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Influence of heat treatment on the microstructure and the physical and mechanical properties of dental highly translucent zirconia

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PURPOSE. Microstructural and physico-mechanical characterization of highly translucent zirconia, prepared by milling technology (CAD-CAM) and repeated firing cycles, was the main aim of this *in vitro* study. **MATERIALS AND METHODS.** Two groups of samples of two commercial highly-translucent yttria-stabilized dental zirconia, VITA YZ-HT^{White} (Group A) and Zolid HT + White (Group B), with dimensions according to the ISO 6872 "Dentistry - Ceramic materials", were prepared. The specimens of each group were divided into two subgroups. The specimens of the first subgroups (Group A_1 and Group B_1) were merely the sintered specimens. The specimens of the second subgroups (Group A_2 and Group B_2) were subjected to 4 heat treatment cycles. The microstructural features (microstructure, density, grain size, crystalline phases, and crystallite size) and four mechanical properties (flexural strength, modulus of elasticity, Vickers hardness, and fracture toughness) of the subgroups (i.e. before and after heat treatment) were compared. The statistical significance between the subgroups (A_1/A_2, and B_1/B_2) was evaluated by the t-test. In all tests, *P* values smaller than 5% were considered statistically significant. **RESULTS.** A homogenous microstructure, with no residual porosity and grains sized between 500 and 450 nm for group A and B, respectively, was observed. Crystalline yttria-stabilized tetragonal zirconia was exclusively registered in the X-ray diffractograms. The mechanical properties decreased after the heat treatment procedure, but the differences were not statistically significant. **CONCLUSION.** The produced zirconia ceramic materials can be safely (i.e., according to the ISO 6872) used in extensive fixed prosthetic restorations, such as substructure ceramics for three-unit prostheses involving the molar restoration and substructure ceramics for prostheses involving four or more units. Consequently, milling technology is an effective manufacturing technology for producing zirconia substructures for dental fixed all-ceramic prosthetic restorations. [J Adv Prosthodont 2022;14:96-107]

KEYWORDS
Computer-aided design and computer-aided manufacturing (CAD-CAM); Zirconia; Heat treatment; Mechanical properties

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INTRODUCTION

Dental all-ceramic prosthetic restorations (inlays, onlays, crowns, and bridges) have been popular in clinical practice over the last 20 years, aiming at restoring fractured or cracked teeth.[1] Compared to metal or metal-ceramic prosthetic restorations, all-ceramics manifest a better esthetic appearance, including translucence, as well as biocompatibility.[1,2] Currently, zirconia (ZrO_2) is the most preferred dental bio-ceramic material in all-ceramic restorations.[3]

Pure zirconia crystallizes in three crystallographic phases, monoclinic (up to 1170°C), tetragonal (between 1170 and 2340°C), and cubic (>2340°C), but only the tetragonal phase displays the best mechanical properties.[4,5] Nevertheless, pure zirconia features two main problems. According to clinical studies, zirconia is vulnerable to accelerated ageing, mainly in a moisture environment, such as the mouth cavity, which is known as "low-temperature degradation" (LTD).[3,4] Moreover, the monoclinic to tetragonal phase transition occurs with a significant volume change, leading to surface deterioration and microcracks propagation.[6,7] In the case of dental materials, this phase transition occurs at temperatures close to the temperatures of the heat treatment cycles applied on the ceramic substrate (e.g. zirconia) to coat it with the four ceramic layers (interface, dentin, enamel, and glaze), i.e. in the order of ~1000°C. Thus, oxides, such as yttria (Y_2O_3), are added to zirconia, resulting in lowering the tetragonal to monoclinic transformation temperature. In other words, they stabilize the tetragonal phase of zirconia even in room temperature, allowing tetragonal zirconia to be used as a bulk material in biomedical applications.[4] The yttria-stabilized tetragonal zirconia (Y-TZP) exhibits good esthetic performance (tooth-like color), excellent mechanical properties, and attractive biological properties.[6,8]

Computer-aided design and computer-aided manufacturing (CAD-CAM) is a modern process, which allows the production of dental prosthetic restorations with high efficiency in daily clinical practice.[9,10] The application of CAD-CAM technology in the production of Y-TZP restorations displays essential advantages, such as high flexural strength and satisfactory esthetics of the final product, less laboratory time, and fewer dental sessions.[11] The combination of the desired physico-mechanical properties of Y-TZP with the absolute dimensional precision provided by the CAD-CAM technology qualifies the wide use of Y-TZP restorations, even in cases of patients with an unfavorable occlusion, parafunctional habits, or fracture history, as well as in cases where there is limited space for restorative materials.[11,12]

According to the ISO 6872 standard "Dentistry-Ceramic Materials", a dental material must exhibit good chemical, mechanical, and optical properties, comparable to natural teeth.[13,14] Hence, in order to produce an attractive alternative material of the widely used metal-ceramic restorations, the all-ceramic restorative materials must mimic the esthetics of natural teeth (enamel and dentin) and match their mechanical properties with natural teeth to avoid the injury of the natural teeth of the opposite jawbone (note that zirconia has a high hardness value (~12 GPa), compared to the tooth (enamel: 3 - 6 GPa)).[14] To reach these requirements, Y-TZP is coated with the four ceramic mass layers mentioned above (if necessary, with esthetic colors). As already stated, the coating process involves a sequence of four firing cycles.

