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Grain size, crystalline phase and fracture toughness of the monolithic zirconia

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PURPOSE. This study evaluated the relationship among translucency, crystalline phase, grain size, and fracture toughness of zirconia. **MATERIALS AND METHODS.** Four commercial zirconia - Prettau®Anterior® (PA), Prettau® (P), InCorisZI (ZI), and InCorisTZI (TZI)- were selected for this study. The bar specimens were prepared to determine fracture toughness by using chevron notched beam method with four-point bending test. The grain size was evaluated by a mean linear intercept method using a scanning electron microscope. X-ray diffraction and Rietveld refinement were performed to evaluate the amount of tetragonal and cubic phases of zirconia. Contrast ratio (CR) was measured to investigate the level of translucency. **RESULTS.** PA had the lowest fracture toughness among other groups ($P < .05$). In addition, the mean fracture toughness of P was significantly less than that of ZI, but there was no difference compared with TZI. Regarding grain size measurement, PA had the largest average grain size among the groups. P obtained larger grain size than ZI and TZI ($P < .05$). However, there was no significant difference between ZI and TZI. Moreover, PA had the lowest CR value compared with the other groups ($P < .05$). This means PA was the most translucent material in this study. Rietveld refinement found that PA presented the greatest percentage of cubic phase, followed by TZI, ZI, and P, respectively. **CONCLUSION.** The different approaches are used by manufacturers to fabricate various types of translucent zirconia with different levels of translucency and mechanical properties, which should be concerned for material selection for successful clinical outcome. [J Adv Prosthodont 2022;14:285-93]

KEYWORDS
Ceramics; Zirconium oxide; X-Ray Diffraction; Fracture; Electron microscopy

INTRODUCTION

The natural appearance of dental restoration requires appropriate material selection, form, surface texture, translucency, and color.[1] Porcelain fused to metal (PFM) restorations have been reliable restorations for fixed dental pros-

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theses for a long time due to the strength from metal framework and esthetics from veneering porcelain. However, there are some drawbacks such as noticeable metallic color, especially in patients with thin gingiva or gingival recession and high lip line. Moreover, some metals used for framework material, especially base metal alloys, can cause an allergic reaction and corrosion more frequently than noble metal alloys or ceramics.[2] Additionally, one of the crucial problems is poor esthetics due to their opacity.

Esthetic demand for dental restoration has been increasing, both in anterior and posterior teeth. The esthetics can be achieved by having natural appearance that blend harmoniously with the rest of the oral and facial structures in the overall impression. Although well-designed PFM restorations can sometimes meet all the requirements, they still have limited clinical application due to their metal opacity and glass veneer chipping or fracture.[3,4]

Translucency is a property of materials that allows some light to transmit through while diffuse and/or reflect others. PFM restoration has a completely opaque metal framework; therefore, light cannot pass through it. This affects its natural appearance and esthetics of the whole restoration when the thickness of the glass veneering is insufficient to obscure the opaque porcelain. Novel ceramics, such as alumina, glass-infiltrated ceramic, and disilicate glass-ceramics, have been developed, and these materials have gained popularity because of their superior strength, esthetics, chemical stability, and biocompatibility compared with conventional PFM.[5] They have been used for crowns and three-unit fixed dental prostheses (FDPs) in the premolar region.[6-8] However, some of them have insufficient strength to withstand the occlusal load in the posterior region, and the veneering process is sometimes still required; therefore, the risk of veneer fracture may compromise the longevity of the final restoration.[7,8]

Zirconia-based ceramics have been introduced to dentistry, and they became popular alternatives to alumina as high strength biomaterials due to their high fracture toughness compared with other ceramic systems.[9] In dental applications, zirconia has been used to fabricate prefabricated posts, fixed dental prostheses, and dental implants. There are three zirconia-based ceramics widely used: yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP), magnesium cation-doped partially stabilized zirconia (Mg-PSZ) and zirconia toughened alumina (ZTA).[8] Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) has been proven to have high fracture toughness. However, Y-TZP generally has high opacity, and therefore, they still cause some esthetic problems that affect natural appearance of dental restorations. Recently, so-called translucent zirconia was developed as a monolithic material to replace bilayered zirconia, avoiding the incidence of veneer chipping, fracturing or delamination. Its greater translucency could reduce the opacity and the white color of the original zirconia.[10-12]

