Original Article

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The relative translucency and fracture toughness of different zirconia ceramics

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Objective: To evaluate the relationship between relative translucency and fracture toughness obtained from the chevron notched beam (CNB) method of different zirconia ceramics.

Materials and Methods: Ten specimens of two commercial zirconia ceramics brands, Dentsply Sirona (Bensheim, Germany) and Zirkonzanth (GmbH, Italy) were cut into tiles to investigate their relative translucency: translucency parameters (TP) and contrast ratio (CR) using a spectrophotometer. Additionally, bar specimens were prepared to determine the fracture toughness using the CNB method and a four-point flexural test. Data were analyzed using two-way ANOVA, and Tukey's HSD test (=0.05).

Results: The results showed the CR value ranged between 0.69 to 0.74 with no significant differences among the groups except Prettau anterior (PRTA), which had significantly a lower CR value than other tested groups. For InCoris group, the TP was in the range of 12 to 13 with no significant differences between groups, whereas there was a significant difference between Prettau and PRTA. The PRTA has the highest translucency with the TP of 14.30±0.75 and the largest grain size, but the least fracture toughness.

Conclusion: More translucent zirconia had a lower fracture toughness compared with those of less translucent ones. However, zirconia ceramics, that were purported to be translucent by the manufacturer may not precisely be more translucent than the conventional zirconia core materials.

Keywords: chevron notched beam (CNB), contrast ratio, fracture toughness, translucency parameter, zirconia

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Introduction

The rapid increase in the demand of patients for aesthetics and the high noble metal cost has led to the establishment of metal-free dentistry. Although metal-ceramics restoration has favorable mechanical properties, concerning aesthetics, it cannot imitate natural tooth translucency due to the completely opaque substructure and the grayish background from metal underneath. [1, 2] Contrary to conventional metal-ceramic restoration, acceptable esthetic, excellent mechanical properties, and good biocompatibility have prompted the use of zirconia for fixed dental prostheses, especially in the posterior region and for long span fixed dental prostheses. [3]

The monolithic zirconia is gaining popularity among dentists for being a material of choice for posterior restorations to avoid undesired consequences such as the chipping of the veneering material, unnecessary tooth preparation and time consumption. [4] However, monolithic zirconia may not totally solve the problem for aesthetic concerned patients due to its opaque white color in nature. Therefore, the drive for more esthetic zirconia with improved optical properties was

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researched, therefore, the second and third generation of zirconia restorations were invented. [5]

An ideal dental ceramic material should exhibit outstanding characteristics such as excellent biocompatibility, translucency resembling natural tooth color, and high fracture resistance. [6] Among all restorative materials, zirconia exhibits the properties for ideal restorative materials mimicking that of a natural tooth. [7] Even though lithium disilicate ceramic materials are preferred over zirconia for anterior restorations, however, zirconia ceramics has greater fracture resistance than that of lithium disilicate glass-ceramics. [8] Fundamentally, zirconia is polymorphous, which consists of three crystalline lattices depending on the temperature condition, monoclinic (at room temperature), tetragonal (1170°C -2370°C), and cubic (2370°C) to the melting point of 2680°C.[9] So-called translucent monolithic zirconia shows lower contrast values and lower flexural strength than conventional zirconia. [9, 10]

Translucency is a vital parameter in matching the appearance of the natural tooth. The translucency of dental materials is commonly measured by using translucency parameter (TP), contrast ratio (CR), opalescence parameter (OP), relative translucency, and transmittance (T) among which TP and CR are the widely used as standard methods to determine mean translucency for all-ceramic materials. [11] TP is calculated by comparing the color difference of the specimen over black and white backgrounds, whereas CR is the reflectance ratio of a specimen over a black and white backgrounds, where CR is the reflectance ratio of a specimen over black and white background of known reflectance, and it is an estimate of opacity. [12]

Generally, the translucency of zirconia depends on multiple factors such as the amount and type of additives, sintering temperature, and the atmosphere during the sintering procedures, surface treatments and grain size, and the amount of light. [13,14] However, all-ceramic materials allow a certain amount of light to pass through, depending on the crystalline content, which results in a certain degree of translucency. [15] Additionally, the increased translucency of zirconia was obtained when there is a reduction in Al_2O_3 content, so as to improve light transmission. [16]

