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Original Article

Comparison of optical and crystal properties of three translucent yttria-stabilized tetragonal zirconia polycrystals with those of lithium disilicate glass-ceramic material

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KEYWORDS

Lithium disilicate; Spectrophotometer; Translucency; X-ray diffraction; Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) **Abstract** *Background/purpose*: Among the ceramic materials used for all-ceramic crowns, zirconia has high biocompatibility and favorable mechanical properties, but its main drawbacks include low translucency and stress-induced phase transformation. To stabilize high-strength tetragonal zirconia polycrystal (TZP), 3–5 mol% yttria is usually added to prepare yttria-stabilized TZP (Y-TZP). In this study, the optical properties of three commercial Y-TZP ceramics were compared with those of the clinically available glass-ceramic material of lithium disilicate, and the relationship between translucency and crystal properties was analyzed in vitro. *Materials and methods*: Twelve 5-mm-thick standardized disks were prepared from three Y-TZP ceramics and one lithium disilicate block. Absolute translucency was measured using a spectrophotometer with an integrating sphere. X-ray diffraction was used to quantify the main structural parameters (i.e., preferred plane, quantitative phase, and grain size) of Y-TZP crystals.

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Results: The product-dominated phase of Y-TZP exhibited a tetragonal lattice pattern, and the preferred planes had minor variations. The diffraction patterns of the three Y-TZP ceramics demonstrated minor effects on translucency, without significant differences (p > 0.05). The grain size of 54–70 nm was negatively related to translucency in Y-TZP. Lithium disilicate specimens had significantly higher translucency than the three Y-TZP specimens (p < 0.001).

Conclusion: Grain size reduction played an essential role in developing highly translucent Y-TZP ceramics. The three Y-TZP ceramics were essentially opaque but exhibited poorer translucency than lithium disilicate in terms of esthetics.

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Introduction

All-ceramic crown treatment has become popular in fixed prosthodontics.¹ Among the ceramic materials used for this treatment, zirconia has the best mechanical strength and biocompatibility and has been widely employed in clinical dentistry.^{2,3} The value of prostheses greatly influences aesthetics. Human sensitivity to lightness or darkness produces a shade value that is higher than chroma and hue because the number of rod cells in the human eye is greater than that of cone cells.⁴ Mimicking the shade-matching performance of natural teeth that appear translucent is essential in esthetic treatments. The most important factor that affects shade value is material translucency,^{5,6} which is typically compared with that of an enamel-the outer layer structure of teeth.⁷ Belser⁸ proposed that material translucency should exhibit an optical phenomenon similar to natural teeth, such as opalescence and halo effects, to achieve esthetic restoration.

Zirconia has lower translucency compared with other ceramic materials, such as feldspathic porcelain, alumina, and leucite-based ceramics.^{6,9} Lithium disilicate ceramics have been widely used as aesthetic dental materials due to their good and adjustable translucency.⁸ Highly translucent commercial zirconia have been gradually developed and used clinically to address its limited translucency.¹⁰

According to standardized testing definitions^{11,12} and principles of optical physics experiments,¹³ a standardized light source, strict environmental conditions, integrating sphere, and a spectrometer with automatic calculation and subtraction are required in measuring absolute translucency. The spectrometer must accurately record light transmittance under different wavelength conditions, and the result is expressed as a percentage (%). The formula is as follows:

$$T_{\lambda} = \frac{\mathsf{S}_2 - \mathsf{D}_{\lambda}}{\mathsf{R}_2 - \mathsf{D}_{\lambda}} \times 100\%$$

where S_{λ} is the sample intensity, D_{λ} indicates dark intensity, and R_{λ} is the reference intensity at wavelength λ . The wavelength range in this experiment is the visible light wavelength of 380–780 nm because the energy of electromagnetic waves is similar to the energy jump of zirconia crystal molecules at short wavelengths (<380 nm). Scattering occurs when zirconia crystals are passed through short wavelengths, therefore, the measured data will show excessive fluctuations and are difficult to analyze.¹⁴

X-ray diffraction (XRD) method is generally used to analyze the crystal information of Y-TZP. The external standard method involves the use of diffraction peak intensities as preferential orientations of a phase to compare the different commercialized brands of zirconia.^{15,16} For grain size analysis, grain size is calculated via Scherrer's method¹⁷ using the following equation:

$$\tau = \frac{K\lambda}{\beta\cos\theta}$$

where τ is the grain width, *K* (approximately .9) is the shape factor constant, λ is the X-ray wavelength, β is the full width at half maximum (FWHM) converted to radians: FWHM * π /180, and θ is the Bragg angle.

