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Does Diamond Stone Grinding Change the Surface Characteristics and Flexural Strength of Monolithic Zirconia?

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Clinical Relevance

Dentists commonly need to perform grinding procedures to gain space when adapting a prosthesis; however, this grinding may induce damage to the surface. Thus, it is important to perform grinding that does not impair the properties, and the best way is without irrigation for conventional and monolithic zirconias.

SUMMARY

Purpose: The present study evaluated the effect of grinding on the surface morphology, mean roughness, crystalline phase, flexural strength, and Weibull modulus of monolithic (MZ) and conventional (CZ) zirconias.

Methods and Materials: CZ and MZ bars and square-shaped specimens were distributed into three subgroups, combining grinding (G) and irrigation (W) with distilled water: Ctrl (Control: no grinding, 20 × 4 × 1.2 mm and 12 × 1.2 mm), DG (dry grinding, 20 × 4 × 1.5 mm and 12 × 1.5 mm), and WG (grinding with irrigation, 20 × 4 × 1.5 mm and 12 × 1.5 mm). The grinding (0.3 mm) was performed on a standardized device using a low-rotation wheel-shaped diamond stone. The four-point flexural strength test was performed on the EMIC 2000 machine (5 KN, 0.5 mm/min). Scanning electron microscopy (SEM) was used to evaluate the surface morphology. An X-ray diffractometer (XRD) was used to obtain the crystalline structures that were analyzed by the Rietveld method. Flexural strength (FS) values were subjected to the Shapiro-Wilk test and two-way analysis of variance followed by the Tukey's test (for all tests, α=0.05).

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Results: Grinding, either with or without irrigation, did not change the FS of the MZ but increased the FS of the CZ. Both MZ and CZ showed similar morphologic patterns after grinding, and in the WG groups, the grinding was more aggressive. The MZ had greater monoclinic content in all groups; grinding without irrigation caused the smallest t→m transformation.

Conclusion: The grinding, when necessary, should be carried out without irrigation for conventional and monolithic zirconias.

INTRODUCTION

Since the 1960s, when Helmer and Driskell[1] published the first work on different biomedical applications of zirconia,[2] researchers and manufacturers have made admirable efforts to promote its clinical indications. Zirconia is a polymorphic material primarily with three phases, monoclinic (m), tetragonal (t), and cubic (c),[3,4] which changes according to thermo-mechanical factors that can impact the properties of the zirconia.

Initially, studies concentrated on the addition of oxides, such as yttria[5–8] to stabilize the material in the tetragonal phase at room temperature. Then, in the 1990s, the yttrium oxide–stabilized tetragonal zirconia polycrystal (Y-TZP) was indicated for dental use due to its high flexural fracture strength and its toughening process. Y-TZP also features biocompatibility, low radioactivity, interesting optical properties, high hardness, grinding resistance, good frictional behavior, an absence of magnetism, high corrosion resistance in acids and alkali, elasticity modulus similar to steel, and thermal expansion coefficient similar to iron.[2]

Among dental ceramics, Y-TZP has shown the best mechanical properties due to transformation toughening.[2–4,9] This property relates to zirconia's ability in the tetragonal phase to prevent the spread of small cracks. Compared with the dental tissues, this ability would be similar to the blockage provided by the interface of the dentino-enamel junction and the crystalline microstructure of the enamel.[10]

Due to these advantageous properties, Y-TZP was used for infrastructures of all-ceramic fixed crowns and prostheses, implants, abutments, and orthodontic brackets.[2,4,11] It is considered the most appropriate material to resist the high tension that affects multiple all-ceramic frameworks up to five elements.[11] However, there are contraindications such as those related to patients with poor oral hygiene, high caries activity, active periodontal disease and parafunction,[12,13] and failures related to marginal adaptation, delamination, and chipping.[14,15]

To solve delamination and chipping, improvements in the zirconia/veneer interface have been made,[16–20] such as the development of a zirconia without esthetic coating, named monolithic zirconia (MZ).[21,22] MZ was developed by adding different dopants and coloring liquids, changing the sintering temperatures, eliminating porosities, and changes in grain size,[21,23–25] resulting in greater transmittance and translucency.[24,25] In addition, MZ has good fracture resistance,[26] and its acceptable esthetics allows for use as veneers in posterior teeth[27] and requires less tooth removal because a smaller material thickness is sufficient.[28]