Fracture toughness (K_{IC}) or critical stress intensity is a material property to resist the crack propagation. It means the lower the fracture toughness of the material is, the more brittle the material acts. [17] For zirconia, stress-induced transformation toughening occurs when the zirconia particles are in the metastable tetragonal form and transform to the monoclinic phase to resist the stress from the volume increase due to the transformation. [18] High sintering temperature along with the long duration of dwelling time increased the grain size and the number of micropores, gradually resulting in a material with reduced mechanical properties, but greater translucency. [19]

Nowadays, there have been high demands for adequate translucency for better esthetics and the enough strength to resist high stress generated during mastication. This has driven to the introduction of numerous new zirconia ceramic materials. [20] The monolithic zirconia claims for greater translucency, however, the studies for the strength properties were limited to evaluate the translucency and mechanical properties of these recently introduced zirconia materials. Therefore, the purpose of this study was to evaluate and compare relative translucency, the fracture toughness of dental zirconia ceramics.

Materials and Methods

The material details used in current study are presented in Table 1.

Table 1 Zirconia materials used in the study

Material	Code	Manufacture	Batch No.	Composition
inCoris ZI	ZI	Sirona, Bensheim, Germany	2014353219	$ZrO_2+HfO_2+Y_2O_3 \geq 99.0$ %, $Y_2O_3 > 4.5 \leq 6.0$%, $HfO_2 \leq 5$%, $Al_2O_3 \leq 0.5$%, $Fe_2O_3 \leq 0.3$%
inCoris TZI	TZI	Sirona, Bensheim, Germany	2014252283	$ZrO_2+HfO_2+Y_2O_3 \geq 99.0$ %, $Y_2O_3 > 4.5 - \leq 6.0$% $Al_2O_3 \leq 0.5$%, $Fe_2O_3 \leq 0.3$%
inCoris TZI C	TZIC	Sirona, Bensheim, Germany	2014454044	$ZrO_2+HfO_2+Y_2O_3 \geq 99.0$ %, $Y_2O_3 > 5.6$%, $Al_2O_3 \leq 0.35$%, other oxides (except Er_2O_3)
Prettau®	PRT	Zirkonzahn, GmbH, Italy	ZB62960	ZrO_2, Y_2O_3 4% -6 % Al_2O_3 <1%, SiO_2 & Fe_2O_3, max 0.02%, Na_2O max 0.04%
Prettau® Anterior®	PRTA	Zirkonzahn, GmbH, Italy	ZB51761	$Y_2 0_3 < 12$%, $Al_2O_3 < 1$% SiO_2 & Fe_2O_3, max 0.02%, Na_2O max 0.04%

Sample preparation

I. Fracture toughness

All the specimens were prepared as bars from pre-sintered dental milling blanks by cutting with a low-speed diamond saw (Isomet®, BUEHLER®, Illinois, USA). The specimens were sintered according to the manufacturers' instructions. The final dimension of the specimens according to ISO 24370 [21] and ISO 6872 [22] was 3.0 x 4.0 x 45.0 ± 0.05 mm^3, where four-point bending strength was measured. A total of 50 specimens (n=10) were prepared for this study.

The chevron notch was subsequently prepared along with sample holder that was designed specifically for making the notch half angulation of 26^0. The revolution and cutting speeds were 5000 rpm and 4mm/min, respectively. An optical light microscope (Nikon MM-11C, Eclipse E400 POL Tokyo, Japan) was used to observe the chevron tip dimension that is a_0: 0.80 ± 0.08, avoiding overcuts and undercuts as shown in Figure 1 and Figure 2

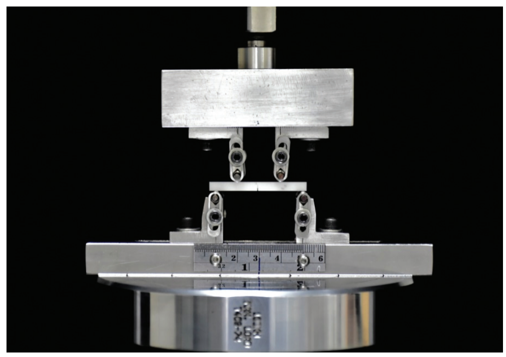

Figure 1 Load configuration under four-point bending test.

