem nem vitória, nem derrota.”

(Theodore Roosevelt)

RESUMO

Dissertação de Mestrado
Programa de Pós-Graduação em Ciências Odontológicas
Universidade Federal de Santa Maria

EFEITO DO DESGASTE COM INSTRUMENTOS DIAMANTADOS E DA DEGRADAÇÃO A BAIXAS TEMPERATURAS NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP
AUTOR: GABRIEL KALIL ROCHA PEREIRA
ORIENTADOR: LUIZ FELIPE VALANDRO
Data e Local da Defesa: Santa Maria, 05 de agosto de 2014

Objetivos: (1) Comparar os efeitos do desgaste da cerâmica Y-TZP gerados por lixas e pontas diamantadas (com granulações semelhantes) na micromorfologia da superfície, na transformação de fase (t→m), na resistência à flexão biaxial e confiabilidade estrutural (análise de *Weibull*), (2) Avaliar o efeito da LTD nos desfechos citados acima. **Métodos:** Trezentos e sessenta discos (15 mm x 1,2 mm) de Y-TZP foram confeccionados segundo as instruções da ISO 6872 – 2008 para ensaios de flexão em material cerâmico e sinterizados de acordo com as recomendações do fabricante, posteriormente foram divididos em grupos, de acordo com dois fatores em estudo: "*tratamento de superfície da cerâmica*" - 5 níveis (sem tratamento, disco de granulação 120, ponta diamantada de granulação super grossa, disco de granulação 600, ponta diamantada extra fina) e "LTD" - 2 níveis (sem e com). Para abrasão com lixas de diferentes granulações foi utilizada uma politriz, enquanto que para a abrasão com as pontas diamantadas foi utilizado um dispositivo que garantiu a perpendicularidade da ponta diamantada à superfície da amostra, padronizando os movimentos de abrasão e a pressão aplicada. A LTD foi realizada em autoclave sob 134° C à 2 bar por 20 horas. **Resultados:** Nossos achados suportam que a LTD embora promovesse aumento de fase monoclínica e alterações micromorfológicas não causou perda de propriedades mecânicas da zircônia, o desgaste em primeiro momento também não promoveu, mas quando esta superfície foi submetida aos efeitos da LTD, os defeitos introduzidos pelo desgaste podem ser prejudiciais a resistência do material. Sob um ponto de vista metodológico o uso de discos diamantados não deve ser empregado para simular o desgaste executado clinicamente com pontas diamantadas. **Conclusão:** Dessa forma o desgaste da superfície da zircônia deve ser evitado e quando necessário deve ser realizado com instrumentos de menor granulação.

Palavras-chave: Instrumentos Odontológicos. Prótese Dental. Cerâmica. Materiais Odontológicos. Zircônia parcialmente estabilizada por óxido de ítrio.

ABSTRACT

Master Course Degree
Post Graduate Program in Dental Science
Federal University of Santa Maria

EFFECT OF GRINDING WITH DIAMOND INSTRUMENTS AND LOW-TEMPERATURE DEGRADATION ON THE MECHANICAL BEHAVIOR OF A Y-TZP CERAMIC.

AUTHOR: GABRIEL KALIL ROCHA PEREIRA
ADVISER: LUIZ FELIPE VALANDRO
Defense Place and Date: Santa Maria, 05 August, 2014

Objectives: (1) Compare the effects of grinding on a Y-TZP ceramic executed by diamond discs and diamond burs (with similar grit sizes) in the micromorphology of surface, phase transformation (t→m), flexural strength and structural reliability (Weibull analysis), (2) evaluate the effect of LTD (low-temperature degradation) in the outcomes mentioned above. **Methods:** Three hundred and sixty discs (15mm x 1,2 mm) of Y-TZP were made according to ISO 6872 – 2008 for flexural strength determination on ceramic materials and sintered according to the manufacturer`s instructions, than they were divided into groups according to two factors in study: “surface treatment” - 5 levels (without treatment, extra-fine diamond bur, 600-grit diamond disc, coarse diamond bur and 120-grit diamond disc) and “LTD” on 2 levels (with and without). For grinding with diamond discs a polishing machine was employed, while for grinding with diamond burs a device was employed to assure the perpendicularity between diamond tip and abrading surface, that way the abrasion movements and the applied pressure were standardized. The LTD was induced in an autoclave at 134ºC under 2 bar for 20 hours. **Results:** Our findings support that LTD although promoted increase in m-phase content and micromorphological alterations did not promoted decrease in zirconia’s mechanical properties, grinding at a first moment did not affected too, but when ground Y-TZP was submitted to the LTD effects, the defects introduced by grinding could be detrimental to the material`s resistance. From a methodological point of view, diamond discs should not be employed to simulate clinical abrasion performed with diamond burs on Y-TZP ceramics. **Conclusion:** Thus grinding of zirconia should be avoided and when it was really necessary tools with low grit sizes should be employed.

Key Words: Dental instruments. Dental prosthesis. Ceramics. Dental materials. Zirconium oxide partially stabilized by yttrium.

SUMÁRIO

1. INTRODUÇÃO GERAL

Atualmente, existe no mercado um grande número de sistemas cerâmicos disponíveis para o uso clínico (Blatz et al. 2003; Ban et al. 2008). Com o avanço dos procedimentos CAD/CAM (*computer assisted design/computer assisted machining*) nos anos 80, foram criadas alternativas aos sistemas convencionais otimizando os materiais cerâmicos (Kamada et al. 1998). Dentre estes, podemos destacar a zircônia, cujas propriedades apresentam boa estabilidade química e dimensional, resistência mecânica e tenacidade, além de módulo de Young na mesma ordem de grandeza das ligas de aço inoxidável (Piconi & Maccauro 1999).

As propriedades mecânicas da zircônia são hoje as mais altas reportadas entre todas as cerâmicas dentárias. Isto pode permitir a realização de próteses parciais fixas posteriores e permitir uma redução substancial na espessura da infraestrutura. Estas capacidades são altamente atrativas para prótese dentária, onde resistência e estética são proeminentes (Denry & Kelly 2008).

A zircônia é um polimorfo que ocorre em três formas cristalinas: a zircônia pura é monoclínica (m) à temperatura ambiente até 1170°C, acima desta temperatura, os cristais passam à forma tetragonal (t) e acima de 2370°C, a conformação cristalina estável é a cúbica (c). Durante o resfriamento, a transformação de fase t→m está associada a um aumento de volume de aproximadamente 3-4%. A tensão gerada por esta expansão pode originar trincas na estrutura da cerâmica (Piconi & Maccauro 1999). Por este motivo, adicionaram-se óxidos "estabilizadores" à zircônia pura, permitindo que a conformação tetragonal se mantenha em temperatura ambiente.

Porém, apesar de a zircônia apresentar em sua composição óxidos que a estabiliza a temperatura ambiente, ela pode sofrer modificação de fase ($t \rightarrow m$) basicamente pela ação de dois processos: devido à aplicação de carga (tensão) ou à ação de baixas temperaturas (*low-temperature degradation*) (Kobayashi et al. 1981; Sato & Shimada 1985; Papanagiotou 2006; Chevalier et al. 2007).

A LTD (*Low-Temperature degradation*) foi pela primeira vez reportada por Kobayashi et al. (1981), que demonstrou que esta ocorre espontaneamente quando a zircônia é exposta à umidade e a baixas temperaturas (150 – 400º C) por longo período de tempo. Lughi & Sergo (2010) descreveu este fenômeno demonstrando que a LTD ocorre em amostras de zircônia pura estabilizada com pelo menos 2,5 mol% Y_2O_3, e que esta transformação *t→m* ocorre inicialmente na superfície da amostra e então penetra no corpo do material, o crescimento da área de transformação resulta em introdução de falhas, perda de material (*grain pullout*) e aumento da rugosidade superficial o que acaba por conduzir a uma diminuição da resistência do material, sendo possivelmente causada e certamente acelerada pelo vapor de água e umidade.

Além da LTD, outros fatores que podem alterar as propriedades mecânicas da zircônia são ajustes da superfície de cimentação de cerâmicas Y-TZP, através de desgaste e/ou polimento para um melhor assentamento da peça protética, que são geralmente executados pelo dentista ou pelo protético (Aboushelib et al. 2009). O desgaste da superfície após a sinterização cria uma camada de tensão compressiva, devido à transformação *t→m*, porém alguns defeitos superficiais podem ser introduzidos na superfície do material, quando a profundidade destes defeitos introduzidos é maior do que a camada compressiva formada, eles podem atuar como zonas de concentração de tensão, o que diminuiria a resistência da peça (Kosmac et al. 1999; Guazzato et al. 2005). Já quando estes defeitos são menores do que a camada de tensão compressiva formada esta transformação é benéfica, pois resulta em um fechamento parcial das micro-falhas (*crack*), aumentando a resistência do material à propagação de trincas (Kamada et al. 1998).

Kosmac et al. 1999, 2007 e 2008 notou que quando são executados desgastes na superfície da cerâmica Y-TZP com brocas diamantadas não foi promovida grande alteração de fase *t→m* e foi introduzido uma grande quantidade de defeitos no material, o que levou a degradação de propriedades mecânicas. Estes trabalhos atribuíram esse fato ao uso de alta rotação para o desgaste, o que levou há um aumento significativo da temperatura na superfície da zircônia o que pode ter desencadeado uma transformação reversa *m → t*, o que também pode ser atribuído a uma refrigeração ineficiente durante o desgaste, desta forma não foi promovida a formação de uma camada de tensão compressiva residual pela transformação t→*m* e os defeitos introduzidos agiram como fatores de concentração de tensão levando a fratura catastrófica do material.

Em termos de reprodutibilidade metodológica, o uso de lixas pode ser mais viável em relação a pontas diamantadas à medida que estas podem ser usadas em equipamentos tipo politriz, com o qual é possível padronizar as condições de teste. Por outro lado, o uso de lixas pode não representar a condição clínica de uso de pontas diamantadas para desgaste de superfícies de restaurações, como a superfície interna de infraestruturas de cerâmica Y-TZP.

Diante destes aspectos, a análise do comportamento da zircônia após o tratamento de superfície e o efeito da LTD sobre essa superfície formada após a abrasão parece ser pertinente, à medida que este procedimento é corriqueiramente usado na clínica e não existe consenso na literatura a cerca da degradação das propriedades mecânicas que este material pode sofrer quando submetido a estes procedimentos. Além disso, a formação da camada compressiva pode levar a um aumento de resistência do material dificultando à propagação de trincas, mas pode levar à uma maior susceptibilidade do material a LTD (Kim et al. 2010), levando o material a falha catastrófica a longo prazo.

2. OBJETIVOS GERAIS

(1) - Investigar e comparar os efeitos promovidos pelo desgaste feitos com lixas e pontas diamantadas na superfície, transformação de fase e no comportamento mecânico de uma cerâmica Y-TZP.

(2) - Investigar o efeito da LTD (*low-temperature degradation*) na superfície, transformação de fase e no comportamento mecânico de uma cerâmica antes e após o tratamento de superfície (desgaste) Y-TZP.

Para efeitos de apresentação esta Dissertação intitulada **“Efeito do desgaste com instrumentos diamantados e da degradação a baixas temperaturas no comportamento mecânico de uma cerâmica Y-TZP”** foi formatada dividida em dois artigos científicos que serão submetidos à publicação.

ARTIGO 1– EFFECT OF GRINDING WITH DIAMOND-DISC AND –BUR ON THE MECHANICAL BEHAVIOR OF Y-TZP CERAMICS

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Fator de impacto = 2.368; Qualis B1

ARTIGO 2– EFFECT OF LOW-TEMPERATURE DEGRADATION ON THE MECHANICAL BEHAVIOR OF GROUND Y-TZP

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Fator de impacto = 2.834; Qualis A2.

ARTIGO 1 – Effect of grinding with diamond-disc and -bur on the mechanical behavior of a Y-TZP ceramic.