However, even with computer-aided design/computer-aided manufacturing technology, clinical adjustments through grinding are indispensable for obtaining adequate occlusal contacts in total monolithic zirconia crowns,[21,22] and such procedures increase surface roughness, as well as induce transformation t→m. Although these transformations make the zirconia surface more tenacious, over time it may have negative effects on mechanical stability depending on the total or partial loss of the materials' ability to prevent or delay the spread of cracks and could become critically unreliable.[12]

The available evidence on the influence of grinding/finishing on the flexural strength of zirconia is still contradictory.[29–38] Regardless of the grinding method used, changes in the material surface may favor or damage the mechanical properties depending on the quantity of t→m transformation, and zirconia with higher monoclinic content will be more subject to the impact of long-term degradation.[39]

The aim of the present study was to evaluate the effect of grinding on surface morphology, mean roughness, crystalline phase, flexural strength, and Weibull modulus in MZ and conventional zirconia (CZ). The first null hypothesis was that both zirconias have the same surface characteristics and the second was that both zirconias have the same mechanical properties.

METHODS AND MATERIALS

Specimen Preparation

After a pilot study, the sample size (n) for each test was calculated using α=0.05 and β=0.80. Two types of zirconia were used, one CZ (group CZ, control, ICE Zirkon, Zirkonzahn GmbH, Gais, Bolzano, Italy) and

Table 1: *Distribution and Nomenclature Adopted for the Experimental Groups*

Groups	Treatments
CZ	Conventional zirconia without grinding
CZDG	Conventional zirconia with dry grinding
CZWG	Conventional zirconia with wet grinding
MZ	Monolithic zirconia without grinding
MZDG	Monolithic zirconia with dry grinding
MZWG	Monolithic zirconia with wet grinding

the other MZ (group MZ, Prettau Zirkon, Zirkonzahn GmbH). As previously described, bars and square-shaped specimens were obtained[24,32] with the following dimensions: 25 × 5 × 1.5 mm and 15 × 1.5 mm for specimens that would not be ground (CZ and MZ groups) and 25 × 5 × 1.9 mm and 15 × 1.9 mm for specimens that would be ground.

Surface irregularities were removed with silicone tips (Exacerapol, Edenta, Labordental, Hauptstrasse, Switzerland), and a final polishing was performed with #1200, #1500, and #2000 silicon carbide abrasive papers (401Q, 3M ESPE, Tidal, SP, Brazil). Bar-shaped specimens had their edges beveled at 45°.

Sintering was performed according to the manufacturer's recommendations in a Zirkonofen furnace (600/V2, Zirkonzahn GmbH) with a heat treatment program of eight hours at a maximum temperature of 1500°C for the CZ and 8.2 hours at a maximum temperature at 1600°C for the MZ. The dimensions after the sintering (volumetric contraction ~20%) were 20 × 4 × 1.2 mm and 12 × 1.2 mm for the CZ and MZ groups and 20 × 4 × 1.5 mm and 12 × 1.5 mm for the groups that were ground. The final dimensions were measured using digital calipers (500-144B, Mitutoyo Sul Americana, Suzano, SP, Brazil).

The specimens that would be ground were further subdivided into two subgroups: grinding without irrigation (dry grinding [DG]) and grinding with irrigation (wet grinding [WG]). The experimental groups' distribution and nomenclature adopted are shown in Table 1.

Grinding Procedure

A standardized grinding apparatus capable of controlling the amount of grinding and rotation speed was used.[24,32] The surface grinding (0.3 mm) was performed with a diamond stone of medium grit (MasterCeram, 133-104-SDN, Polierwerk, Straubenhardt, Baden-Württemberg, Germany) in a low-speed electric handpiece (Micro Electric Motor Bench for Prosthetics LB 100, Beltec, Araraquara, SP, Brazil) at 20,000 rpm; specimens from WG were ground under distilled water flowing from a triple syringe. After grinding, all specimens possessed 1.2-mm thicknesses. Before the tests, all specimens were cleaned in an ultrasonic bath using distilled water (five minutes) and isopropyl alcohol (five minutes).

