©Operative Dentistry, 2018, **43-3,** E119-E128

Mechanical and Surface Properties of Monolithic Zirconia

LM Candido • LN Miotto • LMG Fais • PF Cesar • LAP Pinelli

Clinical Relevance

Restorations produced with monolithic zirconia are frequently used to replace those made with conventional zirconia and veneering porcelain. However, for correct use, it is important to know key material features, such as mechanical strength and fractographic behavior.

SUMMARY

Purpose: This study compared monolithic zirconia with conventional ones based on mean roughness (Ra), Vickers hardness (VHN), topography, transmittance, grain size, flexural strength (FS), Weibull modulus, and fractographic behavior.

Methods and Materials: One monolithic (Prettau Zircon [PR group]) and two conventional (ICE Zirkon Transluzent [IZ group] and Bloom-Zir [BL group]) zirconias were used. Specimens were tested using a profilometer, a microhardness tester, a scanning electron microscope, a spectrophotometer, and a Universal Testing Machine (EMIC DL 2000). Ra, VHN, grain size, and transmittance were analyzed using the Kruskal-Wallis test associated with Dunn test (α=0.05). FS was analyzed using one-way analysis of variance with the Tukey honestly significant difference test (α=0.05).

Results: Means and standard deviations of roughness, after sintering (Ra, in μm) and VHN, were, respectively, 0.11 ± 0.01, 1452.16 ± 79.49, for the PR group; 0.12 ± 0.02, 1466.72 ± 91.76, for the IZ group; and 0.21 ± 0.08, 1516.06 ± 104.02, for the BL group. BL was statistically rougher (*p*<0.01) than PR and IZ. Hardness was statistically similar (*p*=0.30) for all groups. Means and standard deviations of FS (in MPa) were 846.65 ± 81.97 for the PR group, 808.88 ± 117.99 for the IZ group, and 771.81 ± 114.43 for the BL group, with no statistical difference (*p*>0.05). Weibull moduli were 12.47 for the PR group, 7.24 for the IZ group, and 6.31 for the BL group, with no statistical differences. The PR and BL groups had higher transmittance values and grain sizes than the IZ group (*p*<0.05). Although the BL group had some fractures that originated in the center of the tensile

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DOI: 10.2341/17-019-L

surface, fractographic analyses showed the same fracture pattern.

Conclusions: All tested zirconia showed similar VHN, and the monolithic zirconia had similar roughness compared to one of the conventional zirconias. In addition, the monolithic zirconia showed similar flexural strength and Weibull modulus compared to the others even though its mean grain size was larger. The total transmittance of monolithic zirconia was higher than only one of the conventional zirconias tested.

INTRODUCTION

More than a decade ago, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was introduced in dentistry as a framework material that, for esthetic reasons, needs to be veneered with glass-ceramic or feldspathic porcelain.[1-3] Since then, zirconia has been widely used in various clinical situations due to its high esthetic potential, high biocompatibility, good dimensional and chemical stability, and high fracture toughness when compared to other dental ceramics.[4-6]

However, some clinical problems emerged with the use of Y-TZP, as it requires being veneered due to its high opacity.[7,8] Unfortunately, the veneering layers are prone to fracture[9-11] and have been associated with delamination and chipping.[2,7,12-16] Apart from significant improvements related to heating and cooling rates during porcelain sintering,[1,7,17,18] better framework designs,[7,19] and higher uniformity of the veneering layer (thickness),[7,20] Pjetursson and others[21] reported that 15% of Y-TZP restoration replacement occurred due to delamination and 20% due to chipping after five years of clinical follow-up.

In order to eliminate the weak veneering layer, monolithic zirconia restorations with higher translucency were developed by means of adding different dopants, coloring liquids, and changing the sintering temperatures.[7,8] Monolithic zirconia is a unique ceramic system with multiple clinical applications, including those with high esthetic demands,[22] and it is more easily processed than bilayered ones, having lower final cost.