The optical properties of ceramic are mainly regulated by atomic structures in polycrystalline bodies.[12,13] Loss of optical translucency of polycrystalline material consisting of optically transparent grains, such as zirconia, is mainly due to light scattering.[12] Scattering is a physical process occurred when some forms of radiation (light or sound) or moving particles pass through non-uniformities in a medium and are forced to deviate from its straight path to one or more paths, leading to interior reflection and refraction. When light transmits into the polycrystalline bodies, the scattering may result from many factors, such as pores, impurities, defects, and grain boundaries.[12,14,15] Therefore, to improve the optical translucency, these factors must be considered and manipulated.

Light scattering at the zirconia grain boundaries can be generally reduced by increasing the grain size. When light passes through the polycrystalline bodies, there are fewer interactions between light and the grain boundaries. On the other hand, reducing the grain size is another effective method to improve translucency due to high in-line transmission.[15] The porosity and microstructure of material can be controlled by sintering parameters. Increasing sintering temperature and sintering time can enlarge the zirconia grain size, leading the zirconia to become more translucent.[16-18] Moreover, the amount of yttria content also affects the translucency. 3 mol% Y_2O_3 can stabilize the tetragonal phase of zirconia, and at the higher concentration, it can also stabilize cubic phase upon cooling to room temperature, inhibiting tetragonal (t) to monoclinic (m) phase transformations.[19] Thus, more yttria concentration resulted in higher cubic phase in zirconia, leading to more translucency.[15] Adding up the yttria content to 4% and 5% mol results in higher content of stable cubic phase. So-called high-translucent or ultra-translucent zirconia belongs to this recent or 3rd generation of zirconia (ref-wear behavior of different generation of zirconia).[20]

However, it should be noted that the microstructure of material affects their mechanical properties that influences the clinical success of dental restorations.[21] Increasing the grain size decreases the flexural strength of the conventional zirconia.[18] Also, it is well known that the transformation toughening effect in 3Y-TZP ceramics is grain size-dependent.[22] If the average grain size of the tetragonal grain is larger than 1 µm, the spontaneous tetragonal to monoclinic phase transformation is likely to occur. Besides, tetragonal-cubic hybrid zirconia from higher yttria content has inferior mechanical properties compared with tetragonal one.[15] Fracture toughness is an intrinsic mechanical property that indicates the material's resistance to the fracture under applied stress. It can be used to evaluate and predict the clinical performance, success rate, and longevity of the biomaterials. The material with high fracture toughness has enhanced clinical performance and reliability.[23]

While the optical properties of ceramic are regulated by microstructure such as grain size and grain boundaries, methods to improve the optical translucency are usually to alter the microstructure of the materials, which may compromise their mechanical properties.

Accordingly, this study aimed to evaluate the translucency, microstructure, and mechanical properties of the commercial monolithic zirconia. The null hypothesis was that they all present comparable properties.

MATERIALS AND METHODS

Four commercial zirconia used in this study were InCoris ZI (ZI), InCoris TZI (TZI), Prettau® (P), and Prettau® Anterior® (PA). Their components provided by the manufacturers are shown in Table 1. All of the specimens were prepared in pre-sintered states according to manufacturers' recommendation.

According to phase analysis, three representative bar specimens from each group were randomly selected and were placed in a diffractometer holder. The X-ray diffraction (XRD) patterns were collected by X-ray (CuK) diffractometer (XRD) (Model D5000; Simens, Munich, Germany) at room temperature. The X-ray diffractometer was operated at 40 kV and 35 mA, with a step of 0.04° every 2 sec in the 2θ range between 20 - 80°.[24] Rietveld Refinement was used to perform quantitative analysis of the crystalline phase of the zirconia specimen using the FullProf (FullProf Suite, Gif sur Yvette, France). The standard crystallographic information file (CIF) was derived from the crystallography open database (COD).