Surface Morphology

Two specimens of each group were sputter-coated with gold. Overall surface morphology was characterized by scanning electron microscopy (SEM, JEOL JSM-6610LV, Akishima, Tokyo, Japan) with secondary electrons.

Mean Surface Roughness

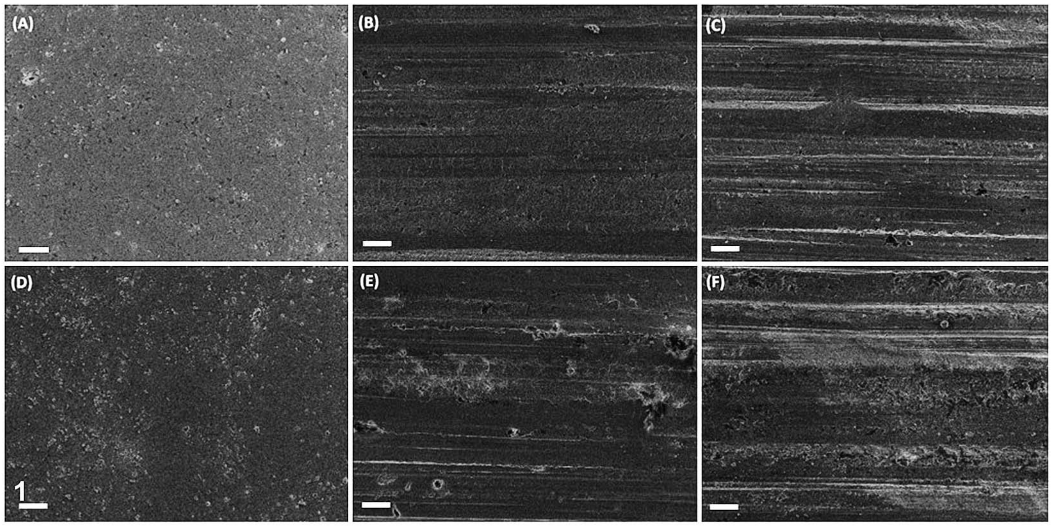
Mean surface roughness values (Ra, μm) were determined in square-shaped specimens (n=13) using a profilometer (Mitutoyo SJ 400, Mitutoyo Corp, Yokohama, Japan) with an accuracy of 0.01 μm, active tip radius of 5 μm, and speed of 0.5 mm/s at three equidistant different locations on one side of the specimens. To respect the cutoff, an λC of 2.5 mm was adopted for the WG groups and 0.8 mm for the other groups; the limited Ra for the cutoff was 2 μm. The measurement locations were the same for all specimens: one in the center and the others equidistant (5 mm). The measurements were performed in the opposite direction of the grinding lines for the groups submitted to this procedure.

Crystallography

To identify the crystalline structure, one specimen from each group was analyzed in a X-ray diffractometer (DRX-6000, Shimadzu do Brazil) using Cu-Kα radiation, 2θ scan between 10° and 80°, an angular step of 0.05°, and a step time of three seconds. The crystalline phases were identified by comparison with standard microfiche of the International Centre for Diffraction Data JCPDS files.

Flexural Strength and Weibull Modulus

Flexural strength (FS, n=13) was carried out by a four-point device attached to the EMIC 2000 testing machine (Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, Paraná, Brazil) with a 5-kN load cell and speed of 1 mm/min (ISO 6872:2008). The FS values were calculated according to the following formula: $\sigma = 3PL/4wb^2$, where σ = flexural strength, in MPa; P = force at the moment of the