Monolithic restorations are manufactured using computer-aided design/computer-aided manufacturing technology. Since these restorations are not veneered with porcelain, they can be finished by means of either polishing or applying a glaze layer.[2,19] In comparison to veneered restorations, full-contour zirconia restorations have the clinical advantage of allowing production of prostheses with significantly reduced thickness (only 0.5 mm for posterior restorations).[19,23-27]

Multiunit, monolithic prostheses need further investigation to determine if they can withstand as much chewing force as prostheses using regular Y-TZP as their framework. In this regard, determination of the flexural strength of ceramic materials can be helpful, especially after verifying whether the obtained strength meets strength standards, such as ISO 6872.[28] Flexural strength data of ceramic materials should preferably be analyzed using Weibull statistics, which describes the asymmetrical strength distribution resulting from the flaw population in the material microstructure.[29-32] In this analysis, the most commonly used parameter is the Weibull modulus (m), which is a measure for the scatter of strength data.[32,33] In addition to obtaining flexural strength data, it is important to carry out a descriptive fractographic analysis, which helps identify the failure origin and provides information about the loading conditions.[34]

Although monolithic zirconia was developed to overcome the limitations of conventional zirconia, comparisons between these two materials regarding their mechanical and optical properties are still scarce. Density, porosity, grain size, and the chemical nature of the material influence not only the optical but also the mechanical properties.[6,8,12,35,36] Thus, the aim of this study was to compare one monolithic zirconia material with two conventional zirconia materials in terms of their mean roughness (Ra), Vickers hardness (VHN), topography, transmittance, flexural strength, Weibull modulus, and fracture mode. The null hypothesis was that there would be no difference among the monolithic and conventional materials for any of the properties evaluated.

METHODS AND MATERIALS

Three commercially available zirconia ceramics were used: Prettau Zircon (PR group, n=15; Zirkonzahn GmbH, Gais, Italy), ICE Zirkon Transluzent (IZ group, reference group, n=15; Zirkonzahn), and BloomZir (BL group, n=15; Bloonden, Bioceramics Co, Hunan, China). PR is monolithic zirconia; IZ and BL are conventional zirconia. IZ and PR are different zirconias from the same manufacturer, and BL is relatively new on the market and, according to the manufacturer, is a high-translucency conventional zirconia. Sample size was calculated after a pilot study considering $\beta = 0.80$ and $\alpha = 0.05$ for all tests.

Bar-shaped specimens (25×5×1.5 mm) were cut from presintered blocks using a high-precision sectioning saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) with a low-speed diamond disk (Series 15LC Diamond, Buehler) under water cooling. A calibrated operator manually polished the bars on all sides using #1200, #1500, and #2000 SiC papers (401Q, 3M, Sumaré, Brazil). A chamfer on the edges was made using rubber tips (126c, Edenta, Labordental, São Paulo, Brazil) according to ISO 6872.[28]

The bars were sintered in a furnace (Zirkonofen 600/V2, Zirkonzahn) following the manufacturer's instructions. The IZ and BL groups were sintered for eight hours at 1500°C, and the PR group was sintered for 8.2 hours at 1600°C.

Mean roughness (Ra, μm) values were determined for all specimens with an accuracy of 0.01 μm using a profilometer (Mitutoyo SJ 400, Mitutoyo Corp., Yokohama, Japan) with length of 2.5 mm, active tip radius of 5 μm, and speed of 0.5 mm/s at three different locations on each side of the specimen. The measurement locations were the same for all specimens, one in the center and the others equidistant (~5 mm) from the center, resulting in six measurements per specimen. The roughness measurements were made before ($Ra_{presintered}$) and after ($Ra_{sintered}$) the sintering process.

VHN was measured for all specimens using a microhardness tester (MMT-3, 1600-6300, Buehler) with a load of 500 gf applied for 30 seconds at four different regions to obtain an average for each bar.