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Running title: Effect of grinding on YTZP

ABSTRACT

This study compared the effects of grinding on the surface micromorphology, phase transformation (t→m), biaxial flexural strength and structural reliability (Weibull analysis) of a Y-TZP (Lava) ceramic using diamond-discs and -burs. 170 discs (15 x 1.2mm) were produced and divided into 5 groups: without treatment (Ctrl, as-sintered), and ground with 4 different systems: extra-fine (25μm, Xfine) and coarse diamond-bur (181μm, Coarse), 600-grit (25μm, D600) and 120-grit diamond-disc (160μm, D120). Grinding with burs was performed using a contra-angle handpiece (T2-Revo R170, Sirona), while for discs (Allied) a Polishing Machine (Ecomet, Buehler) was employed, both under water-cooling. Micromorphological analysis showed distinct patterns generated by grinding with discs and burs, independent of grit size. There was no statistical difference for characteristic strength values (MPa) between smaller grit sizes (D600–1050.08 and Xfine–1171.33), although they presented higher values compared to Ctrl (917.58). For bigger grit sizes, a significant difference was observed (Coarse–1136.32 > D120–727.47). Weibull Modules were statistically similar between the tested groups. Within the limits of this study, from a micromorphological point-of-view, the treatments performed did not generate similar effects, so from a methodological point-of-view, diamond-discs should not be employed to simulate clinical abrasion performed with diamond-burs on Y-TZP ceramics.

Index Words: Grinding, Zirconium oxide partially stabilized by yttrium, Mechanical properties

1. INTRODUCTION

There are a large number of dental materials and ceramic systems currently available for clinical use (Blatz et al 2003). With the increasing demand for aesthetics and the introduction of CAD-CAM technology (computer-assisted design/computer-assisted machining) in the early 80's, several alternatives have been created to substitute conventional feldsphatic ceramic systems with stronger ceramic systems with optimized microstructure (Kamada et al. 1998). Of these new ceramics, zirconia-based ceramics provide the highest mechanical and fatigue properties (Piconi et al. 1999), and therefore are expected to bear greater loads and last longer in the oral environment when compared to the other dental ceramics available in the market. The characteristics of zirconia make this material one of the best options to produce all-ceramic FDPs (fixed dental prosthesis), as it associates good strength with good aesthetics, which are fundamental requirements in the prosthodontics field (Denry et al. 2008).

Zirconia is a polymorphic material that has three crystalline forms. Monoclinic zirconia exists at room temperature and at temperatures up to 1170 °C. Above this temperature, the crystals become tetragonal and show a volume decrease of approximately 4%. Above 2370 °C, zirconia is stabilized in the cubic form. After sintering, a phase transformation from tetragonal to monoclinic (t → m) occurs during the cooling process and is associated with a volume increase of about 3-4%. The stress generated by this expansion can generate cracks in the structure of the ceramic material (Piconi et al. 1999). Therefore, stabilizing oxides (CaO, MgO, CeO_2, Y_2O_3) are added to pure zirconia, keeping the tetragonal form stable at room temperature.

Phase transformation in yttria stabilized zirconia may be triggered by different stimuli, like stress concentration or low temperatures, in the presence of humidity (low temperature degradation - LTD) (Kobayashi et al. 1981; Sato et al. 1985; Papanagiotou

2006; Kosmac et al. 2007; Chevalier et al. 2007). Stress concentration with subsequent phase transformation will occur to dental Y-TZP (Yttrium-stabilized Tetragonal Zirconia Polycrystal) after adjustment of the Y-TZP cementation surface by grinding and/or polishing (Aboushelib et al. 2009).

Grinding the Y-TZP surface after sintering creates a compressive stress layer due to the t→m transformation. However, the same procedure also introduces surface defects. When the depth of the defects introduced is greater than that of the compressive layer, this might produce higher levels of tensile stresses which could increase the incidence of catastrophic failures (Kosmac et al. 2008; Guazzato et al. 2005; Kosmac et al. 1999). Nevertheless, when the depth of these defects is smaller than that of the compressive stress layer, crack propagation is hindered and catastrophic failures are avoided by the surrounding compressive stresses (Papanagiotou 2006; Chevalier et al. 2007).

Polishing/grinding with diamond discs or diamond burs of different grit-sizes might also induce surface damage, like deep scratches and subsurface lateral cracks. These changes are dependent on the grit-size and the load and speed applied during the polishing procedure (Kim et al. 2010; Quinn et al. 2005; Yin et al. 2003; Yin et al. 2006). Greater grit-sizes would create a more significant deleterious impact on the mechanical behavior of Y-TZP ceramics (Kim et al. 2010). In *in vitro* studies, polishing with diamond discs is an easier procedure when compared with polishing with diamond burs, as discs can be attached to polishing machines, allowing for standardized test conditions (i.e. load applied during grinding). However, the use of diamond discs may not represent the clinical situation, since dental prostheses often need to be polished with diamond burs due to the complex geometry of the fixed partial denture. Therefore, comparison of these two grinding methods (discs versus burs) seems to be relevant, as it may validate the use of diamond discs to simulate the effect of diamond burs with compatible grit-sizes.

The present study aimed to compare the polishing/grinding effects of two grinding tools with similar grit-sizes, for methodological purposes. The micromorphology of the surface, the degree of phase transformation (t→m), the biaxial flexural strength and the structural reliability (Weibull analysis) were evaluated with and without grinding procedures. Since the diamond discs and the corresponding burs have similar grit-sizes, it is expected that the effects of both grinding tools on the flexural strength of Y-TZP is similar, and that instruments with larger grit-sizes provide reduced flexural strength when compared to instruments with smaller grit-size.

2. MATERIALS AND METHODS

2.1 Sample Preparation

Disc-shaped specimens (N=170) were manufactured according to ISO 6872 – 2008. Pre sintered blocks of Y-TZP (LOT nº1125100522 - Lava Frame, 3M ESPE, Seefeld, Germany) were ground into cylinders using 600-1200 grit SiC paper (3M, St Paul, MN, USA) under water cooling. The resulting zirconia cylinder was then sectioned using a precision saw machine (ISOMET 1000, Buehler, IL), and slices of 18 mm (Ø) x 1.6 mm (thickness) were obtained. To remove the irregularities inherent to cutting, the disc surfaces were fine ground with 1200 grit SiC paper and cleaned in an ultrasonic bath (1440 D – Odontrobras, Ind. E Com. Equip. Méd. Odonto. LTDA, Ribeirão Preto, Brazil) using isopropyl alcohol 78% for 10 min, and then sintered (Zyrcomat T, Vita Zahnfabrik, Germany) according to the manufacturer (Table 1). The final dimensions of the discs were 15 mm x 1.2 mm (Figure 1).

After sintering, the specimens were carefully selected, those presenting discrepancies in dimension above the recommended deviation (1.2±0.2 mm), preconized by ISO 6872 – 2008, were discarded. The samples (n=30) were divided according to the surface treatment conditions (five levels), as displayed in Table 2.

2.2 Surface Treatment

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

2.2.1 Grinding with Diamond Discs

For abrasion with diamond discs (Dia-Grid Diamond Discs, Allied High Tech Products, Inc. / Rancho Domingues, CA, EUA) of different grit-sizes, samples were embedded in acrylic resin with the treatment surface parallel to the x-axis, to be held for the polishing machine (EcoMet 250, Buehler, Germany). The surface treatments were performed on discs under 60 N load for 10 minutes, with 300 rpm on the machine base (clockwise) and 40 rpm on the head (anti-clockwise) under constant water cooling (≅ 500ml/min)

2.2.2 Grinding with Diamond Burs

Grinding was performed by a single trained operator with diamond burs (#3101G – grit size 181 µm, and #3101FF – grit size 25 µm; KG Sorensen, Cotia, Brazil) coupled to a low-speed motor (Kavo Dental, Biberach, Germany) associated with a contra-angle handpiece (T2 REVO R 170 contra-angle handpiece up to 170.000 rpm, Sirona, Bensheim, Germany) under constant water-cooling (≅ 30ml/min), the diamond bur was replaced after each specimen.

For wear thickness standardization and to ensure that the entire specimen surface was subjected to bur-grinding, the specimens were previously marked with a permanent marking pen (Pilot, Sao Paulo, Brazil) and fixed to a device that assured parallelism between the specimen and diamond bur, allowing movement only in the horizontal direction. The grinding procedure was performed until the pen-marking was completely eliminated.

2.3 Phase analysis by x-ray diffraction

Quantitative analysis of phase transformation was conducted (n=2) 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). Spectra were collected into the 2θ, with a range of 25–35º, at a step interval of 1 s and step size of 0.03º. The amount of m-phase was calculated using the method introduced by Garvie & Nicholson 1972:

$$X_M = \frac{(-111)_M + (+111)_M}{(-111)_M + (111)_M + (101)_T}$$ Eq. (1)

Where: $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(101)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction of the m-phase was calculated according to Toraya et al. in 1984:

$$F_m = \frac{1.311 \cdot X_M}{1 + 0.311 \cdot X_M}$$ 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 described by Kosmac et al. in 1981:

$$PZT = \left(\frac{sen\theta}{2\mu}\right)\left[ln\left(\frac{1}{1 - FM}\right)\right]$$ 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 Eqs. (1) and (2).

2.4 Micromorphological and surface roughness analysis

For the qualitative and quantitative determination of the micromorphological pattern generated by grinding, the specimens were analyzed using a surface roughness tester (n=30, Mitutoyo SJ-410, Japan) and Scanning Electron Microscopy (SEM) (n=2, JSM-6360, JEOL, Japan).

For the roughness analysis, 6 measurements were made for each specimen (3 following the grinding direction, 3 in the opposite direction) according to the ISO 1997 parameters (Ra – arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (µm) and Rz – average distance between the five highest peaks and 5 major valleys found in the standard (µm)) with a cut-off (n=5), λC 0.8mm and λS 2.5µm. After that, the arithmetic mean values of all measurements from each specimen were obtained.

Prior to the micromorphological analysis, all specimens were submitted to the cleaning protocol in an ultrasonic bath, as described above.

2.5 Biaxial flexure test

Samples were subjected to a biaxial flexure strength test according to ISO 6872-2008. Disc-shaped specimens were positioned with the treated surface turned down (tensile stress) on three support balls (Ø=3.2mm) that were positioned 10 mm apart from each other in a triangular pattern. The assembly was immersed in water, and a flat circular tungsten piston (Ø=1.6mm) was used to apply an increasing load (1mm/min) until catastrophic failure in a universal testing machine (EMIC DL 2000, Sao Jose dos Pinhais, Brazil). Before testing, adhesive tape was fixed on the compression side of the discs to avoid spreading the fragments (Quinn 2007) and to provide better contact between the piston and the sample (Wachtman et al. 1972). Flexural strength was calculated according to ISO 6872 - 2008:

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

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

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

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

where ν is Poisson's ratio (according to Borba et al 2011 = 0.32), 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).

2.6 Data analyses

Descriptive analysis was carried out to determine mean and standard deviations of the biaxial flexural strength data (Table 3), Pearson Correlation between the roughness data (Ra) and biaxial flexural data also was performed.

Considering that the failure of ceramic materials originates from the most severe defect, the size and spatial distribution of defects justify the need for a statistical approach (Weibull 1951; Della Bona 2005). Thus, the statistic used to describe the reliability of the ceramic material was based on the Weibull statistical analysis, which is a way to describe the variation of the resistance (Tinschert et al. 2000; Della Bona et al. 2003), obtaining the Weibull modulus (m) and the characteristic strength (σ_c) with a confidence interval of 95%, determined in a lnσc - ln[ln 1/(1-F(σc)] diagram (according to ENV 843-5):

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

where F is the failure probability, σ_0 is the initial strength, σ_c is 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, which expresses the reliability of the material.

3. RESULTS

SEM micromorphological analysis showed that grinding with a diamond bur (Xfine and Coarse) resulted in similar surface patterns, with scratches parallel to the direction of grinding tool propagation and a depth proportional to the grit size of the bur used. The surface treatment carried out with diamond discs (D600 and D120) showed a distinct micromorphological pattern, with a smoother surface, the presence of surface scratches in all directions and lateral projections, with a depth also directly related to the grain size of the disc employed (Figure 2, Table 3).