However, although zirconia is a promising dental material in esthetic restorations, Y-TZP is a new material in dentistry. Additionally, the milling technique (soft milling is involved in CAM) is also a new technology in Y-TZP processing. Literature survey reveals poor documentation on the maintenance of the microstructure and the physico-mechanical properties of the final dental zirconia material after milling and the repeated firing cycles. This study reports on this working hypothesis.

More specifically, bulk samples of highly translucent zirconia were prepared by CAD-CAM technology, with dimensions according to the ISO 6872 standard. Two well known (i.e. with good performance in dentistry) types of zirconia powders (actually in the form of loosely pre-sintered blocks, as explained in the next section) were used. The produced sintered bulk samples were subjected to the four repeated heat treatment cycles, according to the manufacturers' instructions, without applying the coating layers. The mechanical properties of the produced samples were compared to the values of the ISO 6872 standard. The

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influence of the heat treatment on the microstructural features of the samples was also investigated.

MATERIALS AND METHODS

Two commercial highly translucent yttria-stabilized dental zirconia, VITA YZ-HT^{White} (VMK-Master; Vita, Bad Sackingen, Germany) and Zolid HT + White (Amann Girrbach, Koblach, Austria), were used, and the samples produced from them are denoted hereafter as Group A and Group B, respectively (Table 1). It should be noted that these companies provide these zirconia materials in the form of loosely sintered (pre-sintered) disc blocks, which can be easily shaped by the milling (CAD-CAM) process. More specifically, twenty specimens (n = 20) from each group were constructed using CAD-CAM technology with the aid of Siemens NX 12 software (Fig. 1A, B) and a Ceramill Motion 2 milling machine (Amann Girrbach, Koblach, Austria). Their dimensions were according to specification ISO 6872 "Dentistry-Ceramic materials",[13] i.e., width 4.0 (± 0.2) mm, thickness 3.0 (± 0.2) mm, and length 35.0 (± 0.2) mm. In addition, according to this standard, the samples must be at least 2 mm longer than the span between the supporting rods applied in the 3-point bending strength test (see it below), and the ratio of thickness to length (b/L) must be ≤ 0.1. Then, the specimens were subjected to sintering process, according to the manufacturers' instructions for each group. The sintering temperatures and time, as well as the heating and cooling rates for the two groups A and B, are listed in Table 1.

Table 1. Tested materials (groups) and conditions of the sintering procedure, according to the manufacturers' instructions, applied in this study

Sintering procedure	A* (n = 20)	B† (n = 20)
Temperature (°C)	1450	1450
Holding time (min)	120	120
Heating rate (K/min)	17	20 (up to 950°C) 10 (up to 1450°C)
Cooling rate (K/min)	200	20

*: VITA YZ HT^{White} (Highly translucent zirconia).
†: Zolid HT + White (Highly translucent zirconia).

The sintered specimens of each group were divided into two subgroups (n = 10, for each subgroup). The specimens of the first subgroups (Group A_1 and Group B_1) were merely the sintered specimens. The specimens of the second subgroups (Group A_2 and Group B_2) were subjected to 4 heat treatment cycles (Table 2) in a special oven (P510 Programat; Ivoclar Vivadent, Ellwangen, Germany), which were identical to the ceramic mass firing cycles applied in order to produce all-ceramic prosthetic restorations, i.e., according to the manufacturer's instructions, namely the company Creation (Creation Willi Geller International GmbH, Meiningen, Austria). The only difference with real all-ceramic dental restorative material production was that the samples were not coated with the four layers

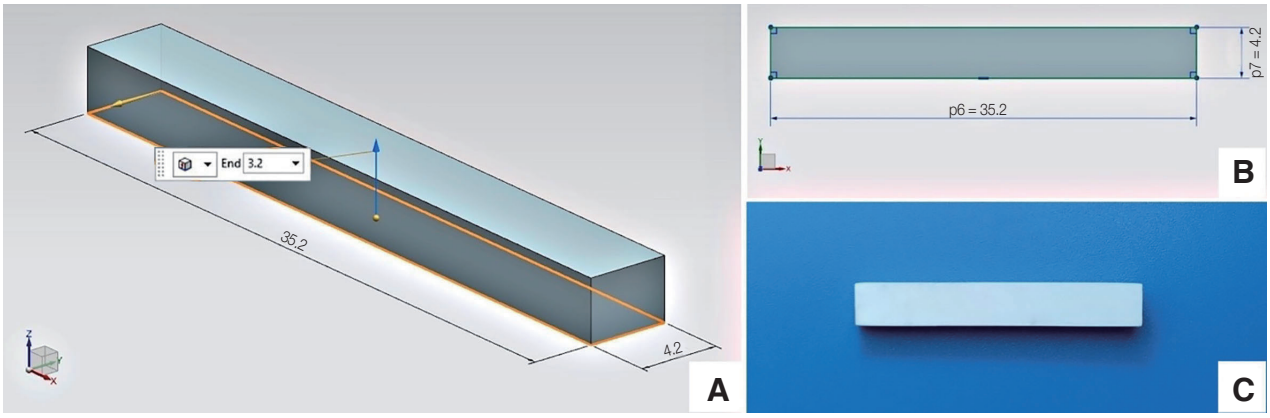

Fig. 1. (A, B) Design of specimens using the CAD-CAM technology with the aid of the Siemens NX 12 software, with dimensions according to the specification ISO 6872 "Dentistry - Ceramic materials",[13] and (C) a resultant highly translucent zirconia specimen (VITA YZ HT^{White}).