Flexural strength (FS) was assessed for all specimens using a universal testing machine (EMIC DL 2000, Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, Brazil) with a four-point bending design (5 kN, 1 mm/min) in accordance with ISO 6872.[28] The FS values were calculated using the formula $\sigma = 3PL/4wb^2$, where σ = flexural strength in MPa, P = force in newtons at the moment of the fracture, 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 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 $1n$ ($1n$ $1/1 - P$) vs σ plots.[33,37-39]

For fractographic analysis, after the FS test, all specimens were cleaned in an ultrasonic bath using distilled water (five minutes) and isopropyl alcohol (five minutes), dried, and examined using a stereomicroscope (CCD, Olympus, Center Valley, PA, USA) in order to identify the fracture origin and to confirm whether the fracture started due to the flexural force. Magnifications ranged from 1× to 5×, and the illumination angle was changed many times to favor observation of crack features. A preliminary observation indicated areas of interest for further examination under scanning electron microscopy (SEM). To illustrate the microstructural features and analyze the fracture,[40] five representative specimens per group were cleaned again, dried, sputter coated with gold,[41] and observed under SEM (SM-300, Topcon, Tokyo, Japan) with magnifications from 300× to 4000×. Other micrographs (5000× and 10,000×) were performed for microstructural characterization of the surface specimens. For better identification of voids, a high-contrast and high-brightness image was made.

Transmittance measurements were made in five specimens per group using a spectrophotometer (CM 3700d, Konica Minolta, Singapore) in transmittance mode, with wavelengths ranging from 360 to 740 nm at intervals of 10 nm. The total transmittance (T) was calculated according to

$$T(\%) = (L_{specimen}/L_{source}) \times 100,$$

where L is the luminance of the specimen and of the source, respectively. L_{source} was obtained by making one measurement of L without any specimen placed in the optical path, resulting in an L_{source} value of 30,000. This value corresponded to 100% of transmittance and served as the baseline for calculations.

The Feret method was used to compare the zirconia grain sizes and the transmittance results.[42] The grains were measured in the 10,000× SEM micrographs (n=3) using Image J software (National Institutes of Health, Bethesda, MD, USA).

All data were submitted to the normality and homoscedasticity tests. Ra, VHN, and FS were analyzed using one-way analysis of variance (α=0.05) and the Tukey honestly significant difference test (α=0.05). The paired t-test (α=0.05) was used to compare the Ra before and after sintering. Grain size and transmittance data were analyzed

Table 1: *Means and Standard Deviations for Roughness ($Ra_{presintered}$ and $Ra_{sintered}$ in μm) and Vickers Hardness (VHN) According to the Experimental Groups*

Groups	$Ra_{presintered}$	$Ra_{sintered}$	VHN
PR	0.08 ± 0.01 Ba	0.11 ± 0.01 Ab	1452.16 ± 79.49 a
IZ	0.08 ± 0.01 Ba	0.12 ± 0.02 Ab	1466.72 ± 91.76 a
BL	0.07 ± 0.01 Ba	0.21 ± 0.08 Aa	1516.06 ± 104.02 a

Different lowercase letters indicate significant differences ($p<0.05$) among rows. Different uppercase letters indicate significant differences ($p<0.05$) among columns.

using the Kruskal-Wallis test associated with Dunn ($\alpha=0.05$).

RESULTS

The means and standard deviations for roughness ($Ra_{presintered}$ and $Ra_{sintered}$) and VHN are shown in Table 1. There was no significant difference among the roughness values before the sintering process ($p=0.10$); however, sintering increased roughness for all groups ($p<0.05$) with significant differences among groups ($p<0.01$). The BL group was rougher than the other groups, and PR and IZ showed statistically similar roughness. VHNs were statistically similar ($p=0.30$).