X-ray diffraction showed that, regardless of the surface treatment used, an increase in the grit size of the grinding tool increased the percentage of m-phase content on the material's surface. Another important observation was that, for larger grit sizes, the use of diamond discs resulted in larger amounts of m-phase while, for smaller grit sizes, grinding with a diamond bur resulted in larger amounts of m-phase (Table 3).

The Weibull analysis of the strength data is shown in Table 4. The characteristic strength values obtained for specimens treated by the grinding tools with smaller grit sizes (Xfine and D600) were statistically similar and significantly higher that what was obtained for the control group. On the other hand, higher $\boldsymbol{\sigma_c}$ values were observed for the Coarse group when compared to D120. In comparison to the control group, the Coarse group obtained a $\boldsymbol{\sigma_c}$ value that was statistically superior while D120 showed values that were statistically lower. In relation to the reliability (Weibull modulus - *m* value), neither grinding with diamond burs nor diamond discs was able to significantly reduce the material reliability.

4. DISCUSSION

The main purpose of this current study was to analyze and compare the polishing/grinding effects of two grinding tools with similar grit-sizes, for methodological purposes on the mechanical behavior of one Y-TZP. The present findings indicated that grinding with diamond burs, regardless of grit size, and with diamond discs of smaller grit sizes, triggers the toughening mechanism of phase transformation on zirconia, leading to increased flexural strengths. The collected data also showed that grinding with diamond discs of a larger grit size resulted in a degradation of the Y-TZP mechanical properties when compared to the control.

The micromorphological differences created by the different surface treatments may be explained by the differences observed in the grinding tool mechanisms (diamond discs *versus* diamond burs). The Xfine and Coarse groups were subjected to a treatment in which the abrasion process occurred only in one direction (horizontally in relation the support base movement), generating parallel scratches. On the other hand, groups D120 and D600 were submitted to a rotational abrasion movement, with the support base and head moving in opposite directions (clockwise and anti-clockwise, respectively) in the polishing machine. This type of grinding movement assured a smoother surface, although it was still possible to note scratches in all directions and lateral projections.

It is important to note that after clinical adjustment of restorations (grinding procedures) prosthesis of zirconia will be exposed to a hazardous environment and these could implicate in an increased susceptibility to suffer LTD (low temperature degradation) (Kim et al. 2010). According to Kobayashi et al. 1981, this type of degradation occurs spontaneously on the Y-TZP surface when the material is subjected to an environment with high humidity and temperature variations. LTD takes place first inside surface grains, where water is incorporated into the zirconia by filling oxygen vacancies (Sato et al. 1985; Yoshimura et al. 1987), spreading to the rest of the material surface and increasing the surface roughness. After saturation of the surface with the m-phase, LTD proceeds into the bulk of the material (Yoshimura et al.1987), leading to a reduction in strength, fracture toughness, and density of zirconia (Hirano 1992; Kim et al. 2009; Lughi & Sergo 2010).

Some studies have shown that the transformation from tetragonal to monoclinic is triggered on areas surrounding surface defects generated by grinding (sides and corners of the exposed grains) (Deville et al. 2006). In these spots, residual tensile stress concentration occurs (Schmauder & Schubert 1986), but the crystal grain does not transform at once, since the transformation process takes place progressively with an increase in water attack, leading to increased stress fields (Lilley 1990; Deville et al. 2004). This type of phase transformation by nucleation and grain growth mechanisms (Chevalier et al. 2007) also leads to crack formation with a subsequent detachment of surface grains, increasing surface roughness and the stress concentration zones, which in turn results in degradation of the mechanical properties and an increase of areas that facilitate water access and the occurrence of further degradation (Schmauder & Schubert 1986; Lilley 1990; Deviller et al. 2004; Griffith 1921).

X-ray diffraction data showed an increase in the monoclinic phase content as the granulation of the grinding tool increased, indicating that the formation of a compressive layer responsible for the zirconia toughening mechanism is directly related to the grain size of the grinding tool, as observed previously (Kosmac et al. 2007; Kosmac et al. 2008; Guazzato et al. 2005; Kosmac et al. 1999; Kim et al. 2010).

As the transformation depth increases towards the bulk of the material in response to a more aggressive surface treatment (increase in grit size or a deficient water-cooling), degradation of the mechanical properties occurs. This increase in depth of the t→m transformation is related to the presence of cracks in deep areas that can be deeper than the compressive stress layer formed, overcoming the benefits obtained by the toughening mechanism (Chevalier et al. 2007; Hirano 1992). According to Griffith 1921, the presence of defects within a material increases the probability of a critical defect acting as a stress concentrator, leading to the catastrophic failure of a material. This may explain the decrease in characteristic strength observed for D120 (727.47 MPa, with 18.75% of m-phase content and 1.05µm transformation depth), when compared to the other groups (Tables 3 and 4).

Roughness data showed that grinding with diamond burs always generated the highest Ra values. However, this higher roughness did not negatively affect the material strength. In fact, grinding with diamond burs resulted in characteristic strength values that were higher than those obtained for the control group (Ctrl), demonstrating that, especially for zirconia based ceramics, a high flexural strength mean value is not a function of a more regular surface. This observation also emphasizes the importance of the t→m transformation as a toughening mechanism in Y-TZP.

These current findings did not corroborate those of Kosmac et al. in 1999, 2007, 2008, 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. Those authors used a high-speed handpiece for grinding, which 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. In the present study, a low-speed handpiece was used, in association with a torque multiplier, resulting in a less aggressive grinding procedure. It is also important to note that the grinding procedure was carried out under constant and abundant water-cooling.

The calculated Pearson linear correlation coefficient between the current strength and roughness data indicated that a very weak correlation exists for the D120 and Xfine groups (0 < (r) < 0.3), and a weak correlation is noticed for the Ctrl, D600 and Coarse groups (0.3 < (r) < 0.6) (Crespo 1997). According to Quinn 2007, the presence of correlation between strength and roughness is commonly observed as the absence of correlation, since grinding introduces deeper cracks in the material (10-20x) than the existing surface flaws. In some cases, the depth of the introduced cracks is similar to that of the existing surface flaws and, therefore, a correlation would be expected. However, when the introduced cracks are deeper than the existing surface flaws, a stronger correlation is noted between roughness and strength.

The hypothesis that larger grit sizes would exhibit a deleterious effect was partially accepted, since larger grit sizes of the diamond disc (D120) led to lower characteristic strength values. However, the same observation was not true for diamond burs. Therefore, considering the same grit sizes, grinding with diamond discs is a more aggressive treatment when compared to treatment with diamond burs.

Some studies (De Munck et al. 2013; Studart et al. 2007) that evaluated the flexural strength of dental ceramics preferred to show and discuss the 5-10% failure probability instead of the usual 63.2% level, namely the characteristic strength of the Weibull analysis. The 5-10% failure probability level (Table 4) is considered more clinically relevant, since it is related to a safer level of reliability of the material for clinical use. Literature shows that from the clinical point of view, maximum masticatory forces may easily achieve 300-400N and far reduced average chewing forces of approx. 220N in the molar region (Proschel et al. 2002; Hidaka et al. 1999). Assigning those forces to a contact area of 7-8mm^2 (single molar tooth) result in an average chewing pressure of 27-31MPa (Lohbauer et al. 2008). Thus, it is noted that all surface treatments evaluated in the current study resulted in characteristic strength values and 5% failure probability values significantly higher than the stress levels generated by clinically relevant forces.

Concerning Weibull modulus (also referred to as the material reliability) literature shows values ranging from 5 to 15 for current dental ceramics (Papanagiotou 2006; Guazzato et al. 2005; Kosmac et al. 1999; Della Bona 2003). In this present study, *m* values ranged from 4.3 to 13.5, but no statistical difference was observed, demonstrating that none of the surface treatments caused degradation of the structural reliability of the material. According to Quinn GD et al. 1991 and Quinn JB. et al. 2009, higher *m* values correspond to materials with a uniform distribution of highly homogeneous flaws with a narrower strength distribution, whereas lower *m* values indicate non-uniform distribution of highly variable crack lengths (broad strength distribution). Thus, if one treatment promotes higher *m* values, it could be considered a good choice for clinical use, even if it has a lower characteristic strength, since the increase in the *m* value represents a more uniform distribution of defects on the material surface.

One limitation of this current study was that no kind of fatigue was simulated with the tested specimens. Additionally, low-temperature degradation was not investigated as a function of the surface treatment performed. Therefore, future studies should be carried out to subject zirconia to LTD and cyclic loading to better understand how these distinct micromorphologic patterns will behave under these detrimental conditions, since aging methods could accentuate the differences observed in the present study.

5. CONCLUSIONS

Under the conditions of the present investigation, grinding of a Y-TZP ceramic with small grit-size tools did not degrade the mechanical properties of the material. From a methodological point of view, diamond discs should not be employed to simulate clinical abrasion performed with diamond burs on Y-TZP ceramics. From a micromorphological point of view, the treatments performed did not generate similar effects.

6. ACKNOWLEDGEMENTS

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The authors claim no conflict of interest.

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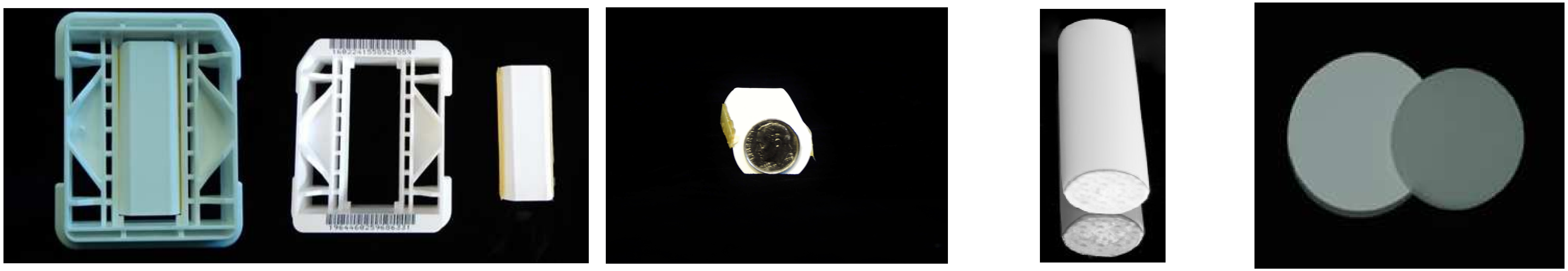
FIGURES



A B C D

Figure 1. A) Pre sintered blocks as provided by manufacturer (3M Espe, USA); B) Device used to standardize the size of the cylinder in 18 mm, attached to the ends of the block; C) Round ground of the blocks; D) Samples after section of the cylinder (left) and after sintering (right).