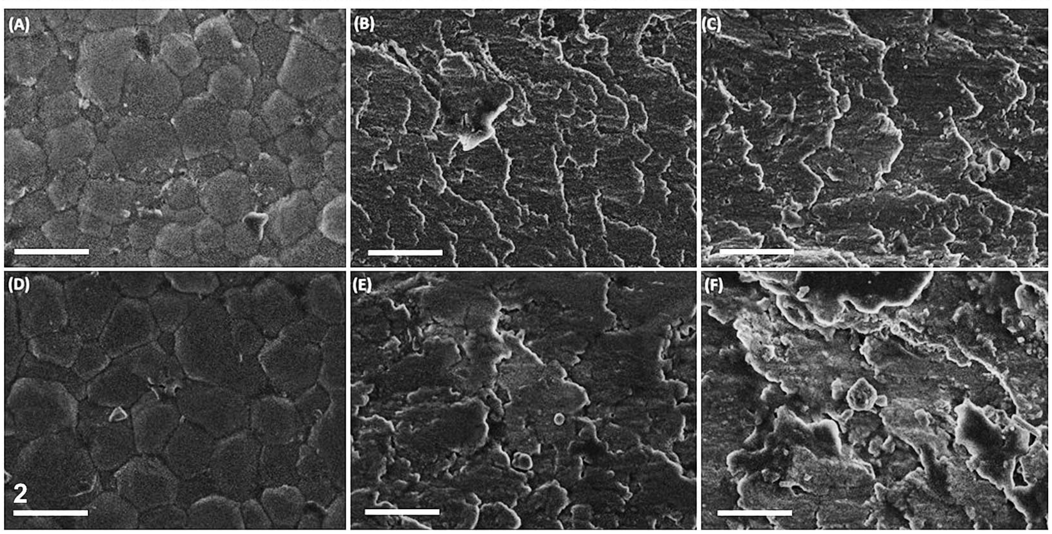


Figure 1. *Surface morphology of the groups at 1000× magnification: (A) CZ, (B) CZDG, (C) CZWG, (D) MZ, (E) MZDG, and (F) MZWG. In A and D, the morphology reveals a flat surface without scratches. In B, C, E, and F, the presence of grooves could be seen that follows the grinding direction and plastic deformation.*

Figure 2. *Surface morphology of the groups at 25,000× magnification: (A) CZ, (B) CZDG, (C) CZWG, (D) MZ, (E) MZDG, and (F) MZWG. In A and D, the morphology reveals similarity in grain conformation with slightly larger grains in MZ. In B, C, E, and F, the presence of scales, loss of grain disclosure, and plastic deformation could be seen, being more evident in C and F. E and F shows bigger scales.*

fracture in newtons; L = the distance between the outer supports in millimeters; w = the width of the specimen in millimeters; and b = the thickness of the specimen in millimeters.

The reliability of the materials was calculated by the determination of the Weibull modulus (m). The equation $P(\sigma) = 1 - \exp(-\sigma/\sigma_0)^m$ was applied to calculate the Weibull modulus, where $P(\sigma)$ is the fracture probability, σ is the fracture strength at a given $P(\sigma)$, σ_0 is the characteristic strength, and m is the Weibull modulus, which is the slope of the 1n (1n 1/1 − P) versus σ plots.[30,40–42]

Statistical Analysis

The SEM images were submitted to descriptive analysis, and XRD data were analyzed by the Rietveld method. Ra and FS were submitted to normality and homoscedasticity tests and subsequently to the analysis of variance (ANOVA) two-way test (α=0.05) followed by the Tukey's *post hoc* (α=0.05) in the BioEstat 5.0 software.

RESULTS

The micrographs are depicted in Figures 1 and 2. In the smallest magnification (1000×), the surface

Table 2: *Means and SDs of Mean Roughness (Ra, in μm)[a]*

Treatments	Groups	
	CZ	MZ
—	0.14 ± 0.02 Ca	0.15 ± 0.03 Ca
DG	1.49 ± 0.46 Ba	1.53 ± 0.36 Ba
WG	2.79 ± 0.59 Ab	3.26 ± 0.43 Aa

[a] *Different lowercase letters indicate statistical difference between columns and uppercase between lines (p<0.05).*

Table 3: *Volume Fraction (%) of the Monoclinic (M), Tetragonal (T), and Cubic (C) Phases in Y-TZP*