Table 2. Tested materials (subgroups) and conditions of the ceramic mass firing procedure, according to the manufacturers' instructions, applied in this study

Ceramic mass‡	Firing conditions§	A_1* (n = 10)	A_2* (n = 10)	B_1† (n = 10)	B_2† (n = 10)
Interface	940°C, 1 min	-	√	-	√
Dentin 1	910°C, 1 min	-	√	-	√
Dentin 2 (or Enamel)	900°C, 1 min	-	√	-	√
Glaze	910°C, 1 min	-	√	-	√

*: VITA YZ HT^{White} (Highly translucent zirconia).
†: Zolid HT + White (Highly translucent zirconia).
‡: The specimens were not coated with the four layers, but they were merely subjected to these four heat treatment cycles, which were identical to the ceramic mass firing cycles applied in order to produce all-ceramic prosthetic restorations.
§: The samples were put in a preheated furnace at 500°C (in the glaze at 550°C) for 6 min (in the glaze for 2 min), the heating rate to the firing temperature was 45 K/min, and then, the samples cooled down naturally inside the furnace with an open door (i.e., with 40 - 50 K/min cooling rate).

but were merely subjected to these four heat treatment cycles (Table 2). The properties of these subgroups (A_2 and B_2) were compared to the samples of the subgroups A_1 and B_1 (Table 2), which were merely the sintered specimens of Groups A and B (Table 1).

The following techniques were carried out for the characterization of the above specimens. Initially, the crystalline phases developed in the produced samples were identified in 2 different specimens per zirconia subgroup by X-ray diffraction analysis (XRD, D8 Advance, Bruker AXS, Billerica, MA, USA; Cu K_α radiation (λ = 1.5406 Å), produced at 30 kV and 25 mA, was used), in the range of diffraction angles (2θ) between 20 and 100° with a 2θ step of 0.004 °/s. The diffractograms were compared to standards complied with the International Center for Diffraction Data (ICDD).

The observation of the microstructure of the zirconia required the proper preparation of the specimens. More specifically, the zirconia specimens (two zirconia specimens from each group) were grounded (RotoPol-25; Struers, Cleveland, OH, USA) using SiC disks (from 160 to 2400 grit). Then, the final polishing (i.e., mirror finishing) took place using diamond pastes, down to 1 μm (Diamant Mecaprex Spray; Presi, Eybens (Isère), France). Finally, the polished specimens were ultrasonically cleaned in a distilled water bath and dried. Next, the polished Y-TZP specimens were thermally etched at 1200°C for 20 min to reveal the grain boundary network. In order to suppress any significant grain size change or grain growth during the thermal etching process, a fast-heating rate of 30 K/min was applied.[15] Afterwards, the samples were sputtered with a Pt thin film (5 nm) in a sputtering machine to obtain a conductive surface. The microstructure of the thermally etched specimens was observed in a scanning electron microscope (SEM, 1455VP, Leo, ZEISS, Oberkochen, Germany), using an acceleration voltage of 20 kV.

Density measurements (ρ, in g/cm^3) were conducted by the Archimedes method (i.e., immersion of the samples in water at room temperature), using the equation (1)

$$p = \left(\frac{W_0 - W_t}{W_0} \right) \times 100 \quad (1)$$

where W_0 refers to the weight of the specimen before immersion in water at room temperature, and W_t refers to the weight of the specimen after immersion.

The mechanical properties of the produced samples were estimated by measuring their flexural strength (σ, in MPa), modulus of elasticity (E, in GPa), Vickers microhardness (HV, in GPa), and fracture toughness (K_{IC}, in MPa·m$^{-0.5}$). The flexural strength and the modulus of elasticity were determined via three-point bending strength tests (Autograph AGS-H; Shimadzu, Kyoto, Japan). More specifically, the cross-sectional dimensions of each specimen were measured (the accuracy was ± 0.01 mm). Then, the zirconia specimen was centered at the gap (the span was 25 mm) between the supporting rods of the test machine. The load was applied on the 4 mm wide face of the sample, perpendicularly to its long axis. The crosshead speed was 1.0 mm/min. The load until fracture was measured. The flexural strength (σ) and the modulus of elasticity (E) were calculated using the equations

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(1) and (2),[13,16] respectively

$$\sigma = \frac{3FL}{2wh^2} \quad (2)$$

and

$$E = \frac{L^3m}{4wh^3} \quad (3)$$

where, F is the fracture load, and L, w, and h are the span between the supporting rods, the specimen width, and the thickness, respectively. The slope of the load versus displacement is denoted with m (needed to calculate the modulus of elasticity in the Eq. 3). Ten specimens from each subgroup (n = 10) were tested.