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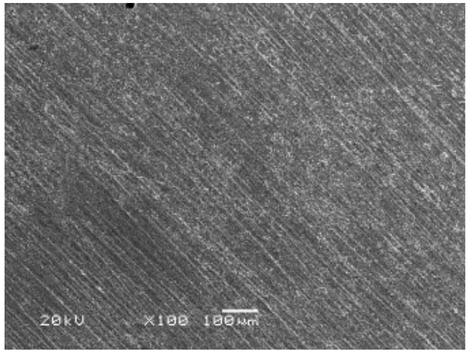
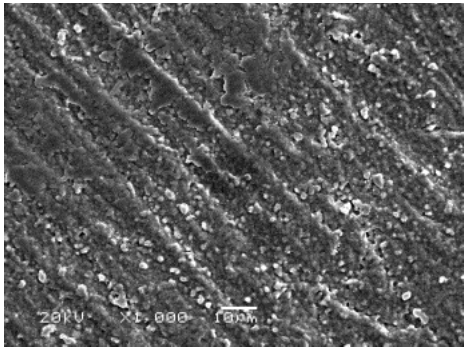
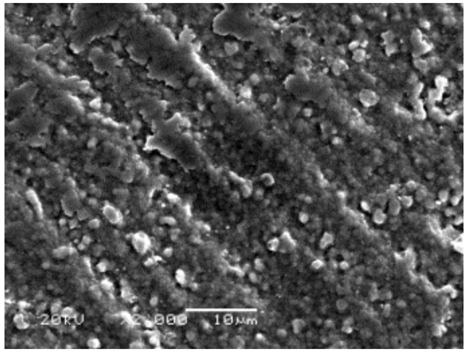
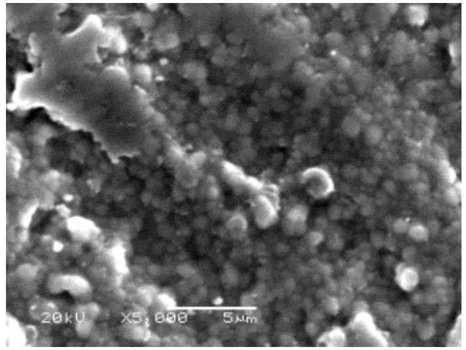
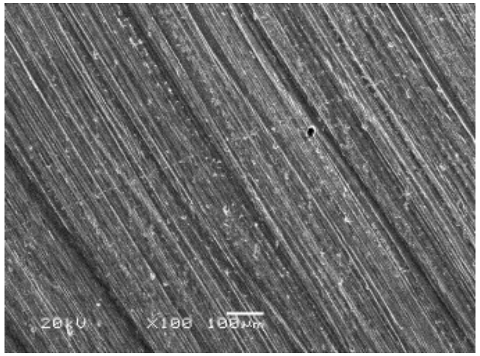
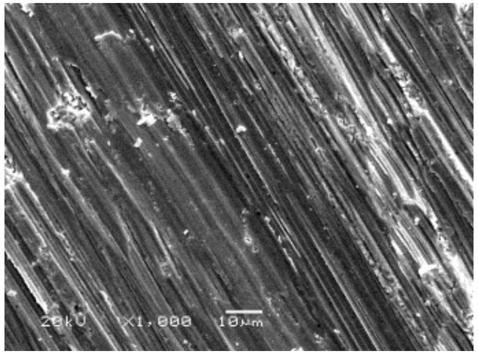
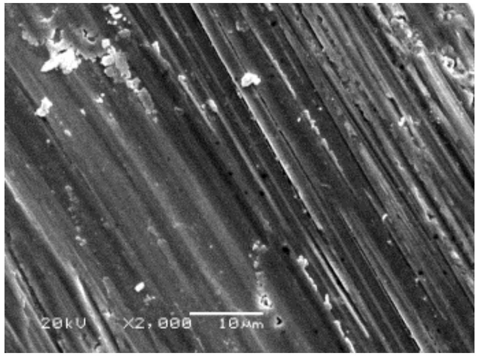
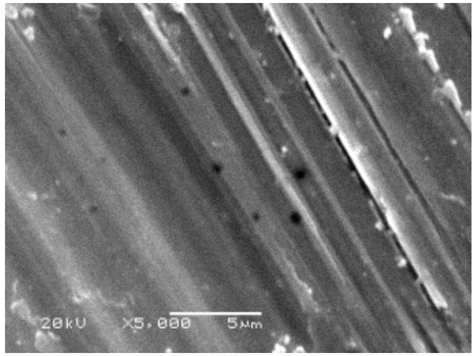
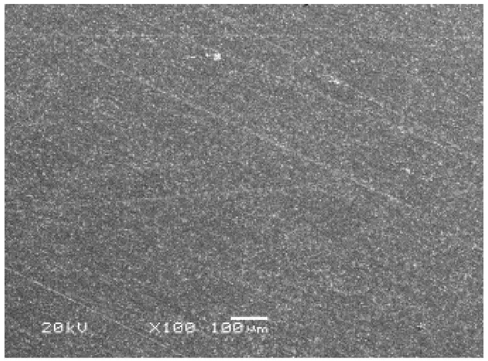
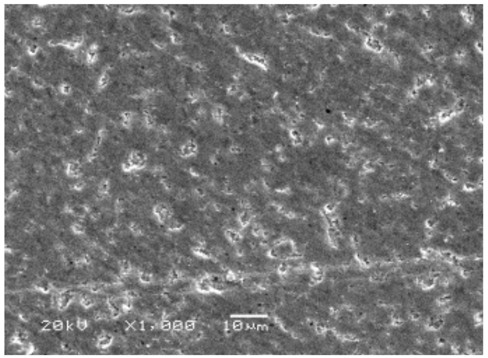
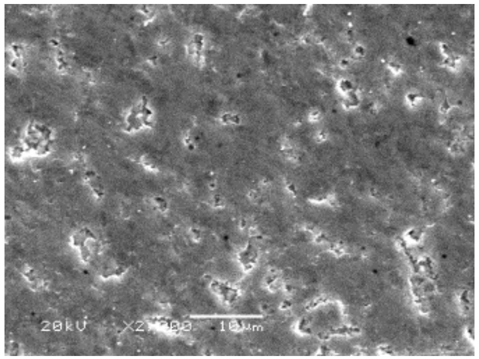
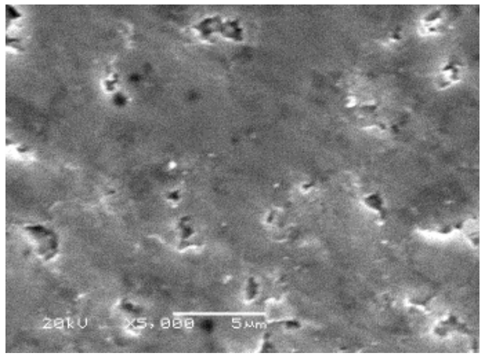
Ctrl group				
Xfine group				
D600 group				

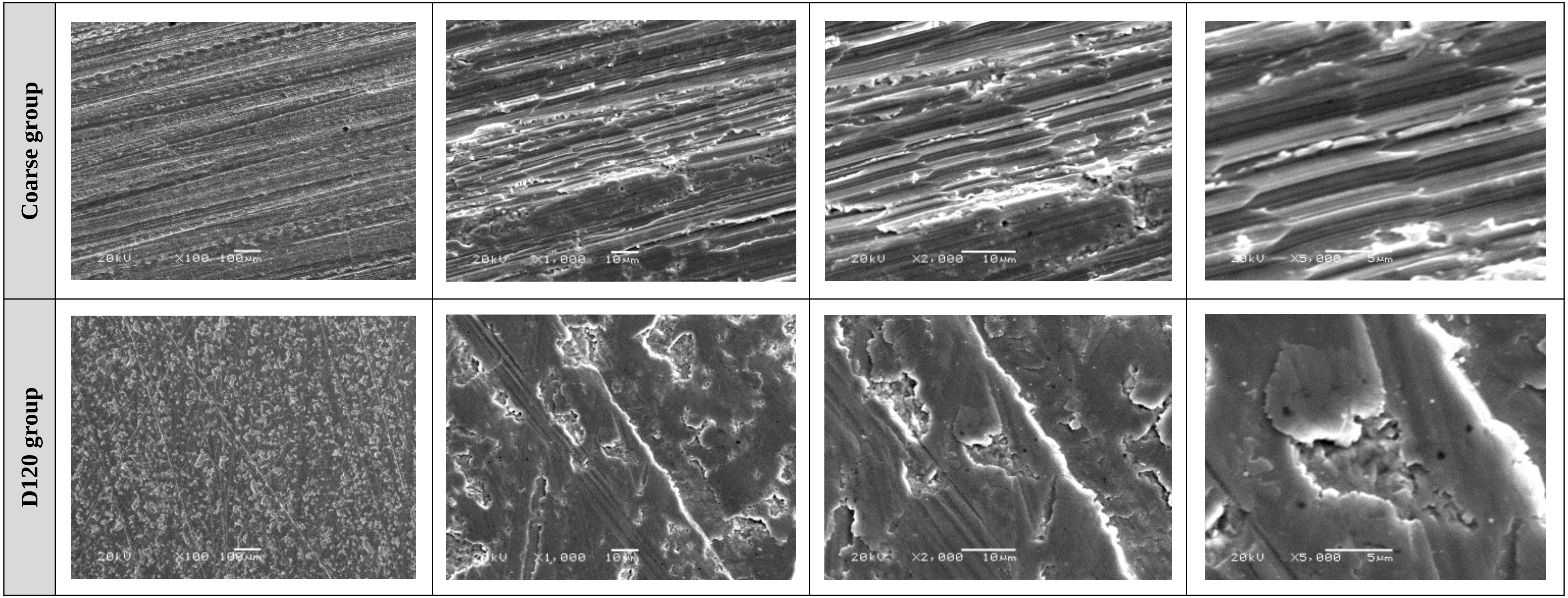


Figure 2 – Micrographics obtained trough Scanning Electron Microscopy of tested groups (100x, 1000x, 2000x and 5000x of magnification)

TABLES

Table 1 – Sintering cycle of Y-TZP (Lava, 3M ESPE, EUA).

Ceramic	Initial temperature	Heating rate	Holding temperature	Holding time
LAVA	40 °C	17 °C/min	1530 °C	120 min

Table 2 – Experimental Design

Group	N	Surface treatment	Material (manufacturer)
Ctrl	34	As-sintered (Without treatment, control)	--------------
D120	34	Diamond disc 120 (average grit size 160 µm)	Dia-Grid Diamond Discs (Allied High Tech Products, Inc. / Rancho Domingues, CA, EUA)
D600	34	Diamond disc 600 (average grit size 25 µm)	
Coarse	34	Coarse Diamond Bur #3101G (average grit size 181 µm)	KG Sorensen, Cotia, Brazil
Xfine	34	Extra-fine Diamond Bur #3101FF (average grit size 25 µm)	

Table 3 – X-ray Difractometry analysis (% of monoclinic Phase, Depth of Transformed Layer), Roughness analysis (Parameters Ra and Rz), descriptive statistical analysis (Mean and Standard Deviation) and Pearson linear correlation analysis (coefficient of linear correlation) between roughness (Ra) and mean strength.

Groups	Monoclinic Phase (%)	Depth of transformed layer (µm)	Roughness Ra (µm)	Roughness Rz (µm)	Mean (σ_c) (MPa)	Standard Deviation (MPa)	Coefficient of linear correlation – r (Ra x σ_c)
CTRL	0.00	0.00	0.67	4.43	865.90	126.10	-0.34 (p=0.07)
XFINE	9.00	0.48	0.86	5.55	1085.80	179.70	-0.07 (p=0.7)
D600	7.16	0.38	0.09	0.68	1001.30	108.60	0.35 (p=0.06)
COARSE	12.78	0.69	1.41	8.43	1076.80	134.90	-0.51 (p=0.003)
D120	18.75	1.05	0.36	2.82	691.10	86.60	-0.17 (p=0.37)

Table 4 – Characteristic Strength (σ_c), Weibull modulus (*m*) and respective Confidence Intervals (CI - 95%).