The means and standard deviations for FS (MPa), the Weibull (m) statistical analysis, and respective confidence intervals (95%) are shown in Table 2 and Figure 1. There was no statistical difference between groups ($p>0.05$) for FS as for the Weibull modulus (m) (all confidence intervals overlapped).

The first- and third-quartile percentages of spectral transmittance (T%), as well as the median, are shown in Table 3 and in Figure 2. The PR and BL groups had similar transmittance percentages, which were statistically higher than those obtained for IZ (Table 3).

The SEM micrographs to identify fracture origin are shown in Figures 3 and 4. The hackle lines, mirror, origin, and direction of crack propagation are highlighted in these figures. The greatest magnification images (from 300× to 4000×) were chosen to show the fracture origin; thus, the compression curl does not appear, but an asterisk (*) was placed on the top side of the specimen in the direction of the compression curl for better understanding. All specimens had the compression curl on the top side where the load was applied and the origin of the fracture at the bottom, being the tension force responsible for the fracture.

The majority of the specimens showed fracture origin on the tensile side of the specimen near its corner, and the direction of crack propagation went from corner to center and top of the specimen (Figure 3). The same fracture pattern was observed for all groups, although some fractures originated at the center of the tensile surface for the BL group, being the direction of crack propagation from center to sides and top of the specimen (Figure 4).

Figure 5 shows SEM images at 5000× magnification; it is possible to note that the grains of the PR (a) and BL (c) groups were larger than those of the IZ group (b). The surfaces of the IZ and PR groups were more homogeneous, with rounded grains, all at the same level related to the surface. On the other hand, the grains of the BL group were much more heterogeneous, with grains at different surface levels. Figure 6 shows a high-brightness and high-contrast image (10,000×) with identification of voids. It can be seen that IZ group presented less voids, while PR and BL showed similar quantities.

Figure 6a through 6c were used to apply the Feret method. Table 4 shows the median and the first and third quartile of grain size (in micrometers). PR and BL showed similar mean grain sizes, which were significantly larger than the mean size obtained for IZ.

DISCUSSION

This study aimed to compare the mean roughness, VHN, topography, transmittance, grain size, flexural

Table 2: *Mean Flexural Strength (FS [MPa]), Weibull Modulus (m), and Respective Confidence Intervals (CI = 95%)*

Groups	FS (MPa)	95% CI (FS)	*m*	95% CI (*m*)
PR	846.65 ± 84.85 a	806.48-890.39	12.47 a	8.30-18.72
IZ	808.88 ± 117.99 a	741.79-874.69	7.24 a	5.01-10.47
BL	771.81 ± 114.43 a	696.65-840.09	6.31 a	4.47-8.92

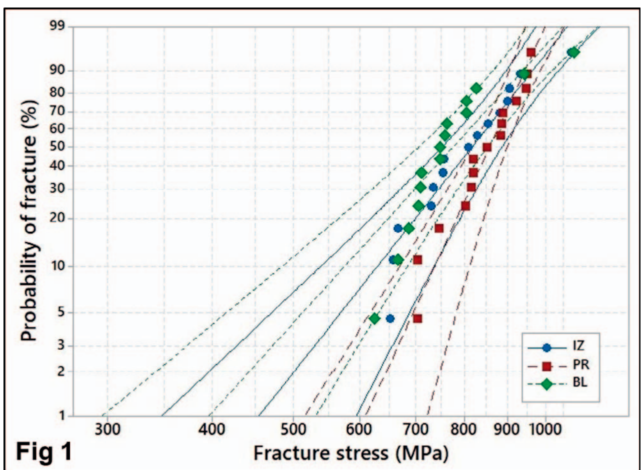
Different letters indicate statistically significant differences ($p<0.05$) among rows.