The diamond indenter of a Digital Microhardness Tester (Time Instrument; Testing Indonesia, Jakarta, Indonesia) was applied on the polished surface of the zirconia specimens embedded in an acrylic resin (resin phenolique; Presi, Eybens (Isère), France) (prepared as reported above for the preparation of the samples for SEM, up to the mirror finishing stage) with a peak load of 500 g (or 4.9 N) for 30 s. The Vickers microhardness was calculated according to the equation (4)[10]

$$HV = 1.854\ P/L^2 \quad (4)$$

where P is the applied load (in kg) and L is the average length of the two diagonals (in mm) of the indentation. The presenting results are the average of 5 different indentations on each sample (n = 5).

From the length (l) of the cracks propagated from the corners of the pyramid indentation, the value of fracture toughness (K_{IC}) was calculated by using the equation (5), given by Lankford[17,18]

$$K_{IC} = 0.0782 \times (HV \times a^{0.5}) \times \left(\frac{E}{HV}\right)^{0.4} \times \left(\frac{c}{a}\right)^{-1.56} \quad (5)$$

where HV is the Vickers hardness (in GPa), α is the indent half-diagonal length (in m), E is the modulus of elasticity (in GPa), and c is the crack length from the center of the indentation to the crack tip (in m). The presented results are the average from 5 independent measurements made on 5 different specimens (n = 5).

The average values of the mechanical properties for each subgroup were calculated. To evaluate the statistical significance between the mechanical properties of the subgroups (A_1/A_2, and B_1/B_2), the t-test was used. In all tests, *P* values smaller than 5% were considered statistically significant (i.e., $P \leq .05$ was considered significant).

RESULTS

The typical appearance of the produced samples fabricated by CAD-CAM after the sintering procedure is shown in Figure 1C (here is a specimen from VITA YZ HT^{White}). The dimensions of all specimens satisfied the specifications of the ISO 6872 “Dentistry-Ceramic materials”.[13]

The microstructure of the polished and thermally etched zirconia specimens, before and after the heat treatment procedure (Table 2), is shown in Figure 2. The samples manifested a homogenous microstructure with no residual porosity. After the thermal treatment, the grain boundaries in all specimens were clearly visible. Nonetheless, the grain size was not uniform, since it ranged between 200 nm to 1 μm. More specifically, the mean values for Group A were ~500 nm and Group B ~450 nm. The heat treatment cycles (corresponding to ceramic mass firing, Table 2) did not affect the microstructure of the tested materials.

The X-ray diffractograms of all the produced samples are summarized in Figure 3. The peaks of all specimens were a good match to the standard patterns of crystalline yttria-stabilized tetragonal zirconia (01-070-4426 zirconia yttrium oxide). The strongest peak of the diffractograms was recorded at 30.16°, followed by three double peaks at 50.15° and 50.57°, 59.28° and 60.02°, and 34.60° and 35.15°, corresponding to the planes (101), (112), (200), (103), (211), (002), and (110) of tetragonal zirconia, respectively. According to the assignment of the peeks, there is no evidence of the formation of other secondary or minor phases, including monoclinic or cubic zirconia.

There is no shift in the peaks’ position after the heat treatment. Even so, the intensity of the peaks seems to slightly increase (insets of Figure 3), suggesting that the heat treatment may increase the crystallinity of the samples. Hence, to calculate the crystallite size, the peaks of the diffractograms were analyzed by the Scherrer equation

$$D = K\lambda / \beta \cos\theta \quad (6)$$

where D is the crystallite size (in nm), K is the Scherrer constant (here, it was set as 0.9), λ is the wavelength of the X-ray source (here λ = 0.15406 nm), β is the full width at the half maximum (FWHM), and θ is the peak