Groups	σ_c	CI (95%)	*m*	CI (95%)	$\sigma_{0,5}$
CTRL	917.58[a]	870.86 – 965.40	8.33[a]	5.83 – 10.70	642.26
XFINE	1171.33[b]	1091.39 – 1254.68	6.15[a]	4.31 – 7.91	722.88
D600	1050.08[b]	1007.42 – 1093.29	10.5[a]	7.34 – 13.48	791.13
COARSE	1136.32[b]	1083.58 – 1190.07	9.15[a]	6.41 – 11.76	821.44
D120	727.47[c]	695.52 – 759.95	9.69[a]	6.78 – 12.45	535.36

***same letters correspond to statistical similarity**
***different letters correspond to statistical difference**

ARTIGO 2 – Effect of low-temperature degradation on the mechanical behavior of ground Y-TZP

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Running title: Effect of grinding and LTD on YTZP

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ABSTRACT

This study evaluated the effects of low-temperature degradation (LTD) on the surface topography, phase transformation, biaxial flexural strength and structural reliability of a Y-TZP ceramic after grinding. Disc-shaped specimens were manufactured and divided according to two factors: “surface treatment”- without treatment (as-sintered, Ctrl), grinding with extra-fine diamond bur (25µm; Xfine) and coarse diamond bur (181µm; Coarse), and “LTD” (absence or presence). Grinding was performed using a contra-angle handpiece under water-cooling. LTD was simulated in an autoclave at 134ºC, under 2 bar, over a period of 20 h. Surface topography analysis showed an increase in roughness based on surface treatment grit-size (Coarse>Xfine>Ctrl) and that LTD promoted different effects on roughness (Ctrl LTD<Ctrl; Xfine LTD<Xfine and Coarse LTD>Coarse). Grinding and LTD promoted an increase in the amount of m-phase, although different susceptibilities to degradation were observed. Weibull analysis showed an increase in characteristic strength after grinding (Coarse=Xfine>Ctrl); however, distinct effects were observed for LTD (Ctrl<Ctrl LTD; Xfine=Xfine LTD and Coarse>Coarse LTD). Weibull moduli were statistically similar. Within the limits of this current study, both LTD and grinding showed to not be detrimental to the mechanical properties of zirconia. However, when both factors are associated, the defects introduced by grinding may be detrimental to the resistance of the material.

Index Words: Grinding, Low temperature degradation, Zirconium oxide partially stabilized by yttrium, Mechanical properties, Structural reliability.

INTRODUCTION

Due to the current high aesthetic requirements of patients and the continuous search for materials with appropriate mechanical properties, numerous studies have been performed on metal-free ceramic restorations. These restorations combine the aesthetic properties of veneering porcelain with the high strength of an Yttrium-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) infrastructure[1].

Y-TZP stands out among other restorative materials due to its high chemical and dimensional stability and superior mechanical properties[2]. Due to the poor metastability of zirconia crystals, yttria (3% mol) is added to pure zirconia to stabilize the tetragonal phase at room temperature[2]; consequently, the volume expansion (around 3%) that takes place when crystals transform from the tetragonal to monoclinic phases[3,4] is prevented. The *t→m phase* transformation will eventually happen after applying local stress[5,6,7] and in the presence of water[6,7,8,9], which is known as aging or low-temperature degradation (LTD)[10].

Although current CAD/CAM (computer-aided design and manufacturing) systems result in ceramic prosthetic crowns with very good marginal and internal fit, clinicians may need to adjust the intaglio surface of Y-TZP frameworks by grinding to achieve the best outcome possible in terms of crown adaptation[11]. Grinding the Y-TZP surface introduces different types of damage, like scratches and cracks of various depths, which penetrate towards the bulk of the material[7,12,13].

The *t→m* transformation, associated with localized expansion, results in compressive stresses at an existing crack, which counteracts tensile stresses in this region[4]. This phenomenon is called transformation toughening and limits crack propagation. If the existing tensile stress at the crack tip surpasses the compression stresses generated by transformation toughening, catastrophic crack propagation may still occur[2,4].

The current literature shows that grinding the Y-TZP surface with a diamond bur will trigger transformation toughening, while also introducing important flaws on the ceramic surface. The balance between the tensile stress around these defects and the compression stress generated by phase transformation will determine whether or not cracks propagate, leading to catastrophic failure[4,14,15]. Another important aspect is that grinding with diamond burs can lead to a temperature increase at the Y-TZP surface, consequently triggering a reverse $m \rightarrow t$ transformation that works against transformation toughening[4,15].

Kobayashi K, et al. 1981[10] observed that, when Y-TZP is submitted to an environment with high humidity and temperatures between 150-400ºC, it spontaneously suffers a degradation process known as low-temperature degradation (LTD). LTD initially occurs at superficial grains, where water is incorporated into zirconia grains by filling oxygen vacancies, and later spreads to the surface, thus increasing surface roughness[16,17]. After the surface is saturated with *m*-phase, LTD proceeds into the bulk material[17], leading to a reduction in the strength, fracture toughness, and density of Y-TZP products[8,18,19].

Since phase transformation of Y-TZP crystals takes place in the presence of water or steam, steam autoclave treatments at increased temperatures (120–140ºC) have been used to effectively induce LTD[6,7,8,20,21,22]. According to Kim et al. 2009[9], a LTD simulation method using steam autoclave displays a strain-induced transformation ($t \rightarrow m$) that can induce positive or negative effects on the mechanical properties of Y-TZP ceramics, depending on the applied temperature and the amount of resulting *m*-phase.

The current literature shows that both LTD[4,9,23] and surface treatments[6,8,9] on zirconia can influence the mechanical strength. Also, when LTD occurs after Y-TZP surface alterations, different final amounts of *m*-phase are detected on the surface of the ceramic[9,22]. However, the influence of surface alteration associated with LTD on the mechanical strength of Y-TZP has not been explored.

Therefore, the aim of this present study was to investigate the effects of grinding using diamond burs with different grit sizes on the mechanical behavior, surface topography and phase transformation of a Y-TZP ceramic, with and without LTD. The following hypotheses were tested: (1) aged surfaces are relatively rougher than non-aged surfaces; (2) grinding associated with LTD leads to an increase in m-phase content; (3) grinding associated with LTD leads to an alteration in the Y-TZP flexural strength.

MATERIALS AND METHODS

Sample Preparation

Disc-shaped specimens (N=204) were manufactured according to ISO 6872-2008[24]. Pre-sintered blocks of Y-TZP (LOT nº1125100522 - Lava Frame, 3M ESPE, Seefeld, Germany) were shaped into a cylinder using 600-1200 grit SiC paper (3M, St Paul, MN, USA) under water cooling, than sectioned using a precision saw (ISOMET 1000, Buehler, IL) in 18 mm diameter slices that were 1.6 mm thick. To remove irregularities inherent with specimen preparation, the surfaces were fine ground with 1200 grit SiC paper and cleaned in an ultrasonic bath (1440 D – Odontrobras, Ind. E Com. Equip. Méd. Odonto. LTDA, Ribeirão Preto, Brazil) using 78% isopropyl alcohol 10 min. The specimens were sintered (Zyrcomat T, Vita Zahnfabrik, Germany) according to the manufacturer’s instructions (1530°C, holding time: 120min), obtaining discs with final dimensions of approximately 15 mm x 1.2 mm[6].

After sintering, the specimens were carefully selected. Specimens presenting discrepancies in length above the standard variation (1.2 ± 0.2 mm) were discarded. Then, samples (n = 34) were divided according to the surface treatment conditions (three levels) and LTD (two levels) as displayed in Table 1.

Surface Treatment

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

Grinding

Grinding was performed by a single trained operator using diamond burs (#3101G – grit size 181 µm, and #3101FF – grit size 25 µm; KG Sorensen, Cotia, Brasil) in a slow-speed motor (Kavo Dental, Biberach, Germany) associated with a contra-angle handpiece (T2 REVO R170 contra-angle handpiece up to 170,000rpm, Sirona, Bensheim, Germany) under constant water-cooling (≅ 30ml/min). Each diamond bur was replaced after each specimen.

For standardization of the wear thickness and to guarantee that the entire specimen surface was submitted to the surface treatment, the specimens were marked with a permanent marking pen (Pilot, São Paulo, Brazil) and were fixed to a device to assure parallelism between the specimen and diamond bur, which allowed movement only in the horizontal direction. Then, the grinding procedure was performed until the marking was completely eliminated. This procedure standardized the wear thickness and improved the reproducibility of the grinding treatment, although this strong control of the direction of treatment is not available in a clinical typical setting.

Low Temperature Degradation – LTD

Low Temperature Degradation was simulated in an autoclave (Sercon HS1-0300 nº1560389/1) at 134ºC, under 2 bar, over a period of 20h.

Phase analysis

Quantitative analysis of phase transformation was conducted (n=2) 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). Spectra were collected in the 2θ range of 25–35º at a step interval of 1 s and step size of 0.03º. The amount of *m*-phase was calculated using the method developed by Garvie & Nicholson 1972[3]:

$$X_M = \frac{(-111)_M + (+111)_M}{(-111)_M + (111)_M + (101)_T}$$

Eq. (1)

Where: $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(101)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction of the m-phase was calculated according to Toraya et al. 1984[25]:

$$F_m = \frac{1.311 \cdot X_M}{1 + 0.311 \cdot X_M}$$

Eq. (2)

The depth of the transformed layer was calculated on the basis of the amount of the *m*-phase, considering that a constant fraction of grains had symmetrically transformed to *m*-phase along the surface, as described by Kosmac et al. 1981[26]:

$$PZT = \left(\frac{sen\theta}{2\mu}\right)\left[ln\left(\frac{1}{1-FM}\right)\right]$$

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 Eqs. (1) and (2).

Surface topography and roughness analysis

For the qualitative and quantitative determination of the surface topography pattern generated by grinding, the specimens were analyzed in a surface roughness tester (n = 30, Mitutoyo SJ-410, Japão) and Scanning Electron Microscope (SEM) (n = 2, JSM-6360, JEOL, Japan).

For surface roughness analysis, 6 measurements were made for each specimen (3 along the grinding direction, 3 in the opposite direction) according to the ISO 1997 parameters (Ra - arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (µm) and Rz - average distance between the five highest peaks and 5 major valleys found in the standard (µm)) with a cut-off (n = 5), λC 0.8 mm and λS 2.5 µm. After that, arithmetic mean values of all measurements from each specimen were obtained.

Prior to the surface topography analysis, all specimens were submitted to the cleaning protocol in an ultrasonic bath as described previously.

Biaxial flexure test

Samples (n = 30) were subjected to a biaxial flexure strength test according to ISO 6872[24]. Disc-shaped specimens were positioned with the treated surface facing down (tensile stress) on three support balls (Ø = 3.2 mm) which were positioned 10 mm apart from each other in a triangular position. The assembly 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 using a universal testing machine (EMIC DL 2000, Sao Jose dos Pinhais, Brazil). Before testing, adhesive tape was fixed on the compression side of the discs to avoid spreading the fragments[27] and to provide better contact between the piston and sample[28]. Flexural strength was calculated according to[24]:

$$\sigma = -0.2387 \cdot \frac{P(X-Y)}{b^2}$$

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:

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

Eq. (5)

$$Y = (1+\nu)\left[1 + ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-\nu)\left(\frac{r_1}{r_3}\right)^2$$

Eq. (6)

where ν is Poisson's ratio (according to Borba et al 2011[29] = 0.32), 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 analyses

Descriptive analysis was carried out to determine mean and standard deviations of the biaxial flexural strength data (Table 2). Pearson Correlation between the roughness data (Ra) and biaxial flexural data was also performed. Two-way ANOVA and post-hoc Tukey`s t test of the roughness data (Ra and Rz) were performed considering 2 factors (surface treatment and aging – LTD) and the interaction of both factors.

Considering that the failure on ceramic materials originates from the most severe defect, the size and spatial distribution of defects justify the need for a statistical approach[30]. Thus, the statistic used to describe the reliability of the ceramic material was based on the Weibull statistical analysis, which is a way to describe the variation of the resistance[31,32] obtaining the Weibull modulus (*m*) and the characteristic strength (σ_c) with a confidence interval of 95%, determined in a diagram (according to ENV 843-5):

$$\ln \ln\left(\frac{1}{1-\mathrm{F}}\right) = m \ln \sigma_c - m \ln \sigma_0$$

Eq. (7)

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 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 the reliability of the material.

Failure analysis

Fractography examination was performed using a light microscope (Stereo Discovery V20; Carl Zeiss, São Paulo, Brazil) and Scanning Electron Microscope (JSM-6360, JEOL, Japan) on a representative part of the specimens.

RESULTS

SEM micrographs show that grinding with diamond burs (Xfine and Coarse) created the same surface pattern, regardless of grit-size, with the presence of parallel scratches following the direction of bur movement, while LTD did not cause a relevant alteration of this pattern (Figure 1, Table 2).