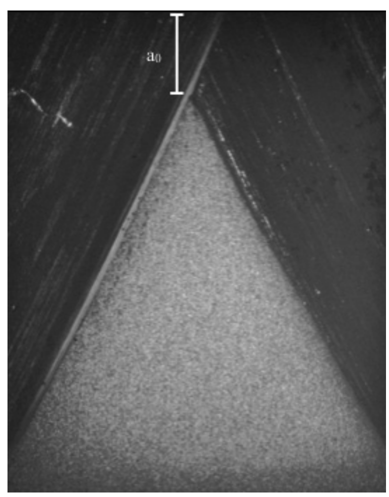

Figure 2 SEM micrographs showing the fracture surface of zirconia specimen with chevron notch after testing.

The prepared specimens were thoroughly cleaned with acetone in an ultrasonic bath (Bandelin ultrasonic) for 5 minutes and then air dried. The specimen measurement was ensured with a digital caliper (Digital micrometer, IP65, Mitutoyo MC, Tyoko) with an accuracy of ± 0.01mm. The sample was placed into the 4-point flexure test assembly (Instron 8872, Instron, MA, USA) as shown in Figure 3. The cut notched bar was loaded to failure at a crosshead speed of 0.05 mm/min to avoid unstable crack growth during loading.

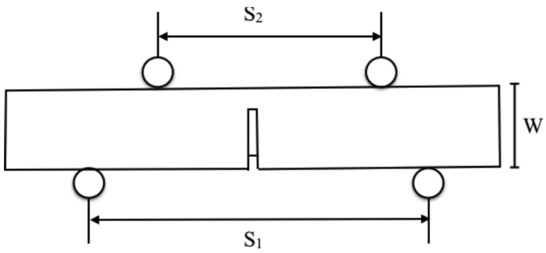

Figure 3 Test specimen for fracture toughness with Chevron Notch

where,

S_1 is the outer span length, in millimeter, S_2 is the inner span length, in millimeter, W is the test specimen width, in millimeter

The K_{IC} was calculated using the following equation according to ISO 24370.

$$K_{I,\,CNB} = \frac{F\,(S_O - S_I)}{BW^{3/2}} \times \frac{Y^*min}{\sqrt{1000}}$$

where,

$Y^*min\ (I_o/W,\ I_1/W) = 0.3874 - 3.0919\ (I_0/W) +4.2017\ (I_1/W) - 2.3127\ (I_1/W)^2 + 0.6379(I_1/W)^3$ $1.000 - 2.9686\ (I_0/W) +3.5056\ (I_0/W)^2 - 2.1374\ (I_0/W)^3 + 0.013\ (I_1/W)$

$K_{I,\,CNB}$ is the fracture toughness value in MPa $(m^{0.5})$ or $MN(m^{-3/2})$

F is the total load to failure, in Newton, S_1 is the outer span length, in millimeter, S_2 is the inner span length, in millimeter, B is the test specimen thickness, in millimeter, W is the test specimen width, in millimeter, Y*min is the stress intensity factor coefficient (dimensionless)

II. Translucency measurement

The specimens were prepared from pre-sintered dental milling blanks with a low-speed diamond saw (Isomet®, BUEHLER®, Illinois, USA) into final dimensions of 13 x 13 x 0.5 mm ± 0.05 mm after sintering, which was controlled during polishing using a digital caliper (Digital micrometer, IP65, Mitutoyo MC, Tyoko) with an accuracy of ± 0.01mm. A total of 50 (n=10) specimens were prepared.