Grain size determination was also examined. The specimens (n = 5) of each group were wet-polished with abrasive papers up to 2000 grits and subsequently sintered as recommended by the manufacturer. Subsequently, they were ultrasonically cleaned for 10 min in acetone and dried at room temperature for 24

Table 1. Materials used in this study

Brand	Lot Number	Manufacturer	Component
InCoris ZI (ZI)	LOT 2014353219	Sirona, Bensheim, Germany	Y_2O_3 > 4.5 - ≤6.0% Al_2O_3 ≤ 0.5%, Fe_2O_3 ≤ 0.3%
InCoris TZI (TZI)	LOT 2014252283	Sirona, Bensheim, Germany	Y_2O_3 > 4.5 - ≤ 6.0% Al_2O_3 ≤ 0.5%, Fe_2O_3 ≤ 0.3%
Prettau® (P)	LOT ZB62960	Zirkonzahn GmbH, Ahrntal, Italy	Y_2O_3 4% -6% Al_2O_3 < 1%, SiO_2 & Fe_2O_3, max 0.02%
Prettau® Anterior® (PA)	LOT ZB51761	Zirkonzahn GmbH, Ahrntal, Italy	$Y_2 0_3$ < 12% Al_2O_3 < 1% SiO_2 & Fe_2O_3, max 0.02%

hr. After that, the specimens were thermally etched at 1200°C for 20 mins and sputter-coated with gold for 45 sec. The coated specimens were observed under the scanning electron microscope (SEM) operating at 20kV. At least five different areas of the test specimen were evaluated to determine the average grain size of each group at the magnification of ×20000 with ImageJ™ image analysis software (National Institutes of Health (NIH), Bethesda, MD, USA).[25]

The average grain size of each group was calculated using the linear intercept method according to the standard (EN 623-3 Advanced technical ceramics - Monolithic ceramics - General and textural properties).[26]

The mean intercept distance (g_{mli}) was calculated using the equation:

$$g_{mli} = \frac{[L(t) - L(p)] \times 10}{N(i) \times m}$$

Where: L(t) is the total line length in mm.
L(p) is the total line length that crosses large pore in mm.
N(i) is the counted number of intersections on each micrograph
m is the calibrated magnification of the micrograph.

Regarding contrast ratio investigation, four groups of pre-sintered state zirconia were cut using a low-speed diamond saw (Isomet wafering blades; Buehler, Lake Bluff, IL, USA) into plate shape and wet-polished with abrasive papers up to 2000 grits. These specimens (n = 5) of each group were subsequently sintered as recommended by the manufacturer. The final specimen dimension is 10 × 10 × 0.5 mm. Then the specimens were ultrasonically cleaned in acetone for 10 min and air-dried. The quantitative measurement of translucency was made by using the contrast ratio. The luminous reflectance (Y) of specimens over black (YB) and white (YW) background were measured with a spectrophotometer (UltraScan PRO; Hunterlab, Reston, VA, USA) (Fig. 1). Each specimen was measured three times, and the mean value was used to calculate the contrast ratio using the equation

$$CR = Y_B / Y_W$$

Where: Y_B is the luminous reflectance of specimen with black background.
Y_W is the luminous reflectance of specimen with white background.

The value of 0 represents a totally transparent material, and the value of 1 represents a completely opaque material.

All blocks of yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) samples (n = 10) were prepared for fracture toughness test. All samples were cut from their pre-sintered state using a low-speed diamond saw into rectangular bars with a dimension of 4 × 5 × 46 mm before sintering for fracture toughness test. These specimens were ground with abrasive papers up to 1200 grits, and they were subsequently sintered as recommended by the manufacturer. The final specimen dimension was measured using a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Cali-