Roughness data (Ra and Rz) indicated that there was an increase in roughness ($p<0.05$) as a function of the bur grit size (Table 2). Aging (LTD) significantly affected the roughness of all groups. While the roughness of the Ctrl and Xfine groups significantly decreased after LTD, the roughness values increased for the Coarse group (Table 2).

X-ray diffraction showed that the greater bur grit sizes yielded a greater amount of *m*-phase detected on the materials surface. This analysis also showed that aging increased the monoclinic content for all groups, although in distinct intensities, as the Xfine LTD group and the Coarse LTD group presented lower *m*-phase content (15.75% and 28.75%, respectively) and transformation depth (0.87 µm and 1.72 µm, respectively) when compared to the Ctrl (53.33% and 3.86 µm), although the same aging protocol was performed (20 hours, 134ºC, 2 bar).

Weibull statistical analysis shown a significant increase in characteristic strength after submitting the specimens to the grinding procedure (Xfine=Coarse>Ctrl). LTD promoted distinct effects on all groups, while for Ctrl it presented statistically higher $\boldsymbol{\sigma_c}$ values after aging, lower $\boldsymbol{\sigma_c}$ values were observed for Coarse, but were still statistically similar to the Ctrl without LTD (Coarse>Coarse LTD=Ctrl). For the Xfine groups, LTD did not promote any statistical change in $\boldsymbol{\sigma_c}$ values. Regarding the reliability (*m* value), neither grinding nor LTD were able to reduce the Weibull modulus of the Y-TZP tested (Table 2 and Figure 2).

Figure 3 shows 6 representative fracture surfaces, from the Coarse, Ctrl and Ctrl LTD groups. In all cases, the initial defect is clearly observed on the tensile surface of the material (Figure 3), probably generated during processing of the sample (Ctrl and Ctrl LTD groups) or due to the scratches created by grinding (Coarse group) (Quinn 2007)[27].

DISCUSSION

The main purpose of this current study was to analyze the effect of LTD on the surface topography and mechanical strength of a Y-TZP ceramic after grinding with diamond burs. The present study showed that grinding initially triggered transformation toughening on the Y-TZP surface, leading to higher $\boldsymbol{\sigma_c}$ values, and that LTD promoted distinct effects on the mechanical behavior varying accordingly to the surface pattern generated by the grinding procedure. It is important to note that, although a high rate of $t \rightarrow m$ transformation was observed in all groups, degradation of the mechanical properties were observed only for the Coarse LTD group, which could indicate that a high rate of m-phase content is not the only important aspect for predicting the mechanical behavior of the material, while other aspects like topography and roughness should be taken into consideration.

Several studies have been performed to understand the effects of exposing the Y-TZP ceramic to low temperatures in the presence of water over long time periods[6,7,20,21,22,23]. These studies showed that these procedures led to different amounts of *t*→*m* phase transformation on the zirconia surface, but few of them evaluated the mechanical strength of the ceramic after LTD, and most importantly, the interaction between this phenomenon with the surfaces that were ground with diamond burs used in dental offices[9,21].

According to Chevalier et al. 2004[33], the amount of *m*-phase is strongly dependent on the temperature and dwell time: a higher temperature and longer dwell time tend to produce a higher transformation rate[9,21,23]. Transformation rate could also be related to grain size: larger tetragonal grain size typically provide for lower phase stability[22,34]. Lava™ ceramic exhibited a large grain size, as already reported[22]; thus, there is a greater possibility of phase transformation for that material[33,35]. Furthermore, residual stress caused by zirconia surface treatments is also a determining factor for the susceptibility of Y-TZP ceramics to LTD[36].

The current literature[16,17] shows that the *t*→*m* phase transformation initially occurs at superficial grains and later spreads to the bulk of the material; as a consequence of this phase transformation, superficial grains may be lost, increasing the surface roughness.

In the present study, SEM micrographs and roughness analysis showed that the Xfine and Ctrl groups presented a more regular surface when compared to the Coarse group, a fact that may indicate poor water accessibility in the deeper layers of the material, making it harder for LTD to occur. Consequently, *t*→*m* phase transformation likely started at more superficial areas, such as with surface grains located at topographic peaks, promoting debonding of those grains and unexpectedly generating an even more regular surface (reduction in mean Ra and Rz roughness values) .

On the other hand, the Coarse group obtained highest Ra and Rz roughness values when compared to the other groups, both before and after LTD. Since the grit size of the bur is larger, a more irregular surface was produced, with deeper defects and increased distance between peaks, giving greater accessibility to water into the Y-TZP structure, favoring the LTD mechanism and enhancing the surface roughness due to the detachment of surface grains[16,17].

There are conflicting results in the literature regarding the effect of LTD on the mechanical properties of materials. Kim et al 2009[9] showed that flexural strength did not decrease when 54% *m*-phase was detected. On the other hand, Ban et al. 2008[8] noticed a decrease in strength with a 50% *m*-phase on the zirconia surface. These differences may be explained by a study by Cattani-Lorente et al. 2011[23], who noticed that the amount of *m*-phase remained unaltered during additional aging, even when the transformation progressed into the bulk of the material. It is important to note that none of these studies considered the surface topography and roughness of the material. The current study demonstrates that these are important aspects for predicting the mechanical behavior and an increase or decrease in the susceptibility of the material to LTD.

The current data indicates that a rougher surface will potentiate the effects of aging (LTD), consequently leading to a degradation of the mechanical properties and increasing the risk of a catastrophic failure, as observed for the Coarse group, which presented a statistical decrease in $\boldsymbol{\sigma_c}$ values after LTD. On the other hand, a smoother surface will hinder the LTD process, decreasing the risks of catastrophic failure, as noted in the Ctrl and Xfine groups, which did not present a statistical decrease for $\boldsymbol{\sigma_c}$ values after LTD.

The Ctrl group exhibited a significant increase in $\boldsymbol{\sigma_c}$ after LTD, probably due to an increased *m*-phase content leading to transformation at deeper layers (Table 2), influencing the magnitude of the increase in toughness[26]. The increased volume due to the *t*→*m* transformation lead to the accumulation of compressive stress, which probably compensated for the tensile stresses exerted during the flexural test[37,38,39]. For the Xfine and Coarse groups, distinct behaviors were noticed, even though they presented a significant increase in *m*-phase after LTD. Both groups presented a lower m-phase content when compared to Ctrl LTD. Furthermore, while the Xfine group maintained statistically similar $\boldsymbol{\sigma_c}$ values before and after LTD, the Coarse group presented a degradation of mechanical properties and presented a decrease in $\boldsymbol{\sigma_c}$ values after LTD.

Grinding promoted the formation of a tension barrier (Ctrl – 0% *m-phase* and 0 µm depth of transformed layer, Xfine – 9% and 0.48 µm, Coarse – 12.78% and 0.69 µm), in opposition to the crack propagation, the toughening mechanism[26,37,38,39]. The intensity of this mechanism, associated with the surface topography pattern, was responsible for distinct susceptibility to low temperature degradation in the present study. Another important fact is that surfaces subjected to grinding seem to be less susceptible to *t*→*m* transformation during aging (LTD), since the Xfine LTD and Coarse LTD groups presented lower *m*-phase content when compared to the Ctrl LTD group (Ctrl LTD – 53.33% and 3.86 µm, Xfine LTD – 15.75% and 0.87 µm, Coarse LTD – 28.75% and 1.72 µm), most likely caused by a protective effect achieved by the formation of the tension barrier by *t*→*m* transformation on the surface on the material.

It is important to note that the current findings conflict with Kosmac et al. in 1999[4], 2008[15], where grinding with diamond burs did not promote an increase in m-phase content and introduced severe defects on the material surface, leading to degradation of the mechanical properties. Those authors used a high-speed handpiece for grinding, which can lead to a temperature increase at the Y-TZP surface, consequently triggering a reverse m→t transformation that works against transformation toughening. In the present study, a low-speed handpiece was used with abundant water-cooling (≅ 30ml/min), resulting in a less aggressive grinding procedure.

In some situations, surface flaws introduced by surface treatments can overcome the effects of the compression layer and be detrimental to fatigue properties[4,5,15], leading to a decrease in biaxial strength and survival after aging (at 134ºC)[8,40]. In this present study, grinding with a coarse diamond bur produced high roughness values which were not sufficient to promote a decrease in biaxial strength. When LTD was induced in the specimens prepared using a course bur, the *t*→*m* transformation induced a formation of a thin layer of tension barrier (Coarse LTD presented lower *m*-phase content (28.75%) and transformation depth (1.717 µm) when compared to the Ctrl (53.33% and 3.86 µm respectively), which was not enough to prevent crack propagation, causing degradation of mechanical properties.

It was proposed that the compressive stresses developed on the zirconia surface would initially increase the flexural strength[4,9,23]. However, in an opposite effect, with the progression of the *t*→*m* transformation, microcracks and residual tensile stresses could appear and lead to a decrease in strength. This hypothesis is supported by the findings of Chevalier et al. 2007[41], who observed that the phase transformation starts at the surface, as observed in in the current study, and proceeds into the bulk of the material, as shown under fatigue conditions[5,8,40]. Thus, fatigue studies should be conducted that consider the present experimental factors (roughness and m-phase content), since they can intensify material degradation under cyclic loading.

Some studies[42,43] have shown strength data ($\sigma_{0.5}$, σ_1) for 5-10% probability of failure from Weibull analysis, as described in Table 3. These values are considered clinically more relevant, since they are related to premature failure of the material. The current literature shows that, from a clinical point of view, maximum masticatory forces may easily achieve 300-400N, with lower average chewing forces of approx. 220N in the molar region[44,45]. Assigning those forces to a contact area of 7-8mm^2 (single molar tooth) results in an average chewing pressure of 27-31MPa[46]. Thus, all surface treatments evaluated in the present study generated $\sigma_{0.5}$ values that were significantly higher than those involved in a clinical situation, even after LTD, although an evaluation of these effects under mechanical fatigue still seems to be necessary, as it could result in a different response.

CONCLUSIONS

These present findings support that LTD was not detrimental to the mechanical properties of zirconia. Grinding was not detrimental; however, when the zirconia was submitted to LTD, the defects introduced by grinding could be detrimental to the material resistance. Roughness seems to be an important factor that should be taken into consideration for predicting a material`s mechanical behaviors after LTD, as a smoother surface decreases the susceptibility of LTD and a rougher surface showed to be more prone to LTD. Thus, grinding zirconia should be avoided; although, tools with low grit sizes should be employed when grinding is necessary.