After sectioning, the specimens were sintered according to the manufacturers' instructions using the corresponding furnaces. The specimens were cleaned ultrasonically in distilled water for 10 min and then air dried for 20 secs before testing. The spectrophotometer (UltraScan PRO, Hunterlab) was used to determine relative translucency. TP was determined by calculating the color difference between the same specimen against white (W) and black (B) background according to the equation below. L* refers to the brightness, a* refers to greenness, and b* refers to blueness. High TP value indicates high translucency of the material. [23]

$$TP = [(L_B - L_W)^2 + (a_B - a_W)^2 + (b_B - b_W)^2]^{½}$$

The contrast ratio (CR) was determined from the spectral reflectance of light, the specimen (Y) over a black (Y_b) and white (Y_w) background, using the equation, CR= Yb/ Yw

Less CR value indicates high translucency because CR is the direct measurement of opacity. Therefore, the value of totally transparent material is 0, while the value of completely opaque material is 1. [24]

III. Grain size analysis

For the measurement of the grain size, contrast ratio specimens were used. They were ultrasonically cleaned for 5 minutes in 80% ethanol and then air dried. The specimens were subsequently gold sputtered for 45 seconds. The surface topography was evaluated under a scanning electron microscope (JSM-6610LV, JOEL USA Co. Ltd, Massachusetts, USA) that was operated at

20 kV with the working distance of 12 mm. The grain size was measured using SEM micrographs using ImageJ software (Imagej.net) and linear intercept method.

Results

Statistical analysis was performed using a statistical software (IBM SPSS Version 22, IMB). The distribution of data was determined by the Kolmogorov-Smirnov and Shapiro Wilk tests. The descriptive statistics of all groups were computed. The data were analyzed using two-way ANOVA followed by Tukey's post-hoc test at 95% confident level. Measurement of correlation between TP and K_{IC} was determined using the Pearson correlation test.

The mean relative translucency values are summarized in Table 2. According to the TP and CR, Prettau® Anterior® (PRTA) was the most translucent material compared with that of the other zirconia materials in this study. No differences were found among the other groups except the TP of Prettau and inCoris TZIC. Based on the spearmen rank correlation test, a significant correlation between CR and TP was found (P<0.001)

The mean fracture toughness is summarized in Table 2. According to the data, there was no significant difference of the fracture toughness among the zirconia materials in this study except that the fracture toughness of the PRTA group was lower than that of the other groups.

The same superscript indicates no statistically significant differences (P< 0.05) among the tested materials. Bold font indicates the most translucent and least fracture resistance.

Figure 4 shows the grain size of the zirconia materials, which varied from 0.40 to 0.77 µm from which the PRTA had the largest grain size and TZIC had the smallest grain size. The SEM images of the specimens are shown in Figure 5.

Table 2 Translucency Parameter, contrast ratio and Fracture Toughness (Mean ± SD; n=10) of different zirconia ceramics.

Materials	Translucency parameter (TP)	Contrast ratio (CR)	Fracture toughness (Mpa m $^{0.5}$)
inCoris ZI	12.99 ± 0.48^{bc}	0.74 ± 0.03^{A}	$9.86 + 0.63^{c}$
inCoris TZI	13.42 ± 0.90^{bc}	0.72 ± 0.01^{A}	8.24 ± 0.59^{ab}
inCoris TZI C	13.36 ± 0.85^{b}	$0.73 + 0.01^{A}$	8.82 ± 0.97^{a}
Prettau®	12.14 ± 0.67^{c}	0.73 ± 0.08^{A}	7.60 ± 0.66^{b}
Prettau® Anterior®	14.30 ± 0.75^{a}	0.69 ± 0.01^{B}	2.39 ± 0.32^{d}

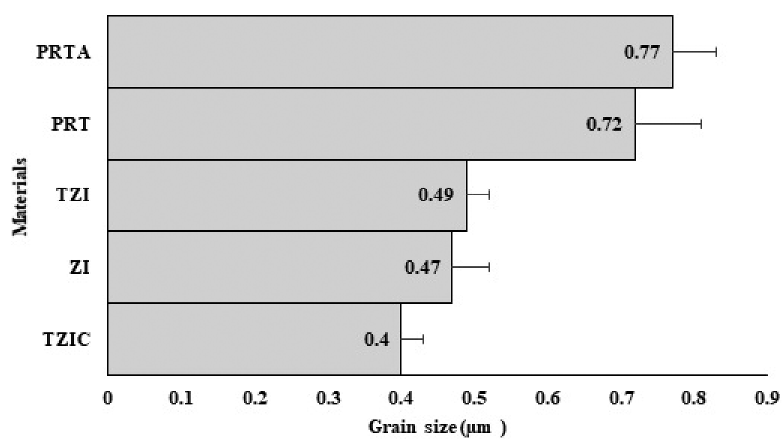

Figure 4 The grain size (µm) of five zirconia groups.