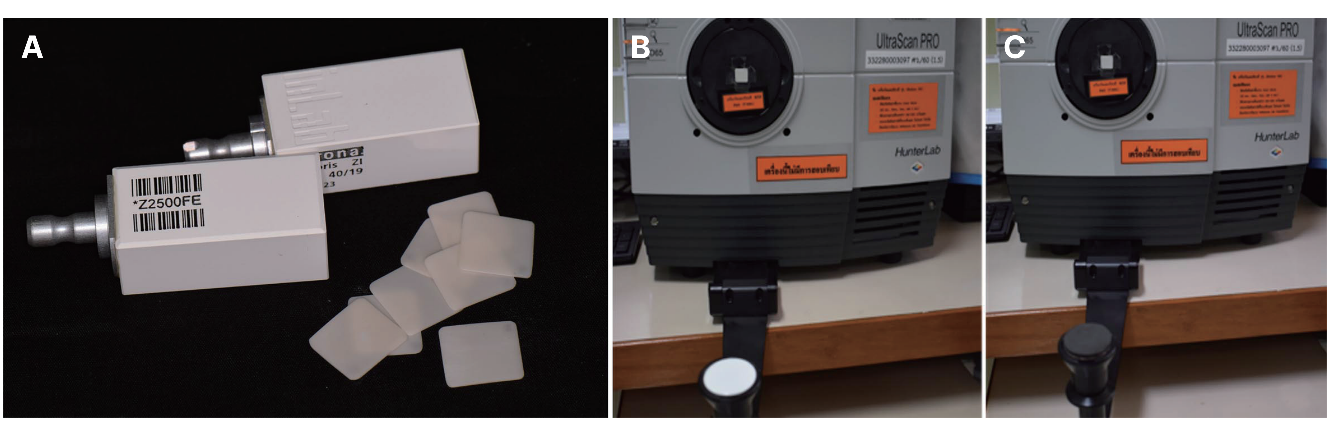

Fig. 1. (A) The examples of zirconia specimens used for contrast ratio measurement, (B) Spectrophotometer with a sample holder with white background and (C) with black background.

per; Mitutoyo Corporation, Kawasaki-shi, Kanagawa, Japan) to ensure that they were within the dimension recommended by ISO 24370[27] (3 ± 0.20 × 4 ± 0.20 × 45 mm in W × H × L) (Fig. 2A).

The triangular notch was created using a low-speed diamond saw at the center of each specimen with notch tip dimension of 0.8 ± 0.08 mm and the notch thickness of less than 0.3 mm (Fig. 2B). Notch tip dimension and notch thickness were observed using a measuring microscope at the magnification of 30 × (Nikon MM-11C; Hollywood traditional group, Tokyo, Japan). Finally, four groups of specimens (n = 10) were defined as ZI, TZI, P, and PA.

The specimens were tested by a 4-point flexure test according to ISO 24370 using a universal testing machine (Denison-Mayes, Leeds, UK) at a displacement rate of 0.05 mm/min until fracture. The minimum stress intensity factor coefficient (Y*min) and the fracture toughness ($K_{I,\,CNB}$) was calculated according to the following equation.

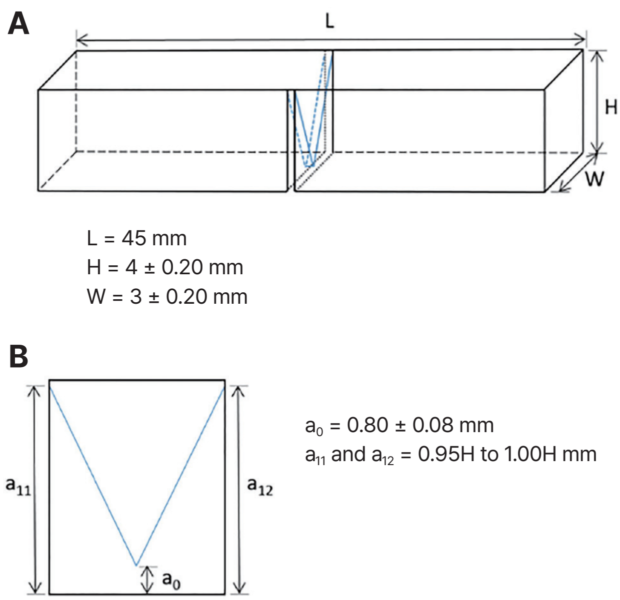

Fig. 2. (A) Dimension of a chevron-notched specimen and (B) the dimension of V-notch.