Table 3: *Total Transmittance Medians (T%) of Each Group*	
Groups	**T%**
PR	25.02 (21.10, 27.01) a
IZ	21.46 (18.94, 23.09) b
BL	23.72 (21.39, 25.54) a

Values in parentheses are the first quartile and the third quartile, respectively. Different letters indicate statistically significant differences among rows.

strength, Weibull modulus, and fractographic behavior of three different zirconia ceramics to better understand the behavior of monolithic materials. The null hypothesis was partially rejected because there were significant differences for some properties of the zirconia evaluated.

Several studies have examined the mean roughness (Ra) of dental restorative materials[22,36,43-47] due to its importance in early biofilm interlocking and further maturation process[43] as well as its crucial role in the resistance of dental ceramics,[44-46] usually with a significant, negative correlation with FS.[26,39,48] Moreover, roughness is directly associated with the translucency of the material[22,36,47] since smooth surfaces could contribute to better esthetic



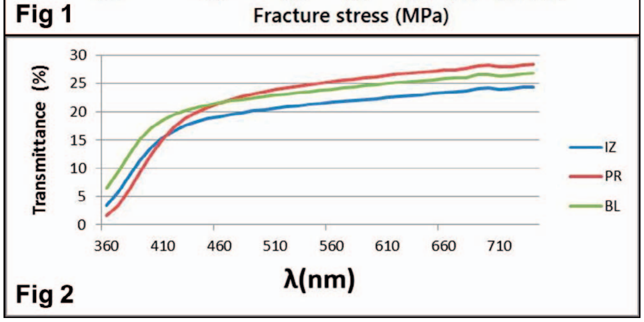


Figure 1. *Weibull plots of fracture data for PR, IZ, and BL groups.*
Figure 2. *Spectral transmittance of each experimental group.*