Treatments	Groups					
	CZ			MZ		
	T	M	C	T	M	C
—	76.23	7.20	16.57	69.12	10.76	20.12
DG	94.26	5.74	0.00	92.44	7.56	0.00
WG	89.64	10.36	0.00	85.71	14.29	0.00

morphology of the CZ and MZ revealed similarity (Figure 1A,D), but when analyzed in the highest magnification (25,000×), it was possible to visualize slightly larger grains. Grinding caused grooves that followed the grinding direction (Figure 1B,C,E,F), as well as loss of grain disclosure and the presence of scales and plastic deformation (Figure 2B,C,E,F). These changes were more evident when irrigation was done: with a surface with aggressive cuts, deeper and irregular valleys (Figure 1C,F), and larger scars (Figure 2C,F). In addition, when comparing the types of zirconia, it is possible to visualize that the changes in the monolithic zirconia were more evident (Figures 1E,F and 2E,F).

The means and standard deviations of Ra, in micrometers, are shown in Table 2. Statistically significant differences were observed among the treatments ($p<0.05$), indicating the lowest value of Ra for the no grinding groups and the highest value for the groups with wet grinding. Between zirconias, the statistical difference was observed just in the WG group ($p<0.05$).

The quantification of the crystalline phases of the samples is provided in Table 3. The CZ and MZ showed all three phases: tetragonal (T), monoclinic (M), and cubic (C). In both zirconias, grinding (with or without irrigation) transformed the entire cubic phase in the tetragonal phase of the specimens' surface. WG in both zirconias presented a more monoclinic phase than the other groups.