$$Y^{*}min(l0/W,l1/W) = \frac{0.3874 - 3.0919\left(\frac{l0}{W}\right) + 4.2017\left(\frac{l1}{W}\right) - 2.3127\left(\frac{l1}{W}\right)^2 + 0.6379\left(\frac{l1}{W}\right)^3}{1.000 - 2.9686\left(\frac{l0}{W}\right) + 3.5.56\left(\frac{l0}{W}\right)^2 + 0.6379\left(\frac{l0}{W}\right)^3}$$

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

where

$K_{I,\,CNB}$ is the fracture toughness value, in MPa·$m^{1/2}$ or MN·$m^{-3/2}$

F is the total force, in newton (Maximum force, F_{max}, Plus tare force, F_{Tare})

So is the outer span length in millimeter

Si is the inner span length in millimeter

B is the specimen thickness in millimeter

W is the specimen thickness in millimeter

Y^{*}_{min} is the stress intensity factor coefficient (dimensionless)

All data were analyzed using SPSS statistic software (IBM SPSS Statistic for windows, Version 28.0. Armonk; NY: IBM Corp, Armonk, NY, USA). The samples were calculated using G*power program (V. 3.1.9.2; Universital Kiel, Kiel, Germany). Shapiro-Wilk test was performed to validate the normality of the data and Levene's test was performed for equality of variances among group data. One-way ANOVA followed by Games-Howell and Tukey's post-hoc test was applied to analyze the data from grain size and fracture toughness. The data from the contrast ratio examination were analyzed using Kruskal-Wallis followed by pairwise comparison. The significance level was set at 0.05.

RESULTS

According to phase analysis, the illustration represents the x-ray diffraction pattern of all zirconia samples (Fig. 3). All the specimens were observed in the 2θ range from 20 - 80°. The main peak of all specimens' patterns was detected at about 30°, indicating tetragonal and cubic phases. PA showed the highest relative peak intensity. In P, TZI and ZI groups, the tetragonal doublets could be observed in the 2θ re-

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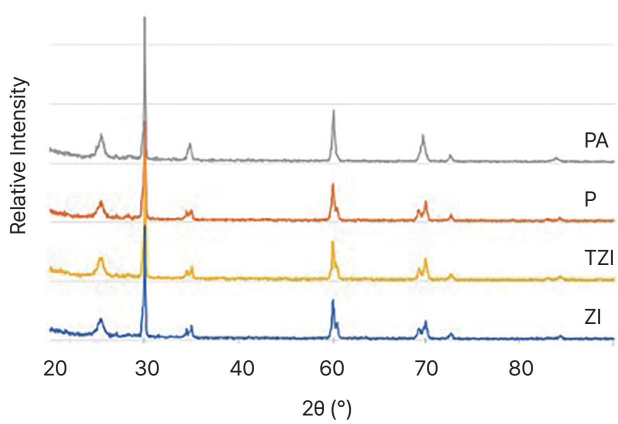

Fig. 3. X-ray diffraction patterns of Prettau® (P), Prettau® Anterior® (PA), InCoris ZI (ZI), and InCoris TZI (TZI). The peak detected in 2θ about 30° indicated tetragonal and cubic phase.