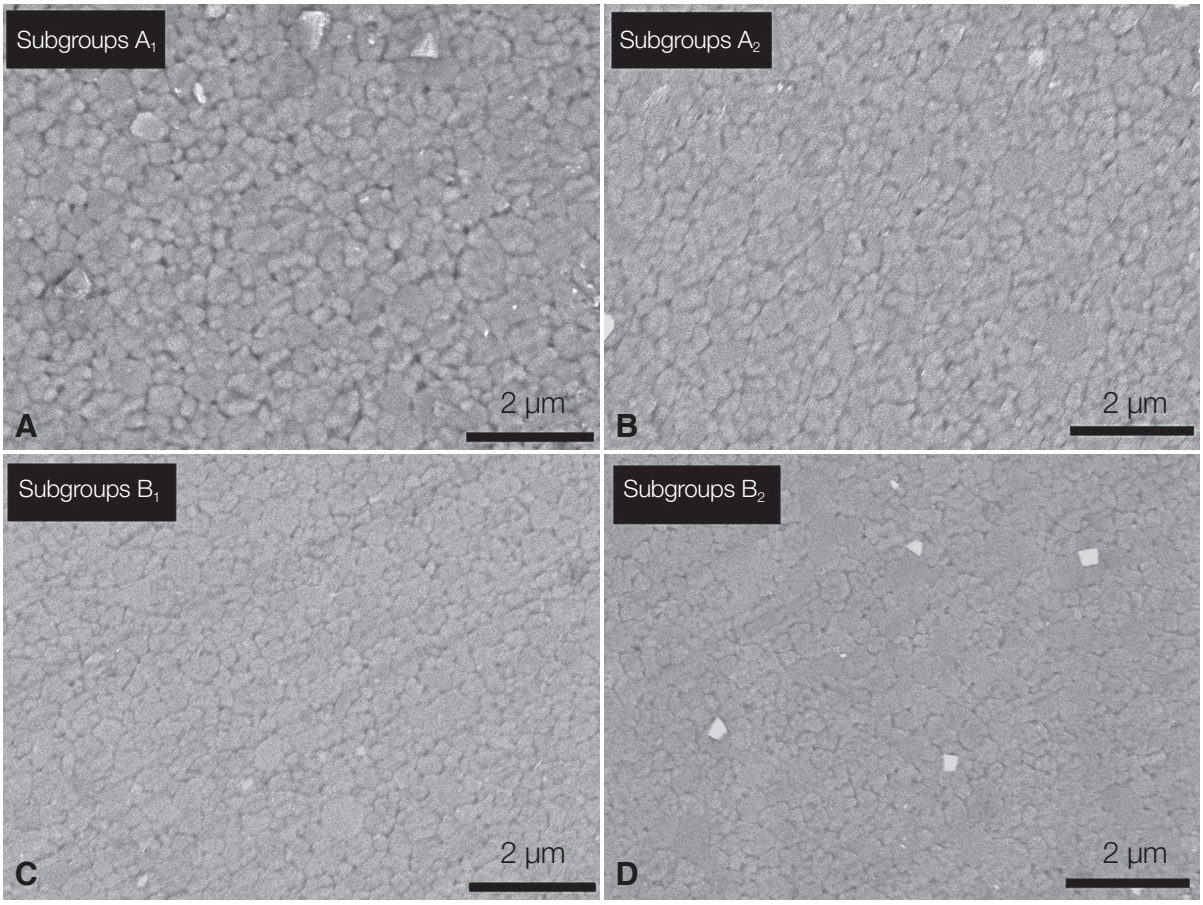

Fig. 2. Microstructure (after thermal etching) of zirconia specimens made of the two tested zirconia powders (i.e., pre-sintered blocks), before (A, C) (subgroups A_1 and B_1) and after (B, D) heat treatment procedure (subgroups A_2 and B_2) (see Table 2). (A) subgroup A_1, (B) subgroup A_2, (C) subgroup B_1, (D) subgroup B_2.

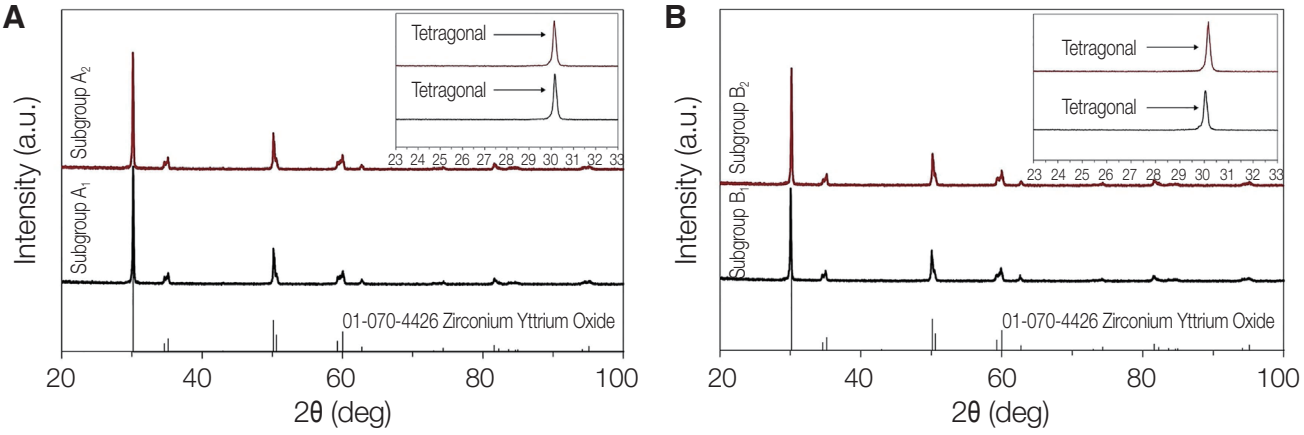

Fig. 3. X-ray diffractograms of zirconia specimens ((A) subgroups A_1 and A_2, and (B) subgroups B_1 and B_2) made of the two tested zirconia powders (i.e., pre-sintered blocks) before (subgroups A_1 and B_1) and after the heat treatment procedure (subgroups A_2 and B_2) (see Table 2). The standard patterns of crystalline yttria-stabilized tetragonal zirconia (card 01-070-4426) are also plotted at the bottom of the diagrams. The insets show an enlarged view of the main peak at 30.16°.

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position (in radians). The results showed that D increased from 34.66 nm for subgroup A_1 to 38.48 nm in subgroup A_2, and from 32.79 nm for subgroup B_1 to 36.43 nm in subgroup B_2, confirming the above hypothesis. In ceramics, the increase of crystallite size leads to more fragile materials.[19]

The density values varied between 6.0 - 6.2 g/cm^3 (SD is < 1%) for all the specimens and were not affected by the heat treatment procedure. The results of the mechanical properties measurements are summarized in Figure 4 and Table 3. Slightly poorer mechanical properties were generally recorded in the samples after the heat treatment procedure, which can be related to the increased crystallinity of the samples, mentioned above. However, statistical analysis revealed that there were no statistically significant differences in the values of the four mechanical properties (flexural strength, modulus of elasticity, Vickers hardness, and fracture toughness) between the subgroups before (A_1 and B_1) and after heat treatment (A_2 and B_2).