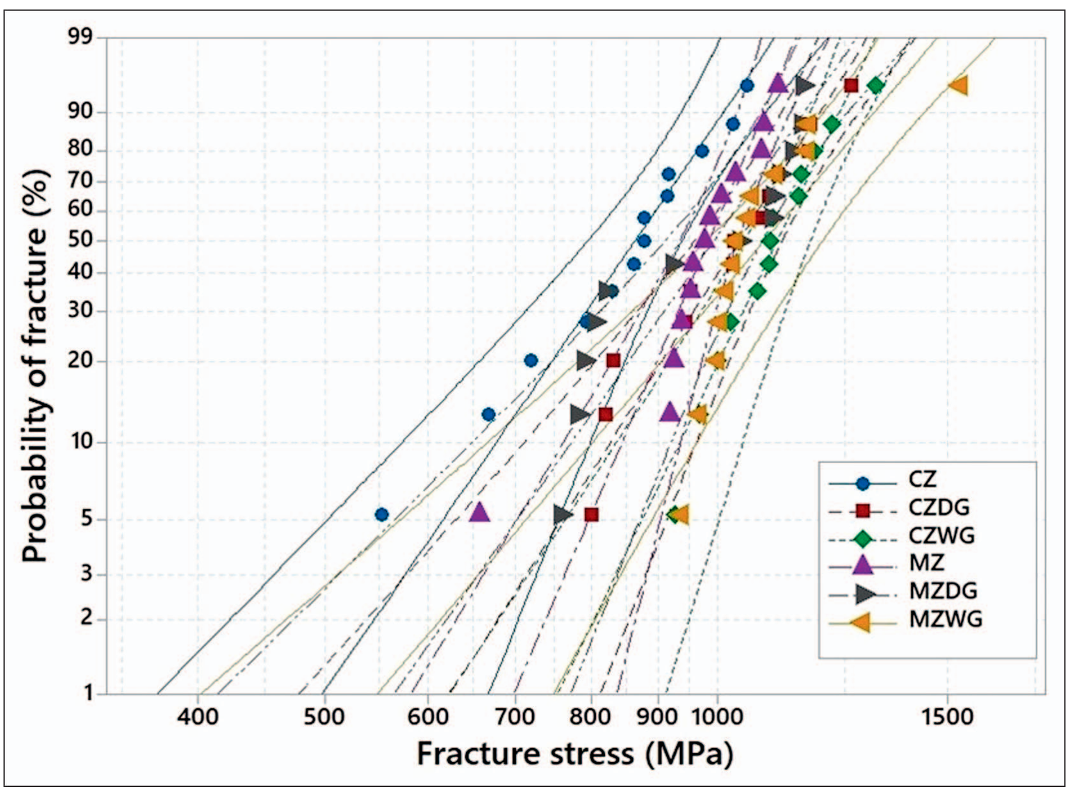


Figure 3. *Weibull plot for fracture data of the groups.*

Table 4: *Mean FS (in MPa), Weibull Modulus (m), and Respective 95% CI[a]*

Treatments	Groups							
	CZ				MZ			
	FS	95% CI (FS)	m	95% CI (m)	FS	95% CI (FS)	m	95% CI (m)
—	851.2 Ba	783.1-925.3	7.7 Aa	5.0-11.9	971.7 Aa	921.6-1024.5	12.3Aa	8.0-19.1
DG	1021.99 Aa	945.53-1104.64	8.3 Aa	5.4-12.8	977.99 Aa	897.25-1066.0	7.5Aa	4.8-11.8
WG	1096.07 Aa	1032.27-1163.81	10.9 Aa	7.3-16.3	1069.75 Aa	965.94-1184.71	6.2Aa	4.3-8.9

[a] Different lowercase letters indicate statistical difference between columns and uppercase between lines ($p<0.05$).

Table 4 shows the mean FS (in MPa), the Weibull Modulus (m), and the respective confidence intervals (95% CI). Figure 3 shows the Weibull plot. Statistically, there was no interaction between groups ($p=0.08$) for the FS and no difference between the two types of zirconias ($p=0.56$). Grinding did not change the FS of the MZ ($p=0.10$) but increased the FS of the CZ ($p<0.05$). There was no difference between dry or wet grinding ($p>0.05$). No significant statistical difference was found for the Weibull modulus comparisons.

DISCUSSION

MZ has recently been introduced into the market to eliminate the delamination and chipping, which are the main problems related to CZ. The indication of MZ significantly increased in situations where the interdental space is insufficient causing fractures in the crown.[43] Nevertheless, it is common that dentists need to perform grinding procedures to gain space when adapting a prosthesis,[44,45] and this grinding may induce damage to the surface.[46] It is fundamental to understand if grinding procedures could damage the MZ and how. Thus, the present study evaluated the effect of grinding on flexural strength, surface morphology, and the crystalline phases of an MZ compared with a CZ. For the surface characteristics, the null hypothesis was rejected because all the properties changed after the experimental treatments. For the mechanical properties, the null hypothesis was partially accepted because the Weibull modulus of both zirconias and the monolithic flexural strength did not change after the treatments.

When zirconia is ground, the introduced surface defects can become microfissures, ie, grooves that follow the main direction, some with lateral projections and even cracks depending on the rotary cutting tool type, grit-size, load, and applied speed).[33] In the present study, both CZ and MZ showed similar morphologic patterns after grinding, with more pronounced changes in MZ (Figures 1 and 2). This difference could be attributed to the microstructural differences between the types of zirconia.[21,23–25] In both types of zirconia, more changes could be observed in the WG subgroup (Figures 1C,F and 2C,F). According to Candido and others,[32] this result could be attributed to a higher cutting performance because water could remove dust that impregnates on the rotary cutting tool. These surfaces also showed superficial plastic deformation that hides grain boundaries, as reported by Preis and others.[22] Previous studies[31,34,47] have shown the same images patterns.

The present study also evaluated zirconia-grinding effects on the FS. The results (Table 4) indicate that grinding did not significantly change the MZ values of the FS, both in wet (MZWG 974.43±164.62 MPa) and in dry (MZDG 1082.31±152.40 MPa) conditions. On the other hand, there were significant changes in the FS values of the CZ after grinding: an increase of approximately 20% in the CZDG and 29% in the CZWG. These differences could probably be explained due to the larger cubic content present in the MZ surfaces (MZ=20.12% and CZ=16.57%). Surfaces with more cubic phases would be expected to have less residual stress than those fully tetragonal with equal grain sizes; therefore, the direct result of this minor residual stress is a reduction in the force to dissipate the $t \rightarrow m$ transformation, resulting in a stabilization of the tetragonal grains.[48] This phenomenon was not observed in the CZ group where the FS increased after grinding. The Weibull modulus (Table 4; Figure 3) did not have a statistical difference for all zirconias and between the treatments, which means that the reliability[30,40–42] of the materials and treatment were equal.

