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### Fatigue limit of polycrystalline zirconium oxide ceramics: Effect of grinding and lowtemperature aging



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#### ABSTRACT

The following study aimed to evaluate the effect of grinding and low-temperature aging on the fatigue limit of Y-TZP ceramics for frameworks and monolithic restorations. Disc specimens from each ceramic material, Lava Frame (3M ESPE) and Zirlux FC (Ivoclar Vivadent) were manufactured according to ISO:6872-2008 and assigned in accordance with two factors: (1) "surface treatment"-without treatment (as-sintered, Ctrl), grinding with coarse diamond bur (181 µm; Grinding); and (2) "low-temperature aging (LTD)" - presence and absence. Grinding was performed using a contra-angle handpiece under constant water-cooling. LTD was simulated in an autoclave at 134 °C under 2-bar pressure for 20 h. Mean flexural fatigue limits (20,000 cycles) were determined under sinusoidal loading using stair case approach. For Lava ceramic, it was observed a statistical increase after grinding procedure and different behavior after LTD stimuli (Ctrl<Grinding; Ctrl<Ctrl Ltd; Grinding=Grinding Ltd); while for Zirlux, grinding and low-temperature aging promoted a statistical increase in the fatigue limit (Ctrl < Grinding; Ctrl < Ctrl Ltd; Grinding < Grinding Ltd). An important increase was observed in m-phase content after both stimuli (grinding and LTD), although with different intensities. Additionally, fatigue test did not promote increase of m-phase content. Thus, tested grinding and low temperature aging did not damage the fatigue limit values significantly for both materials evaluated, even though those conditions promoted increase in m-phase.

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#### 1. Introduction

Nowadays, Y-TZP ceramics (Yttrium-stabilized Tetragonal Zirconia Polycrystal) are being considered one of the best options to produce all-ceramic FDPs (fixed dental prosthesissingle or multi-unit), as they associate superior strength (provided by a Y-TZP framework) with good esthetics (provided by a feldspathic porcelain veneering) (Denry and Kelly, 2014). In fact, zirconia is a polymorphic metastable material (Piconi and Maccauro, 1999) that when required (submitted to stimuli – mechanical, physical, and/or chemical) may respond through a phase transformation mechanism (tetragonal (t) to monoclinic (m)) (Garvie and Nicholson, 1972; Hannink, 2000; Lazar et al., 2008; Amaral et al., 2013; Pereira et al., 2015a).

Literature states distinct effects of this phase transformation mechanism: first, it was noted an increase on mechanical properties, which is known as transformation toughening mechanism (Hannink, 2000; Amaral et al., 2013; Pereira et al., 2015a); then as this transformation spreads through ceramics surface and subsurface (promoting grains detachment/pullout and introduction of micro-cracks on the grains neighbor areas), it promotes roughness increase, reduction in strength, fracture toughness, and density (Chevalier et al., 2007; Ban et al., 2008; Kim et al., 2009; Flinn et al., 2012, 2014; Egilmez et al., 2014; Pereira et al., 2015b). This spontaneous degradation mechanism is known as low-temperature degradation (LTD) (Kobayashi et al., 1981).

Currently, besides veneered FDPs application, Y-TZP ceramics has been proposed for manufacturing monolithic fullcontour restorations (Beuer et al., 2012; Sabrah et al., 2013; Nakamura et al., 2015). One of the advantages of this application is the possibility of an even more conservative tooth preparation, once it requires a thinner thickness and the application of veneering porcelain is dispensable. This could mean in an obvious solution for one of the most reported (clinical trials) reasons of failures of Y-TZP FDPs (chipping or fracture of the veneering porcelain) (Raigrodski et al., 2006; Sailer et al., 2007a, 2007b; Beuer et al., 2010; Christensen and Ploeger 2010; Monaco et al., 2015).