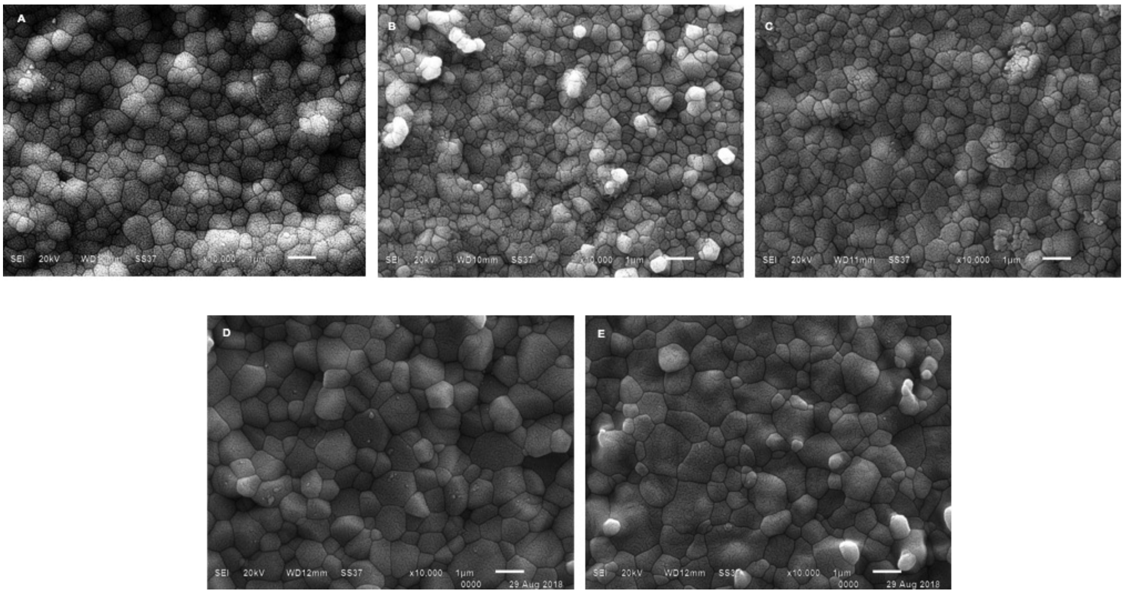

Figure 5 SEM images at magnification X 10,000 of all the tested zirconia materials.
A) inCoris ZI, B) inCoris TZI, C) inCoris TZIC, D) Prettau®, E) Prettau® Anterior®.

Discussion

This study aimed to determine the mechanical and optical properties of zirconia-based materials. The result obtained from this in vitro study clearly shows that the fracture toughness of more translucent zirconia was less than that of the more opaque zirconia. Additionally, this study evaluated the grain size of those zirconia to investigate the microstructure effect on optical and mechanical properties of zirconia materials.

The statistical analysis revealed an inverse relationship between TP and CR among the five different zirconia ceramics. The result was in agreement with another study. [25] The thickness of the material is considered as one of the factors affecting translucency. Generally, increased thickness resulted in greater opacity or higher CR values due to reduced light transmission. [26] In this study, all the specimens were fabricated at the thickness of 0.5 mm for consistent and valid measurement. Additionally, manufacturers and researchers clinically accept and recommend zirconia ceramics with a minimal thickness of 0.5 mm. [27]

Moreover, the various zirconia materials in the present study have a certain level of translucency. However, for the inCoris groups (Dentsply, Sirona), the grain size ranged between 0.4 to 0.5 μm, as shown in Figure 4 with no distinct difference to each other as the composition is substantially similar among the groups. A previous study clinically classified Sirona incoris ZI as “low translucency” and Sirona incoris TZI as “medium translucency.” [28] However, there was no significant difference of the translucency values among the InCoris materials in this study.