Fig 3

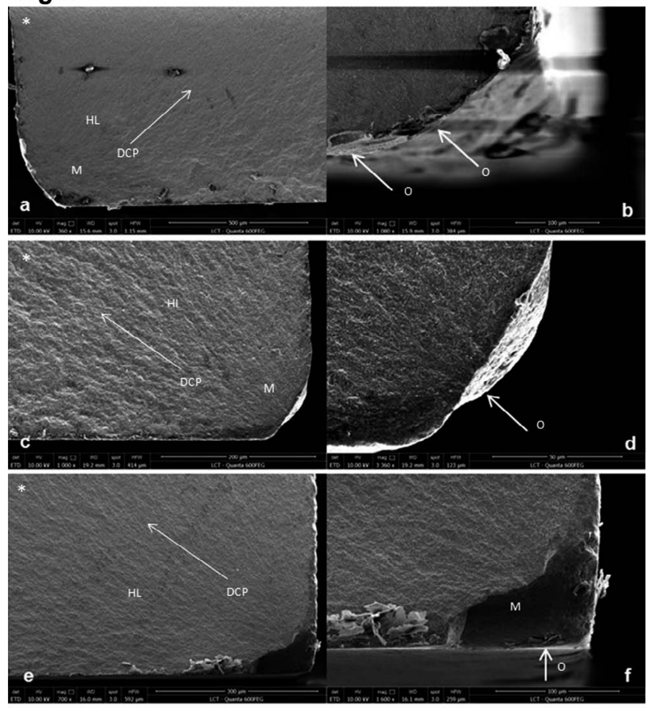


Fig 4

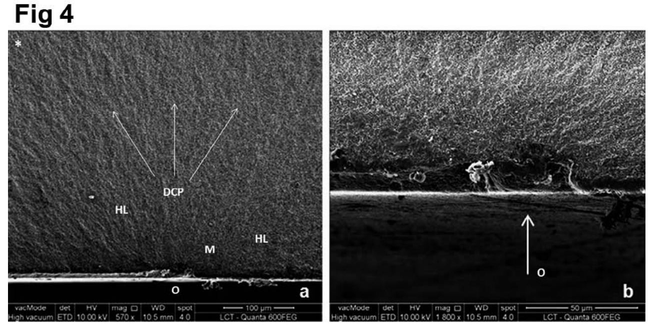


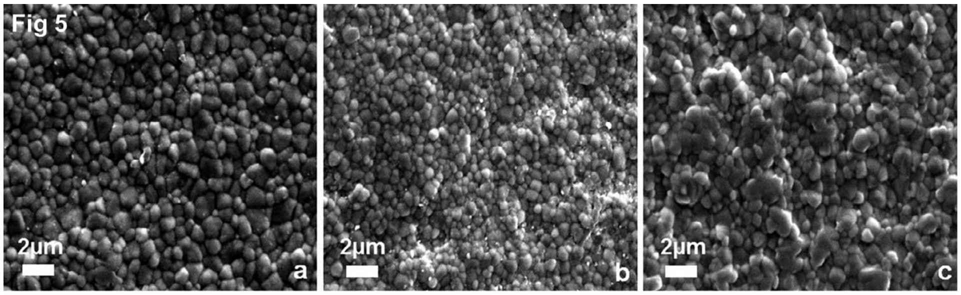
Figure 3. *Scanning electron micrographs: PR group (a and b), IZ group (c and d), and BL group (e and f), with different magnifications: (a) 360×, (b) 1080×, (c) 1000×, (d) 3360×, (e) 700×, and (f) 1600×. HL, hackle lines; DCP, direction of crack propagation; M, region of the mirror; O, origin of the fracture;*, top of the specimen.*
Figure 4. *Fractographic analysis of the BL group: (a) 570× and (b) 1800×. HL, hackle lines, DCP, direction of crack propagation; M, region of the mirror; O, origin of the fracture; *, top of the specimen.*

performance, promoting less additional loss of incident light.[47]

The Ra values obtained in this study are consistent with values obtained by other authors that ranged

Table 4: *Medians of Grain Sizes (μm)*	
Groups	**Grain Sizes**
PR	0.79 (0.63, 0.95) a
IZ	0.48 (0.39, 0.59) b
BL	0.71 (0.60, 0.90) a

Values in parentheses are the first quartile and the third quartile, respectively. Different letters indicate statistically significant differences (p<0.05) among rows.



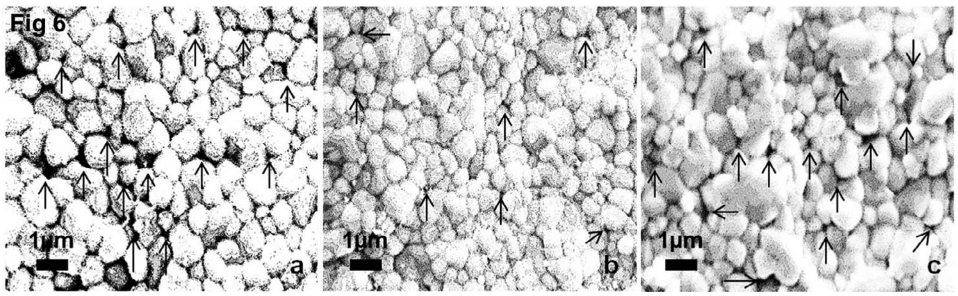


Figure 5. *Surface topography of the groups at 5000× magnification: (a) PR, (b) IZ, and (c) BL.*
Figure 6. *High brightness and contrast micrographs of the groups at 10,000× magnification: (a) PR, (b) IZ, and (c) BL. Arrows indicate voids.*

from 0.18 to 0.98 μm.[44,49-52] This wide range of values can be explained by variations in different surface treatments, polishing systems, zirconia grain sizes, pores, and flaw population.[44,51,53,54] After polishing and before sintering ($Ra_{presintered}$), the Ra was similar for all groups, indicating the standardization of polishing; however, after sintering ($Ra_{sintered}$), all groups had their roughness increased. A small increase in roughness can be expected due to surface modification, such as grain growth; however, for BL, this increase was higher, making it statistically different from the others. The Ra values obtained in this investigation can be considered clinically acceptable since only BL had Ra near 0.20 μm, a known threshold for plaque accumulation.[55] However, all groups showed Ra values above 0.5 μm, which is considered a Ra value detectable by the tongue.[56] The higher Ra measured for BL was explained by the SEM images (Figure 5c) since a more irregular surface and grains with asymmetrical size and form were observed for this material.