DISCUSSION

The experimental results confirmed that the properties of the zirconia (Y-TZP) samples are maintained after the milling process (i.e. the application of the CAD-CAM technology) and the four heat treatments applied to coat them in order to produce all-ceramic prosthetic restorations.

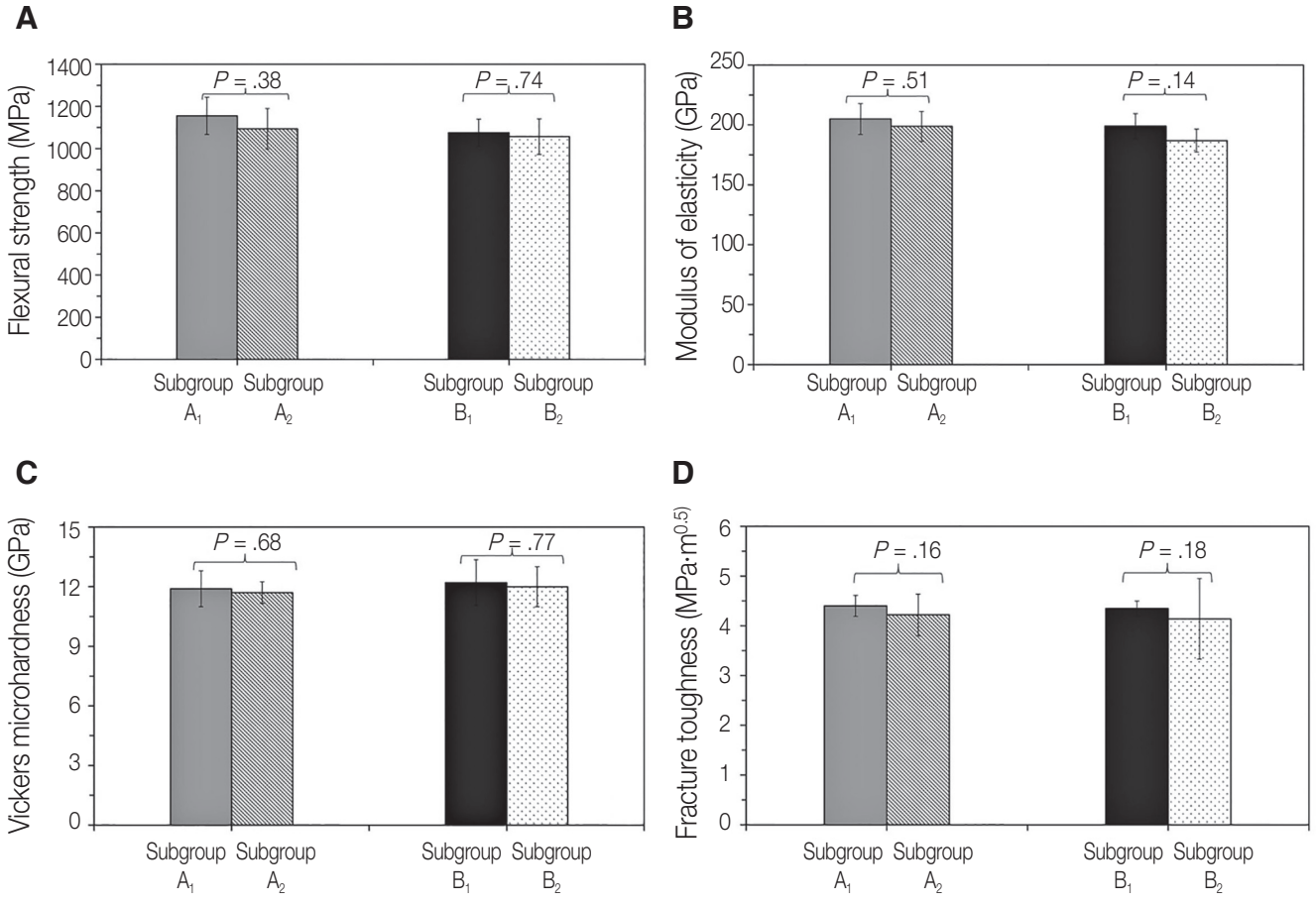

Fig. 4. Mean values (and standard deviation) of the mechanical properties, (A) flexural strength, (B) modulus of elasticity, (C) Vickers microhardness, and (D) fracture toughness, of the fabricated, by milling technology (CAD-CAM), zirconia ceramics, before (subgroups A_1 and B_1) and after heat treatment procedure (subgroups A_2 and B_2) (see Table 2). The *P* values calculated by t-test statistical analysis to compare the subgroups are also presented ($P \leq .05$ was considered significant).