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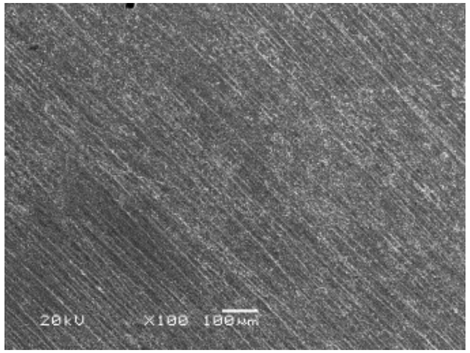
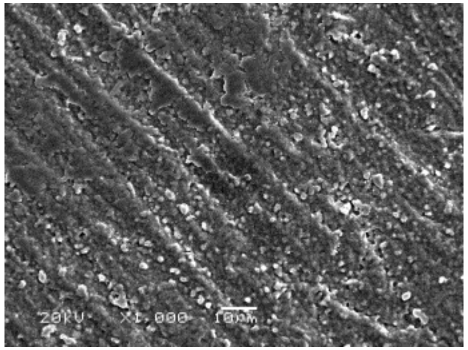
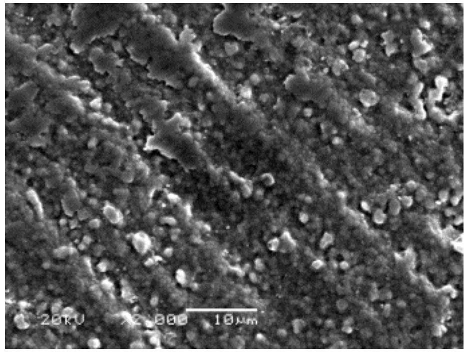
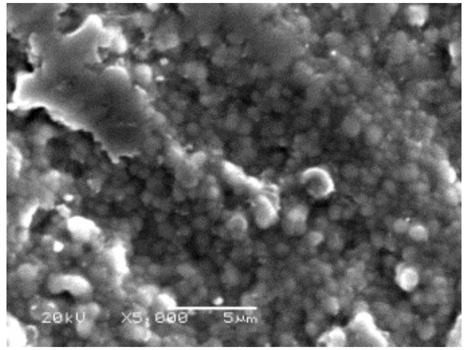
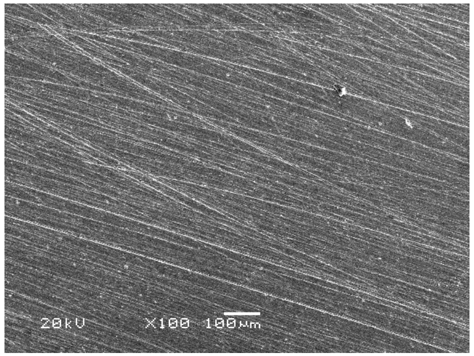
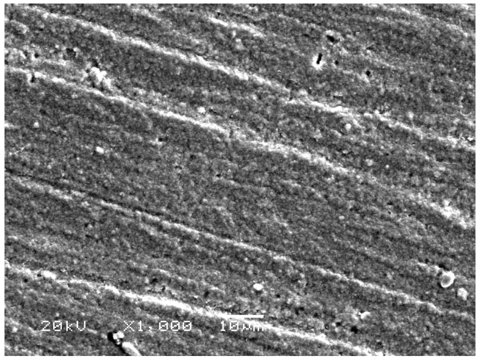
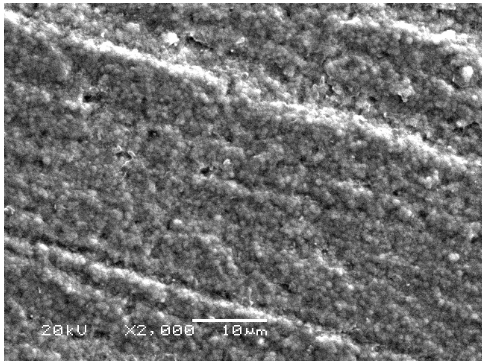
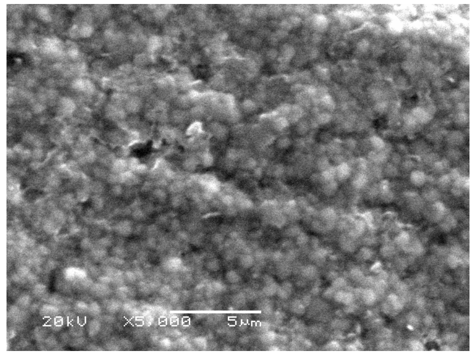
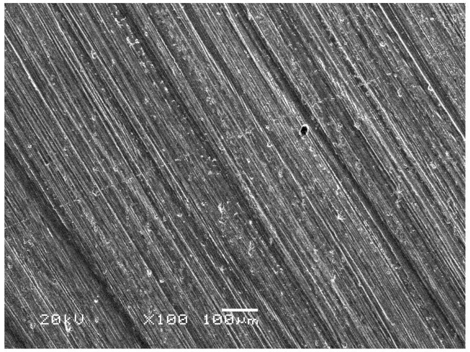
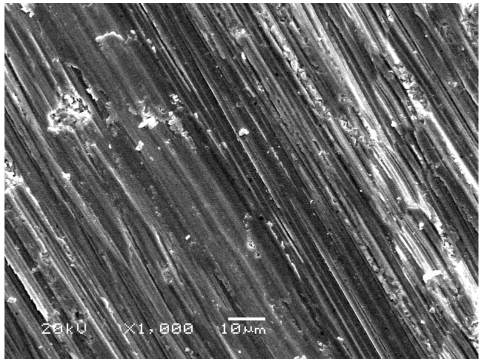
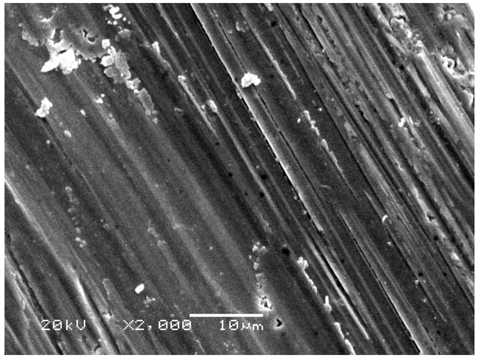
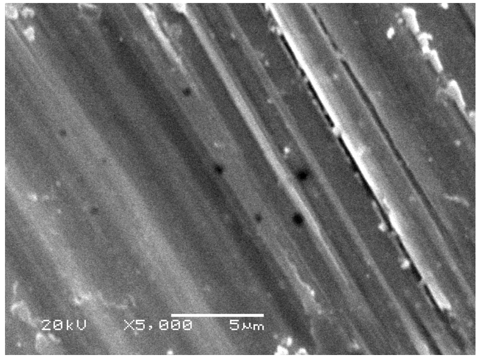
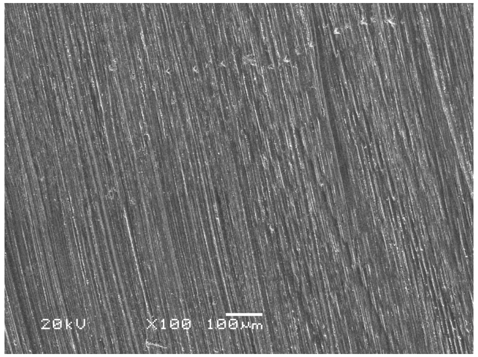
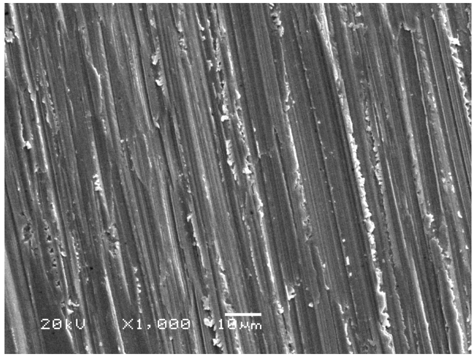
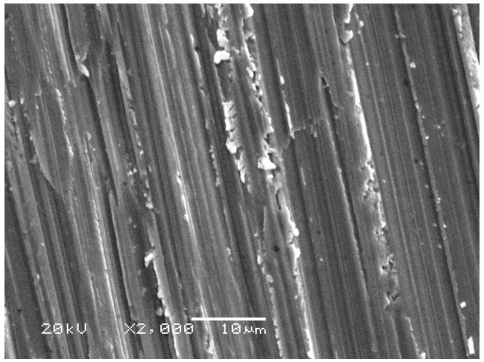
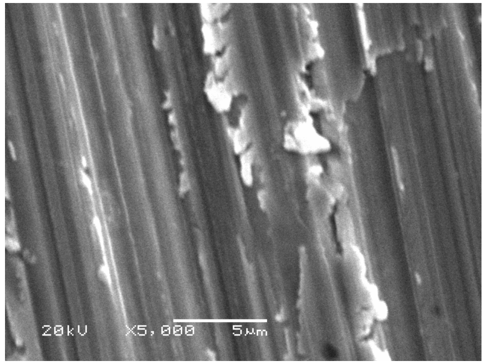
FIGURES

	100x	1000x	2000x	5000x
Ctrl group				
	A	**B**	**C**	**D**
Ctrl LTD group				
	E	**F**	**G**	**H**

Xfine group				
	I	**J**	**K**	**L**
Xfine LTD group				
	M	**N**	**O**	**P**

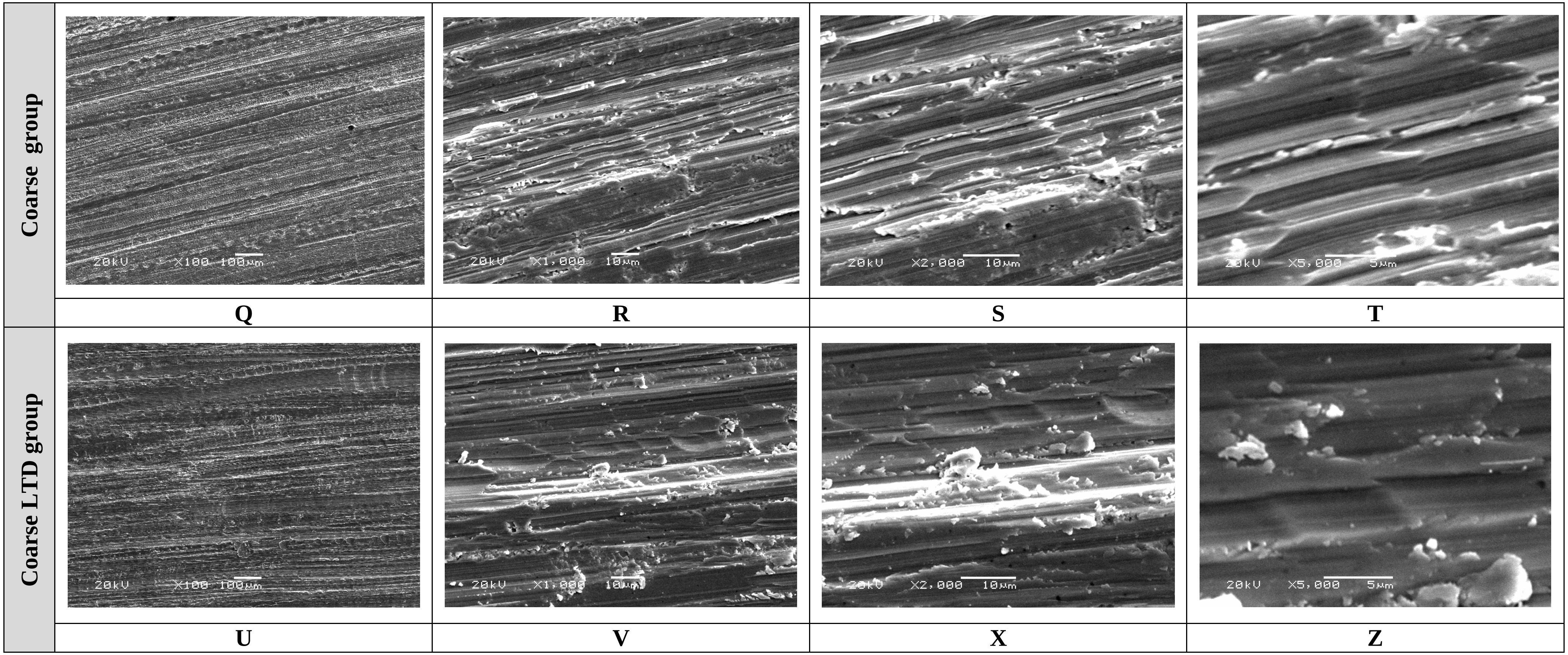


Figure 1 – Micrographics obtained trough Scanning Electron Microscopy of tested groups (100x, 1000x, 2000x and 5000x of magnification

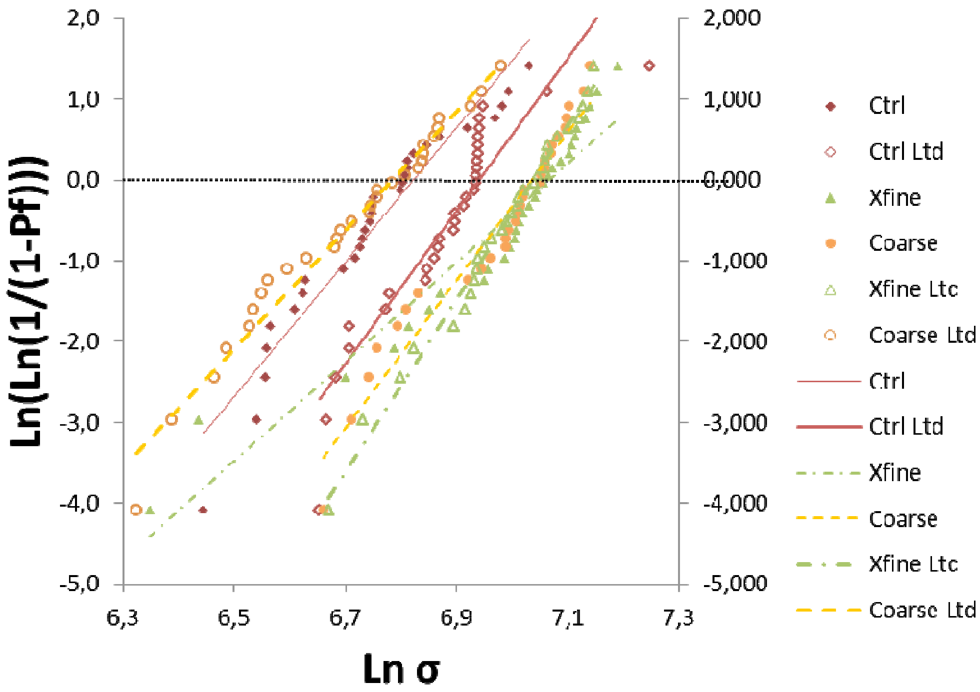


Figure 2 –.Weibull analysis plots for testing group data.