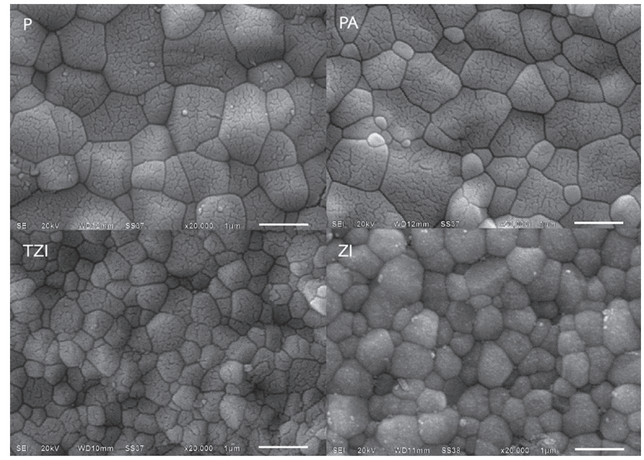

Fig. 4. SEM micrographs of the zirconia specimens (X 20000) Prettau® (P), Prettau® Anterior® (PA), Incoris TZI (TZI) and Incoris ZI (ZI) Zirconia. The difference in grain size can be observed. Bar indicates 1 μm.

Table 2. Percentages of tetragonal and cubic phases and means ± SD of average grain size, contrast ratio (CR), and fracture toughness of each zirconia group are shown

Materials	Phase		Grain Size (μm)	CR	Fracture toughness (Mpa·m$^{1/2}$)
	Tetragonal Phase (%)	Cubic Phase (%)			
InCoris ZI (ZI)	86.1	13.9	0.39 ± 0.01[a]	0.76 ± 0.05[a]	8.82 ± 0.98[a]
InCoris TZI (TZI)	84.6	15.4	0.40 ± 0.01[a]	0.72 ± 0.02[a]	8.29 ± 0.55[ab]
Prettau® (P)	91.8	8.2	0.58 ± 0.02[b]	0.72 ± 0.01[a]	7.85 ± 0.84[b]
Prettau® Anterior® (PA)	69.4	30.6	0.75 ± 0.03[c]	0.69 ± 0.01[b]	2.30 ± 0.33[c]

The same superscript in the column indicates no significant difference at 95% confidence level.

gion of 35° and 70°, while PA displayed unique cubic singlet at these areas. After Rietveld refinement, the percentage of tetragonal and cubic phases of all zirconia specimens was presented in Table 2.

The average grain size of each zirconia group was calculated by the linear intercept method and listed in Table 2. Grain size data showed no violation of the assumption of normality. One-way ANOVA followed by Games-Howell post hoc test found that PA has the largest average grain size among the studied groups. P group obtained larger grain size than ZI and TZI ($P < .05$). There was no significant difference between ZI and TZI ($P > .05$). The represent SEM images of each zirconia group at ×20000 magnification are shown in Fig. 4.

Kruskal-Wallis followed by pairwise comparison was performed to analyze the contrast ratio data. The results in Table 2 showed that PA group had the lowest CR value compared with the other groups ($P < .05$). This means PA is the most translucent material in this study. However, there was no significant difference among other groups.

The mean and standard deviation of the fracture toughness for each group are presented in Table 2. According to Shapiro-Wilk test, the data of all specimens were normally distributed. One-Way ANOVA revealed a significant difference among groups ($P < .05$). Tukey's post hoc test showed that PA group had the lowest fracture toughness among the tested groups ($P < .05$). In addition, the mean fracture toughness of P was also significantly less than that of ZI, but there was no difference when compared with TZI.

DISCUSSION

Understanding the microstructure of the material is crucial because the microstructure usually affects other properties of the material,[4,28] and it can be used to evaluate and predict the clinical performance, success rate, and longevity of the restorations. As mentioned previously, different microstructures in zirconia material result in different properties. This present study investigated the effect of crystal phase and grain size on translucent and mechanical properties.