Table 3. Mechanical properties (mean values and standard deviation) and statistical significance between the subgroups ($P \leq .05$ was considered significant)

	Subgroup A_1	Subgroup A_2	*P*	Subgroup B_1	Subgroup B_2	*P*
Flexural strength (σ, MPa)	1155 ± 88.2	1094 ± 96.5	.38	1075 ± 64.5	1057 ± 83.3	.74
Modulus of elasticity (E, GPa)	205 ± 12.9	198 ± 12.5	.51	199 ± 10.4	187 ± 9.6	.14
Vickers microhardness (HV, GPa)	11.9 ± 0.90	11.7 ± 0.55	.68	12.2 ± 1.15	12.0 ± 1	.77
Fracture toughness (K_{IC}, MPa · $m^{0.5}$)	4.40 ± 0.21	4.22 ± 0.14	.16	4.35 ± 0.15	4.14 ± 0.27	.18

More specifically, the present work aimed at evaluating the influence of milling technology and the repeated firing cycles on the properties of highly translucent dental zirconia restorations. Hence, the conditions of the experiments were carried out according to the instructions of the manufacturers of the materials used. The first heat treatment procedure, called the sintering procedure, is crucial for the clinical behavior of the prosthetic restoration in the oral cavity. According to the manufacturers' instructions, the sintering temperature was 1450°C for both groups A and B. According to Stawarczyk *et al*.,[20] the highest flexural strength values are recorded in zirconia samples sintered between 1400 and 1550°C. Sintering at temperatures above 1600 - 1650°C results in grain growth and leads to "overfiring" effects, such as defects in zirconia microstructure, e.g., hollow holes.

The second heat treatment procedure, called ceramic mass firing, was performed in the temperature range of 900 - 940°C. The experimental results showed that the spontaneous phase transformation of zirconia crystals from the tetragonal phase to the weaker monoclinic phase was effectively avoided.[4]

The microstructure analysis (Fig. 2) showed that the final zirconia samples had a fine microstructure, i.e. with no impurities, secondary phases, or porosity, and with submicron grains, regardless of the heat treatment, i.e. ~500 nm for Group A (and A_2) and ~450 nm for Group B (and B_2), suggesting good mechanical properties. It is also noteworthy that the heat treatment procedure (corresponding to ceramic mass firing) did not lead to grain pullout. Both heat treatment[21] and aging[22,23] procedures usually lead to grain pullout. This detrimental phenomenon increases the surface roughness, causing antagonist tooth abrasion and micro-crack formation.[22-24]

In addition, the ceramic mass firing caused no alterations in the crystallographic regime of zirconia, since tetragonal zirconia was exclusively registered in the X-ray diffractograms (Fig. 3). This is an important finding because the heat treatment cycles of the specimens took place at temperatures (> 900°C, Table 2) close to the temperature of phase transformation of tetragonal zirconia to the weaker monoclinic phase (~1000°C). Indeed, previous studies[25] have reported that in some Y-TZP ceramics used in the fabrication of fixed dental prostheses, weak peaks, assigned to monoclinic zirconia (between 28 - 29°),[15,22] were recorded the X-ray diffractograms together with the strong peaks of tetragonal zirconia as the main crystalline phase.

Apart from the good esthetics (Fig. 1C), which was expected for Y-TZP ceramics, the mechanical properties of the produced materials, summarized in Figure 4 and Table 3, have high importance in their potential clinical application in dentistry. Both esthetics and mechanical properties are direct results of the chemical composition, the microstructure, and the crystallographic regime of the prepared ceramics.[26,27] The ceramic mass firing procedure slightly weakened the mechanical properties, i.e., Vickers microhardness decreased from 11.9 to 11.7 GPa for Group A, and from 12.2 to 12.0 GPa for Group B. Similarly, the fracture toughness values were reduced from 4.4 to 4.22 MPa · $m^{0.5}$ (Group A) and from 4.35 to 4.14 MPa · $m^{0.5}$ (Group B), the flexural strength decreased

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from 1155 to 1094 MPa (Group A) and 1075 to 1057 MPa (Group B), and the modulus of elasticity was reduced from 205 to 198 GPa (Group A) and from 199 to 187 GPa (Group B). It is well known that ceramics (and glass-ceramics) with smaller crystallite size result in ceramics (and glass-ceramics) with better mechanical properties.[19] A typical example is the Empress glass-ceramic, which manifests very high mechanical properties, ascribed to their special nanostructure.[19] Accordingly, the small increases of the crystallite size, calculated for the samples after the ceramic mass firing procedure (subgroups A_2 and B_2), cause a slight decrease of the above mechanical properties. However, these differences were not statistically significant.

Moreover, the mechanical properties of the tested materials were very high, ascribed to the small average grain size of the zirconia specimens prepared, which was below the critical grain size of 1 μm (Fig. 2). It has been reported that 3Y-TZP particles with grain size > 1 μm are less stable and more susceptible to spontaneous phase transformation (from tetragonal to monoclinic).[28] This agrees with the XRD results in the present study since there was no evidence of tetragonal to monoclinic phase transformation after the heat treatment of all the specimens tested (Fig. 3). It is also important to note that, according to the ISO 6872,[13] a material with a minimum flexural strength of 500 MPa and fracture toughness of > 3.5 $MPa \cdot m^{0.5}$ are recommended for extensive fixed prosthetic restorations, such as (a) substructure ceramic for three-unit prostheses involving the molar restoration and (b) substructure ceramic for prostheses involving four or more units. The produced zirconia ceramic materials satisfy these requirements.