The VHN values found in this study for conventional and monolithic zirconia are consistent with values found in the literature, that is, around 1300 VHN,[35,57,58] with no statistical differences observed among the groups. The hardness of monolithic zirconia is important because the absence of veneering porcelain leaves the zirconia surface in direct contact with the antagonist tooth. A material with greater hardness may have greater mechanical strength, but it is difficult to determine whether this will result in higher wear rates for the antagonist tooth.[59] According to Goo and others,[60] the hardness of zirconia is twice that of dental porcelains. Due to this higher hardness, more enamel wear could be expected when monolithic zirconia is used. However, Stawarczyk and others[59] showed that the highest wear rate was observed for veneered and glazed zirconia as compared to polished monolithic zirconia. In the same study, the authors noted that monolithic zirconia resulted in higher rates of enamel cracks.

The means of FS obtained in the present study were either lower[24,61] or similar to those found in previous studies,[5,41] but these strength values are high enough to withstand the masticatory forces applied to three-unit fixed partial dentures.[28] Differences regarding FS obtained in different studies are usually related to the different methodological approaches and to the relationship between the strength of all-ceramic materials and the variation in the flaw population of different materials.[62]

The statistically similar FS results found for PR in comparison with the other two materials is not in agreement with the literature that showed lower values of FS for other monolithic zirconia.[63-65] In contrast, Flinn and others[61] used the Prettau zirconia and obtained higher values than those obtained in the present study of four-point FS test (1328±89.9 MPa). Munoz and others[66] using a biaxial flexure test obtained statistically similar values between Prettau zirconia and Ice Zircon.

Some authors associate the increase in grain size with the decrease in the FS of the material;[63-65] therefore, it was expected that PR would have similar FS in comparison to BL. However, the lack of statistical difference among the FS values of all material indicates that the increase in grain size did not affect the FS of the monolithic zirconia tested. The strength of zirconia specimens is associated with flaws such as porosity, agglomerates, inclusions, and large grains.[38] IZ showed less quantity of voids in the SEM (Figure 6); however, when these voids were statistically analyzed by the Weibull modulus, the materials had a statistically similar flaw population (Table 2). Therefore, the similarity among the FS values observed for these monolithic zirconias may be associated with other factors, such as amount of dopants, chemical composition, and crystalline structure. These variables need to be further investigated.

The Weibull modulus (m) of dental ceramics usually ranges from 5 to 15,[59,67] which is consistent with the present study. A higher m indicates a material that is more reliable under clinical conditions. This is because it has lower variation in flaw size in a certain volume of material, suggesting that the defects are uniform and evenly distributed throughout the entire volume.[30-32,68] Since the m values obtained in this investigation were similar for all materials, it is possible to infer that the flaw population was similar among the materials tested.[32,69]

These results could be corroborated by the fractographic analysis (Figures 3 and 4). Since advanced ceramics such as zirconia usually display linear stress-strain behavior, the lack of ductility, combined with the presence of flaws that have various sizes and orientations, leads to scatter in strength data.[38] Therefore, it is highly recommended that each failed test specimen be examined in order to identify the fracture origins.[38]