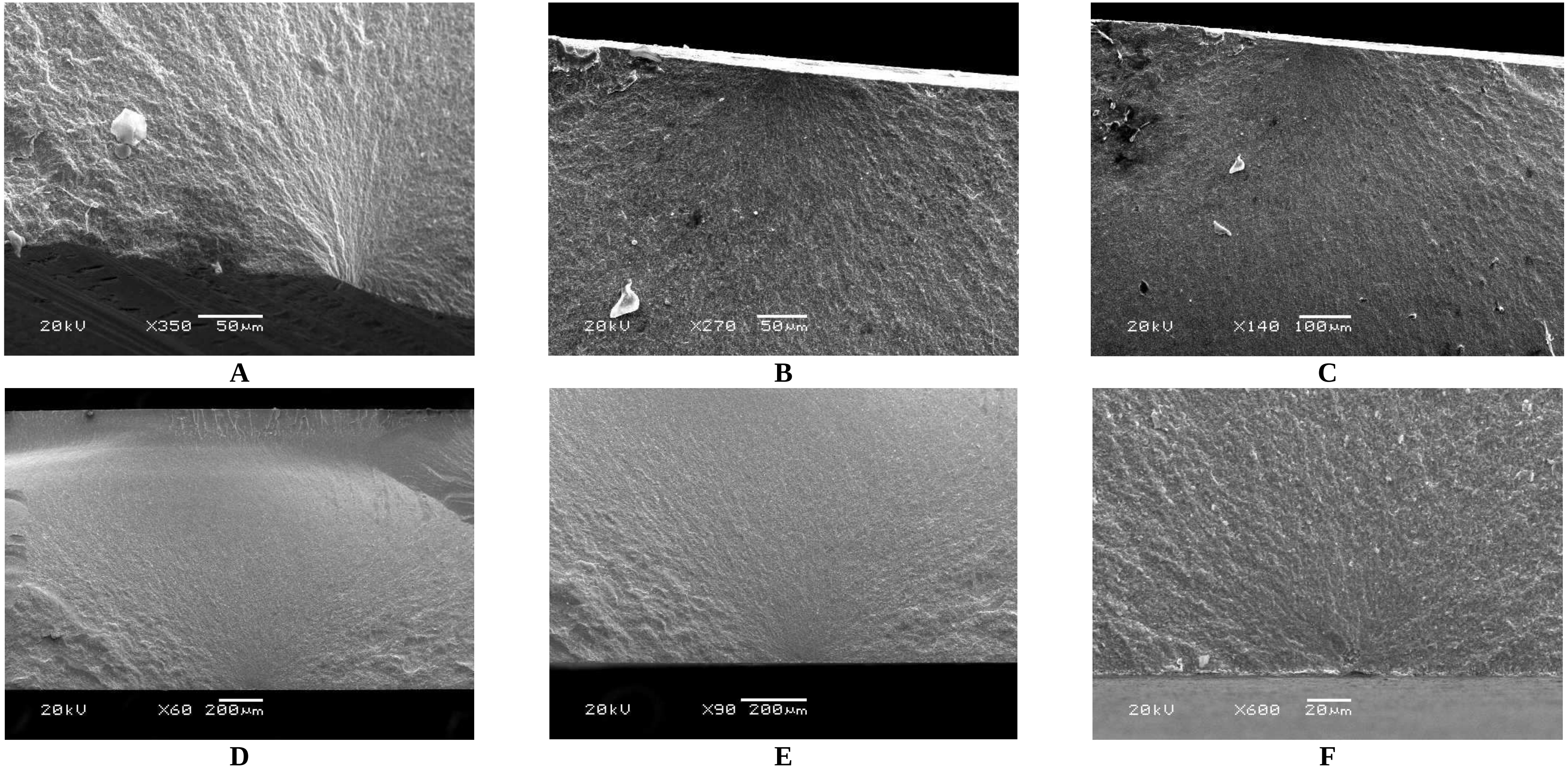


Figure 3 – Representative micrographics of the fracture surfaces (fractography examination) using a Scanning Electron Microscope.

TABLES

Table 1 – Experimental Design

Group	**N**	**Surface treatment**	**Low Temperature Degradation - LTD**
Ctrl	34	As-sintered (Without treatment, control)	Without
Ctrl LTD	34		With
Coarse	34	Coarse Diamond Bur #3101G (average grit size 181 µm)	Without
Coarse LTD	34		With
Xfine	34	Extra-fine Diamond Bur #3101FF (average grit size 25 µm)	Without
Xfine LTD	34		With

Table 2 – X-ray Difractometry analysis (% of monoclinic Phase, Depth of Transformed Layer), Results from Analysis of Variance (two-way ANOVA) from Roughness Data (Ra and Rz) for treatment and aging factor (α=0.05, p = 0.0000), descriptive statistical analysis (Mean and Standard Deviation) of biaxial flexural data and Pearson linear correlation analysis (coefficient of linear correlation) between roughness (Ra) versus mean strength(σ_c).

Groups	Monoclinic Phase (%)	Depth of transformed layer (µm)	Roughness Ra (µm)	Roughness Rz (µm)	Mean (σ_c) (MPa)	Standard Deviation (MPa)	Coefficient of linear correlation – r (Ra x σ_c)
CTRL	0.00	0.00	0.67 – 0.27[d]	4.43 – 1.53[d]	865.9	126.1	-0.34 (p= 0.07)
CTRL LTD	53.33	3.86	0.28 – 0.13[e]	2.24 – 0.62[e]	980.2	122.9	0.03 (p= 0.89)
XFINE	9.00	0.48	0.86 – 0.15[c]	5.55 – 0.85[c]	1085.8	179.7	-0.07 (p= 0.70)
XFINE LTD	15.75	0.87	0.62 – 0.1[d]	4.32 – 0.61[d]	1086.6	120.1	-0.09 (p= 0.64)
COARSE	12.78	0.69	1.41 – 0.3[b]	8.43 – 1.45[b]	1076.8	134.9	-0.51 (p= 0.003)
COARSE LTD	28.75	1.72	1.79 – 0.29[a]	10.64 – 1.47[a]	830.0	135.0	-0.18(p= 0.35)

***same letters correspond to statistical similarity**
***different letters correspond to statistical difference**

Table 3 – Characteristic Strength (σ_c), Weibull modulus (*m*) and respective Confidence Intervals (CI - 95%).

Groups	σ_c	CI (95%)	*m*	CI (95%)	$\sigma_{0.5}$
Ctrl	917.58[c]	870.86 – 965.40	8.33[a]	5.83 – 10.70	642.26
Ctrl LTD	1033.36[b]	986.9 – 1080.62	9.46[a]	6.62 – 12.16	754.85
Xfine	1171.33[a]	1091.39 – 1254.68	6.15[a]	4.31 – 7.91	722.88
Xfine LTD	1138.52[a]	1093.26 – 1184.33	10.72[a]	7.50 – 13.78	863.11
Coarse	1136.32[a]	1083.58 – 1190.07	9.15[a]	6.41 – 11.76	821.44
Coarse LTD	885.04[c]	833.93 – 937.75	7.31[a]	5.12 – 9.40	589.63

***same letters correspond to statistical similarity**
***different letters correspond to statistical difference**

5. CONSIDERAÇÕES FINAIS

Sob as condições apresentadas nesse estudo, podemos observar que sob um ponto de vista metodológico o desgaste da superfície da Y-TZP executado com discos diamantados não pode ser utilizado para simular o desgaste apresentado clinicamente com pontas diamantadas, pois estes não apresentaram efeitos semelhantes.

A LTD promoveu um aumento significativo de fase monoclínica na superfície do material e promoveu um aumento significativo da resistência a flexão biaxial para o grupo *Ctrl*, demonstrando que mesmo 20 horas de envelhecimento em autoclave não foram suficientes para promover degradação das propriedades mecânicas do material.

O tratamento de superfície (desgaste) também promoveu um aumento de fase monoclínica na superfície do material e um aumento na resistência a flexão biaxial, corroborando o mecanismo de tenacificação, demonstrado na literatura, que a zircônia sofre quando submetida ao desgaste. Entretanto quando a zircônia foi submetida ao desgaste e a LTD, apenas para pontas diamantadas de menor granulação (*Xfine*) não foi observada perda estatisticamente relevante de propriedades mecânicas. Para o grupo *Coarse* (maior granulação) foi observada uma diminuição da resistência flexural, indicando que os defeitos introduzidos pelo desgaste com brocas de maior granulação potencializam os efeitos da LTD e podem atuar como fatores concentradores de tensão e levar a fratura catastrófica do material ao longo do tempo.

Dessa forma nossos achados suportam que a LTD, embora tenha impactado em aumento de fase monoclínica e alterado a micromorforlogia de superfície, não provocou efeito estatisticamente significante para as propriedades mecânicas do material e que o tratamento de superfície (desgaste), inicialmente não foi prejudicial às propriedades mecânicas. Entretanto, quando os efeitos do desgaste são somados aos efeitos da LTD, os defeitos introduzidos pelo desgaste podem ser prejudiciais à resistência flexural do material. Por isso o desgaste da superfície da zircônia deve ser evitado e quando necessário deve ser executado com pontas diamantadas de menor granulação.

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ANEXOS

Anexo A - *AUTHOR GUIDELINES FOR JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS.*

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Anexo B - *AUTHOR GUIDELINES FOR JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART B: APPLIED BIOMATERIALS.*

Aims and Scope
Journal of Biomedical Materials Research Part B: *Applied Biomaterials* is an official journal of the Society for Biomaterials, the Japanese Society for Biomaterials, the Australasian Society for Biomaterials, and the Korean Society for Biomaterials. It is a peer-reviewed journals serving the needs of biomaterials professionals who devise, promote, apply, regulate produce, and market new biomaterials and medical devices. Papers are published on device development, implant retrieval and analysis, manufacturing, regulation of devices, liability and legal issues, standards, reviews of different device areas, and clinical applications. Published manuscript fit into one of six categories: original research reports, clinical device-related articles, short research and development reports, review, special report, or columns and editorials. Manuscripts from all countries are invited but must be in English. Authors are not required to be members of a Society for Biomaterials.

Types of Articles Considered for Publication

Original Research Reports: Full-length papers consisting of complete and detailed descriptions of a research problem, the experimental approach, the findings, and appropriate discussion. Findings should represent significant new additions to knowledge.

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