In dentistry, zirconia with translucency has been developed due to increasing esthetic demand. One method is to stabilize zirconia at the cubic phase.[29] In this study, four commercial zirconia have been investigated. TZI, P and PA are designed by manufacturers to be translucent zirconia, and have been used as monolithic restorations, and ZI is recommended by the manufacturer as a framework material. X-ray diffraction and Rietveld Refinement are used to identify the cubic and tetragonal phases presenting in each zirconia specimen. Translucent zirconia, especially PA, showed the highest relative peak intensity. Adding 3 mol% yttria stabilizes zirconia mostly at the tetragonal phase while more yttria decreases the tetragonal phase, and addition up to 8 mol% or more of yttria will stabilize zirconia at cubic phase.[6,15] Among the translucent groups in this study, PA, claimed by the manufacturer to be highly translucent, has the most yttria content (Table 1). As a result, it had the highest percentage of the cubic phase compared with the other groups. Typically, cubic and tetragonal phases of zirconia have very similar structure due to their similar lattice parameters. To distinguish them, Srivastava *et al*.[30] reported that tetragonal structure exhibits characteristic splitting at some position, such as (002) (200), (004) (400), etc., while a single peak is presented at all of these positions for cubic phase. According to the study of Srinivasan *et al*.,[31] the 2θ region containing (002) (200) tetragonal doublets is presented about 34° - 36° and the 2θ region covering the (004) and (400) tetragonal doublets is presented about 73° - 75°. The specimens that exhibited the peak splitting at these areas in P, TZI, and ZI groups indicated that these zirconia groups consist of high tetragonal phase.

Contrast ratio is a direct measurement of opacity calculated by comparing the visible light intensity of specimen on white and black backgrounds. The value of 0 represents a totally transparent material, and the value of 1 represents a completely opaque material. Thus, less CR value indicated high translucency. The result showed a small difference among the tested groups. However, PA is the most translucent material in this study. The cubic crystalline phase is isotropic that means its properties are the same in every crystallographic direction. These characteristics decrease light scattering that occurs at grain boundaries, resulting in higher translucent zirconia from the cubic phase.[15,32] PA, having the highest percentage of cubic phase, presented the lowest contrast ratio value, resulting in the most translucent among all tested zirconia groups. According to the results, P has lower percentage of cubic phase than ZI, but it was more translucent. This might be because P has larger grain size. However, other factors might influence translucency such as differences in the fabrication process and the difference in composition, which is not clearly shown in the information from the manufacturer. Therefore, further studies on the other factors that affect the translucency and the mechanical properties of zirconia should be investigated.

The result shows that the fracture toughness of the translucent zirconia group (TZI, P and PA) was lower than that of the conventional zirconia (ZI). This may be because zirconia that is stabilized at the cubic phase does not exhibit transformation toughening mechanism; therefore, the benefit of stress-induced transformation toughening from tetragonal to monoclinic phase disappears. Consequently, it was reported that the zirconia with more cubic phase content had less strength and fracture toughness.[33] The mean fracture toughness of the zirconia of InCoris group (ZI and TZI) was higher than that of the Zirkonzahn group (P and PA). This may result from their smaller grain size. Corresponding to the previous studies,[15,34,35] smaller grain size of zirconia enhances the mechanical properties of the material. When the grain size increases especially beyond the critical grain size, zirconia becomes less stable and more susceptible to spontaneous tetragonal to monoclinic phase transformation, resulting in the decrease of the strength

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of zirconia.[36] The critical grain size of zirconia is approximately 1 µm[15,37,38] while Trunec reported that the critical grain size was between 1.8 - 2.15 µm.[39] On the other hand, when the grain size is below 0.2 µm, transformation toughening doesn't occur, which leads to the decreasing of fracture toughness of zirconia.[37,40] According to ISO 6872:2015, it is recommended that cubic containing zirconia requires a fracture toughness of more than 3.5 $MPa·m^{1/2}$. In this study, all of the zirconia groups except PA meet the ISO standard. Improving the translucency of zirconia can be done by reducing light scattering, which usually occurs at the grain boundary. The methods are to increase the grain size for minimizing the grain boundaries and to increase the amount of cubic phase by adding more yttria content.

CONCLUSION

According to the results of this study, when the translucency of the zirconia was highly improved, the fracture toughness was significantly compromised. The microstructure, such as grain size, is a crucial influence in increasing the translucency of zirconia, with the tendency showing that the larger the grain size, the higher the translucency, but the fracture toughness is negatively compensated. Furthermore, the high content of cubic phase may also affect the translucency and the mechanical properties. Therefore, clinicians must consider how to select the zirconia restorations for optimal clinical performance to achieve patient satisfaction and longevity of the zirconia restoration.

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