Different results were reported in the literature. Pereira and others[35] obtained higher values of FS for the MZ after grinding with a coarse diamond bur (181 μm); also, Guilard and others[33] obtained these when the MZ was ground with an extra fine diamond bur (30 μm) compared with coarse diamond bur (150 μm) grinding. Regarding the CZ, although dry and wet grinding increased the FS in the present study, corroborating studies of other authors,[32,34] a large number of papers reported contradictory results.[29–31]

In general, it can be assumed that for both CZ and MZ, changes in FS depend on several factors, such as the rotary cutting tool type and its grit size, the size of zirconia grains, changes in crystalline structure, and surface roughness.[49,50] According to Kosmac and others,[29] for the CZ, there is a decrease in FS after grinding with diamond burs and a negative correlation between the FS and the size of zirconia grains. Pereira and others[31] also evaluated the effect of grinding with a diamond disc and bur, verifying that FS increased after grinding for high and low grit size burs, but decreased when larger grit size discs were used. For Polli and others[51] and Hatanaka and others,[52] the grinding with a diamond bur (151 μm) was also responsible for the increased FS.

Another source of influence on the FS would be the surface roughness. The roughness data (Table 1) corroborate the morphologic differences observed in the micrographs (Figures 1 and 2). It can be observed that surfaces with more pronounced grooves and plastic deformation showed higher Ra values. When ground without irrigation, the zirconias did not show differences, but with irrigation, the MZ showed higher roughness. This alteration may be due to the association of severe grinding with the microstructural differences. Moreover, the literature shows that ceramic roughness had a negative correlation with FS[53]; however, in the present study, grinding surfaces with higher Ra (Table 2) did not show any changes (MZ) or even exhibited higher flexural strength (CZ). The Ra-FS relation should be interpreted with caution because the phase transformation toughening mechanism and the depth of the introduced cracks may have a counter-balance effect, excluding the effects of roughening.[54]

It is important to evaluate whether grinding changes the zirconia crystalline structure, because grinding can induce a phase transformation, whereas it can have a negative or a positive impact on the mechanical properties, and it depends on the volume percentage of the t→m transformation and on the conversion's metastability.[49,50] Regarding the quantification of the crystalline structure (Table 3), it is observed that wet grinding was responsible for higher t→m transformation, indicating a rougher procedure. These findings were similar to the results of Subaşı and others,[47] Pereira and others,[31] Preis and others,[22] and Mohammadi-Bassir and others[34] but differ from those found by Polli and others[51] who carried out the grinding in high rotation with a diamond bur, and this difference could be attributed to the grinding severity that was directly related to the amount of t→m transformation.[2,50] In addition, Guilard and others[33] observed that the higher the grit size of the diamond bur, the greater the t→m transformation. Candido and others[32] assumed that this transformation occurs after the surface was ground, because the energy generated by this procedure modifies the structure of the tetragonal grain, causing it to increase in size and become monoclinic. The transformation to a monoclinic phase toughens the zirconia surface.[49,50] The MZ showed more balanced phase stability, because during dry grinding, there was a decrease in the order of 30% in relation to the initial monoclinic content against 21% that occurred when the conventional zirconia was dry ground. When irrigation was used, there was an increase of 32% of monoclinic content for the ZM and 43% for the ZC.

The present study has some limitations such as the static loading for the FS and the specimen geometries that do not reproduce intraoral conditions. Further clinical investigations with dynamic loadings and a specimen with crown design are indicated to better understand the effects of grinding for the mechanical properties of Y-TZP. Chairside adjustments of the monolithic zirconia will still be necessary to optimize the emergence profile, improving adaptation and occlusion.[33] The formation of the monoclinic phase decreases the mechanical properties and its stability in a humid medium.[3] Thus, and based on the results of the present work, the dentist should guide the adjustment procedures to be carried out in low-speed rotation without irrigation to reduce the monoclinic and cubic content.

CONCLUSION

The grinding procedure with a diamond stone, either dry or wet, did not change the monolithic zirconia flexural strength; however, it was able to increase the flexural strength for the conventional zirconia. Grinding with irrigation was rougher, principally for monolithic zirconia. It was concluded that when a diamond stone was used, grinding without irrigation (dry) should be carried out for both zirconias.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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