In the present study, the majority of the specimens showed surface flaws on the tensile side of the specimen, near the chamfer produced in the bend bar (Figure 3). BL also exhibited some fracture origins located at the center of the tensile surface (Figure 4). Fracture origins were identified by means of fractography principals proposed by Quinn[70] and by Scherrer and others.[40] Several fractographic features were identified, such as the compression curl, the hackle lines, the mirror, and then the origin. In Figures 3 and 4, it is possible to note hackle lines, the direction of crack propagation, the mirror, and the origin. Hackle lines are lines that clearly indicate the direction of crack propagation.[70] They commonly form when the crack moves rapidly.[40] The fracture mirror is a smoother region that surrounds the origin of the fracture.[70] In these specimens, the mirror is not so characteristic as described by Quinn[70] for glasses; in Figures 3 and 4, it can be subtly noted between the origin and hackle lines. All specimens had the compression curl on the top and the origin of the fracture on the bottom, indicating that the flexural test was carried out correctly and that the fractures were due to flexural force.

The PR and BL groups obtained higher transmittance compared to IZ. In the visible light region (wavelength from 360 to 740 nm), the median percentages of transmittance was 21.46% for IZ vs 25.02% for PR and 23.72% for BL. The higher transmittance of monolithic zirconia found in the present study is consistent with other studies that also showed higher translucency for these materials in comparison with traditional ones.[22,71] It is known that the translucency of dental ceramics is affected by various factors, such as grain size, pores, sintering temperature, and surface roughness.[22,36,71] Most studies attribute the increase in transmittance to grain sizes that are smaller than the wavelength of the incident light and therefore avoid the birefringence phenomenon, which is responsible for light scattering in Y-TZPs.[36,71-73] However, when the grains become larger than the wavelength of the incident light, light scattering becomes inversely proportional to grain size.[36,74] So there are two main methods to produce a more translucent zirconia, as one can either increase or decrease the grain size. Usually, when the choice is to decrease the grain size, the strength of the material is not affected. However, when the grain size is increased, transmittance increases, and strength decreases. In the present study, groups having higher transmittance were those having higher grain size, PR (0.79 μm) and BL (0.71 μm), vs IZ (0.48 μm). Apparently, for the manufacturers of these materials, the choice was to increase the grain size to increase the transmittance. In the present study, this increase in grain size did not decrease the FS.

In addition, Krell and others[47] showed that the grain boundary causes light scattering. In the present study, BL and PR presented larger grains and therefore fewer grain boundaries, thus decreasing light scattering and increasing the total transmittance. Harianawala and others[22] also attributed the difference in transmittance between conventional and monolithic zirconia to the smaller number of internal defects and pores in the latter, which also decreases light scattering.

The grain sizes measured for the IZ and PR groups are within the range reported in the literature.[71,75,76] However, the grain sizes obtained for the BL group were greater than the size proposed in the literature for conventional zirconia (0.5 μm). It is important to control the grain size for conventional zirconia because larger grains can facilitate the *t-m* transformation and result in degradation of the material over the long term.[52,64,75-77] However, for monolithic zirconia, materials with larger grains showed degradation similar to that of conventional zirconia.[78]

The present study has limitations, such as the fact that static loading for FS does not reproduce intraoral loading conditions[41] and does not take into account the effects of design, variation in thickness of the framework, the nature of human occlusion, and the loading environment. Additional studies, with specimen geometries used in clinical applications, should be pursued in the future.

CONCLUSIONS

Based on the results of the present study, it was concluded that all zirconias tested showed similar VHN and that the monolithic zirconia had similar roughness compared to the conventional zirconia (IZ group). In addition, the monolithic zirconia showed similar flexural strength and Weibull modulus compared to the others, even though its mean grain size was larger. The total transmittance of monolithic zirconia was higher than only one of the conventional zirconias tested (IZ group).

Acknowledgements

The authors wish to thank PhD student Erick de Lima from the Department of Biomaterials and Oral Biology, São Paulo Dental School, Universidade de São Paulo, for his contribution to this study. The group also wishes to thank the dental laboratory Frezzara, Araraquara, Brazil, for the contribution of the materials to the study.

Conflict of Interest

The authors of this article 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.

(Accepted 2 October 2017)

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