Several studies confirmed that the use of additives such as alumina dopant aids in enhancing the mechanical properties of zirconia, thus resulting in reduced material translucency. For this reason, veneering is utilized to alter the optical characteristics to a certain extent. [29]

Several authors identified multiple factors, which affect the aesthetic such as material thickness, grain boundary, the content of the materials, color of the luting cement, zirconia grain size and sintering conditions. [14, 30] The study by Bogna and colleagues used four monolithic zirconia materials and compared their grain sizes. The authors stated that the larger was the grain size, the more translucency of the material would become. [10] Therefore, a similar observation was found in the current study, and the SEM images as shown in Figure 5 reveals that PRTA had the largest grain size among all the other tested materials and was the most translucent zirconia ceramic material in this study. [31]

Among all the suggested methods, CNB is a standardized method to evaluate the fracture toughness of ceramic materials; it provides reproducible results with low data scatter, despite the sophisticated procedure during chevron notch preparation. [21, 32] The benefit of the CNB was that the fracture always initiating from the most critical crack that was the prepared notch. Prior investigations showed that CNB method produces a result with low standard error due to presence of pre-crack, which bends and stabilizes during first crack growth proceeding fractures. [33] In this method, the stress intensity factors (SIF) are known as a function of crack length and the real (natural) crack is measured. [34]

In this study, high fracture toughness value was observed for those materials with greater CR values, and past investigations also showed that a higher flexural strength of ceramic materials resulted in a higher contrast ratio. [15, 19] In this study, the measured fracture toughness ranged from 2.39 to 9.86 MPa $m^{0.5}$ and the minimal fracture toughness required for single unit anterior and posterior prosthesis is 2 MPa $m^{0.5}$ as suggested by ISO 6872. [21] A previous study reported that fracture toughness eventually declines as grain size increased. As a result of internal tensile stress, crack is formed due to increase in number of micropores, thus reducing the mechanical properties of the materials. [35, 36]

Based on the results of the current study, PRTA (Zirkonzahn) showed the largest grain size, and it had the lowest fracture toughness among the tested groups. Whereas Sirona incoris ZI was observed for high fracture toughness as it contains more than 0.5 % wt Al_2O_3 and considered as toughest material by the manufacturer as compared to others tested Sirona inCoris. [37, 38] Generally, conventional zirconia contains 0.5 to 1.0 % wt Al_2O_3 and 3-6% Y_2O_3, but in the present study, inCoris (Dentsply, Sirona) composes more than 0.5 % wt of Al_2O_3 and 4-6 % wt of Y_2O_3. Whereas, PRT and PRTA (Zirkonzahn), contains less than 1 % wt of Al_2O_3 and 4-12 % wt of Y_2O_3. [39] Therefore, it can be concluded that the composition of materials affect the overall characteristics of zirconia ceramics. The translucent zirconia with reduced Al_2O_3 content and increased Y_2O_3 content improved the optical properties whereas the increased Al_2O_3 content improved the mechanical properties. [37]

According to ISO 6872 standard, the minimum fracture toughness of 5 MPa $m^{0.5}$ is required for dental ceramics to fabricate prostheses involving four or more unit. Whereas in this current study, all the materials can be used to fabricate prostheses involving four or more unit except for PRTA of which the fracture toughness of 2.39 MPa $m^{0.5}$, recommended for anterior restorations according to ISO 6872. [22] The clinical indication for all-ceramic restorations indicates that all-ceramic systems can be used preferably on anterior teeth; only zirconia showed adequate mechanical resistance for both anterior and posterior restorations. [40]

Conclusion

This study showed that fracture resistance of experimental zirconia materials decreased as the relative translucency increased. All the materials used in this study had a certain degree of translucency. However, PRTA, generally recommended for anterior restorations, was the most translucent zirconia with the least fracture resistance. Therefore, it can be concluded that when aesthetic is concerned the fracture toughness is considerably diminished.

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