In the design of dental materials, the knowledge of the biomechanical regime in the oral cavity, which involves the knowledge of the mechanical properties of both the dental hard tissue (enamel and dentin) and the dental restorative material, is vitally important for the longevity and the ultimate success of dental restorative material, since the dental prosthetic materials are subjected to dynamically changing loads.[14,29-31] Ideally, the mechanical properties of the dental material should be a good match to those of the dental hard tissue, as shown in the present study (Table 4). Still, in the opposite case, a big mismatch of the above properties may cause injury to the dental hard tissue or fracture of the prosthetic restoration.[14]

More specifically, in all-ceramic prosthetic restorations, hardness reflects the ability of a material to resist a permanent indentation[21,32-35] and, therefore, its susceptibility to abrasive wear.[35] The most recent study of Solá-Ruíz *et al*.[36] reported on the wear sustained in the natural antagonist tooth in cases of monolithic zirconia tooth-supported crowns. Their results showed that the natural tooth antagonist to monolithic zirconia crowns undergoes significant wear over time, greater than the crown's wear. The wear rate depends on the position of the restoration (more common in molars than in pre-molars), gender (less frequent in women than in men), the surface treatment of monolithic zirconia crowns (glazed or polished), and the parafunctional habits of patients. Therefore, many research teams and manufacturers investigate the potential use of monolithic zirconia ceramics as a substrate material, which can be coated with another esthetic ceramic material with a lower hardness value (i.e., veneered zirconia with layered

Table 4. Comparison of mechanical properties of the tested materials with the corresponding values of natural tissues[14,41]

		Flexural strength (σ, MPa)	Modulus of elasticity (E, GPa)	Vickers microhardness (HV, GPa)	Fracture toughness (K_{IC}, $MPa \cdot m^{0.5}$)
Tooth hard tissues	Dentin	230 - 305	15 - 30	0.13 - 0.51	3
	Enamel	60 - 200 (260 - 290)*	70 - 100	3.0 - 6.0	1.0 - 1.5
Studied materials	Group A	1094 - 1155	198 - 205	11.7 - 11.9	4.40 - 4.22
	Group B	1057 - 1075	187 - 199	12.0 - 12.2	4.14 - 4.35

*: If supported by dentin.

porcelain). Unfortunately, this approach causes other problems, such as the bond strength at the interface between the two materials, i.e., the dental veneered zirconia and the esthetic ceramic material.[36]

At this point, consideration should be made on the importance of the magnitude of hardness in the case of zirconia restorations which are coated with esthetic ceramic mass. During the masticatory cycles, the cutting surface of the anterior tooth of the lower jaw comes in contact with the lingual surface of the teeth of the upper jaw. Thus, in the case of zirconia restorations of the upper anterior teeth, the lingual part of the restoration in cingulum area is often not covered by esthetic coating ceramic mass but consists of the zirconia substructure material (in order to provide stability to the esthetic ceramic material). Thus, abrasion of the natural opposite teeth in the biting edge can occur.

However, there are several studies which oppose the conclusions of Sola-Ruiz *et al*. For instance, Esquivel-Upshaw *et al*.[37] studied the wear of enamel antagonists against polished monolithic zirconia crowns, and they found that monolithic zirconia exhibited similar wear of enamel compared to metal-ceramic crowns after one year. Mundhe *et al*.[38] studied *in vivo* the wear of enamel opposing natural enamel, zirconia, and metal-ceramic crowns after 1 year and concluded that zirconia crowns led to less wear of antagonist enamel than metal-ceramic crowns, but more than natural enamel. Deval *et al*.[39] reported on a clinical comparative evaluation of the wear of enamel antagonist to monolithic zirconia and metal-ceramic crowns and found that that monolithic zirconia causes less wear of the antagonist tooth than feldspathic porcelain.

Another important mechanical property is fracture toughness, which measures the resistance of a material to crack propagation and to fracture. Assuming that the fracture toughness value defines the critical stress at which catastrophic failure occurs due to a micro-defect in the bulk of the ceramic material,[40] a low fracture toughness value implies poor clinical reliability of the prosthetic restoration. Nevertheless, the values of fracture toughness of the investigated materials are higher than the values of dentin and enamel (Table 4). Consequently, the produced ceramics should manifest good clinical reliability for prosthetic rehabilitation.

The modulus of elasticity estimates the rigidity of a material. Thus, in the case of extensive fixed prosthetic restorations, the modulus of elasticity and flexural strength play a decisive role in their longevity in the oral cavity. Moreover, in the case where zirconia ceramic is coated with another esthetic ceramic material (i.e., veneered zirconia with layered porcelain), higher values of modulus of elasticity and flexural strength favor the protection of the brittle surface of the ceramic material from fracture. In addition, materials with such mechanical properties provide the possibility for designing a prosthetic restoration with a minimum thickness; thus, less grinding of the natural tooth is needed.[20]

CONCLUSION

According to the experimental results obtained from the present *in vitro* study, it is concluded that:

- the microstructure, and the physical and mechanical properties of the dental highly translucent zirconia specimens fabricated with the CAD-CAM technology were not affected by the heat treatment procedure (i.e. the repeated firing cycles), and
- in the light of the specifications described in the ISO 6872 “Dentistry-Ceramic materials”, the milling technology is qualified as an effective and safe subtractive manufacturing technology for producing zirconia substructures for dental fixed all-ceramic prosthetic restorations, namely extensive fixed prosthetic restorations, such as substructure ceramics for three-unit prostheses involving the molar restoration and substructure ceramics for prostheses involving four or more units.

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