Although the indication of monolithic full-contour restoration has clear advantages, it also means that Y-TZP ceramic will be daily exposed directly to the oral environment (presence of different stimuli, such as: oral mastication forces, exposure to water, temperature (low-temperature degradation), pH changes, oral microorganisms (Chevalier et al., 2007; Inokoshi et al., 2015; Lucas et al., 2015a; Cotes et al., 2014; Egilmez et al., 2014; Turp et al., 2012, Bordin et al., 2015), which means an environment that could accelerate the LTD mechanism development.

In addition, another important aspect is that after machining at CAD/CAM (computer aided design / computer aided machining) systems, clinical adjustments (with diamond grinding instruments) are usually needed to achieve a better adaptation and an adequate emergency/occlusion profile (Aboushelib et al., 2009; Amaral et al., 2013; Pereira et al., 2014; 2015a). Literature has already shown that grinding might introduce different types of damage (defects), such as scratches and cracks of various depths, which penetrate toward the bulk of the material (Ban et al., 2008; Quinn et al., 2005; Papanagiotou et al., 2006). Besides the introduction of defects, it may also trigger the *t*-*m* phase transformation mechanism (Muñoz-Tabares and Anglada, 2012; Pereira et al., 2014; 2015a), but there is few data regarding the effect of this procedure on the Y-TZP ceramic susceptibility to LTD (Kosmac et al., 2008, Amaral et al., 2013, Pereira et al., 2015a).

Clinically, ceramic restorations are susceptible to fatigue failure, mainly due to the presence of moisture and cyclic chewing forces (Gonzaga et al., 2011). Fatigue failure may be defined as the fracture of the material due to progressive brittle cracking under repeated cyclic stresses of intensity below the material normal strength (Zhang et al., 2013; Wiskott et al., 1995). Although this fact is already extensively known, there are few studies so far assessing the fatigue life behavior of Y-TZP ceramics (Kosmac et al., 2008, Nakamura et al., 2015), and to the authors knowledge none took into account surface treatments (grinding) and susceptibility to LTD. It is feasible to notice that these stimuli directly result on introduction of defects onto the materials surface and subsurface, probably increasing the risk of a premature failure in a fatigue life scenario (Hondrum, 1992; Kelly, 2004; Mitov et al., 2011), which might affect the predictability and longevity of the prosthetic rehabilitation.

Thus, before we may recommend the application of Y-TZP monolithic restorations (hazardous condition – directly exposed to oral environment) and aiming to better understand the behavior of Y-TZP as a framework material in FDPs, well delineated in-vitro studies to evaluate the effects of grinding and LTD mechanism in addition to the susceptibility of degradation of this ceramic on the fatigue limit are required. Hence, this study aimed to evaluate the effect of grinding with diamond burs and low-temperature aging in a steam autoclave at the fatigue limit (staircase method) of Y-TZP ceramics for frameworks and monolithic restorations.

#### 2. Material and methods

#### 2.1. Specimen preparation

Pre-sintered zirconia blocks (LOT 637328 Rev.2, Zirlux FC, Ivoclar Vivadent, Amherst, USA; and LOT 70201131797 Lava Frame, 3M ESPE, Seefeld, Germany) were ground into cylinders in a polishing machine (EcoMet/AutoMet 250, Buehler, United States) using a 600 grit silicon carbide paper and then cut under water irrigation with a diamond saw (ISOMET 1000, Buehler, Lake Bluff, IL, USA), resulting in eighty (N=80) zirconia specimens, from each ceramic material, with initial dimensions of 18 mm diameter and 1.65 mm thickness. The discs were then polished with a 1200 grit silicon carbide paper, cleaned in 78% isopropyl alcohol ultrasonic bath for 10 min and sintered according to each manufacturer's recommendation.

After sintering, the specimens were carefully inspected, being discarded those presenting discrepancies in dimensions above the standard variation  $(1.2\pm0.2 \text{ mm} \text{ in length}, 14\pm2 \text{ mm} \text{ in diameter})$ , indicated by ISO:6872-2008. Then the specimens (after approved by the inspection) from each ceramic material were randomly allocated into four groups

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