The null hypothesis of this study is that translucency has no statistically significant relationship with the preferential orientations and grain sizes of the Y-TZP ceramics. Absolute translucency was compared for three recently developed commercialized high-translucent zirconia and one commonly clinically used lithium disilicate ceramic, and the relationship between translucency and the crystal lattice features of Y-TZP was analyzed.

Materials and methods

Sample fabrication

Nine discs of Y-TZP, including three common commercial brands, namely, LavaTM Plus (block #LOT 636535, $3 M^{TM}$ ESPETM, Deutschland GmbH Co., Neuss, Germany), VITA YZ® HT (block #LOT 57200, VITA Zahnfabrik H. Rauter GmbH & Co., Bad Sackingen, Germany), and NexxZr T (block #LOT WETCS, Sagemax Bioceramics Co., Washington, USA) were fabricated following the manufacturers' instructions. Three additional lithium disilicate glass-ceramic discs (e.max CAD®, block #LOT S12302, lvoclar Vivadent AG Co., Schaan, Principality of Liechtenstein) were prepared as a control group.

All green pieces before sintering were fabricated using a dental mill CAD/CAM machine (ARDENTA CNC MILL, CS100-5 A, ARIX Co., Tainan, Taiwan) to prepare pre-sintered porcelain blocks, which were then sintered by a ceramic furnace (VITA VACUMAT® 6000 M, VITA Zahnfabrik H. Rauter GmbH & Co., Bad Sackingen, Germany) following the manufacturer's instructions. The sintered specimen was gradually polished with water sandpaper from #600 to #2000. All specimens were unglazed, and surfaces were cleaned with 70% alcohol after polishing with an ultrasonic cleaner (DC200H, Delta Ultrasonic Co., New Taipei City, Taiwan). The standard dimension of all samples was $12 \text{ mm} \times 12 \text{ mm} \times .5 \text{ mm}$ (length x width x thickness) and verified to ensure that all thickness deviations were within 1%.

Translucency measurement

Absolute translucency was measured with an optical fiber spectrometer (USB 4000 spectrometer, Ocean Optics, Inc., Dunedin, FL, USA) following the manufacturer's instructions. SpectraSuite program (Ocean Optics, Inc.) was used to filter background radiation to obtain absolute translucency. The equipment setup and typical program running curves displayed on the screen are shown in Fig. 1. Each sample was measured five times and evenly distributed with a distance of 3 mm. The size of the test disc was $12 \text{ mm} \times 12 \text{ mm}$, which was 90,000 times that of an optical fiber probe (400 $\mu m \times$ 400 $\mu m);$ the requirements of American society for testing and materials (ASTM)¹¹ to test one sample by at least 20 times repeatedly was followed.¹⁸ Five average random positions of the same test disc can be regarded as different measurements. The observation wavelength adopted was the visible light wavelength range of 380-780 nm. All test discs were measured simultaneously in the same environment. Comparisons were performed in two different approaches. First, translucency obtained at a wavelength of 550 nm, the most sensitive to human eyes, was compared.^{3,19} Second, the translucency performance of each material at a 380-780 nm wavelength was compared by transforming it into a function.

X-ray diffraction

XRD analysis was performed using SHIMADZU LabX XRD-6000 (Shimadzu Co., Kyoto, Japan). Data were collected at a step size of 0.02° and a scan rate of 2 deg/min under the X-ray generated by Cu radiation (1.542 Å) at 40 kV, 30 mA, and a 2θ of $20^{\circ}-85^{\circ}$. The four highest Bragg angles of the corresponding preferential orientations of crystal lattice planes were used to obtain and compare peak intensities. The FWHM of the diffraction peak was used for crystal quantification and grain size calculation.¹⁵

Statistical analysis

The translucency values measured at 550 nm were compared using repeated-measure ANOVA. The translucency performance of each material at 380–780 nm was first transformed to a function via linear regression analysis. The crystal characteristics of each result (R-squared, 95% confidence intervals, slope, and intercept) were then compared. The statistical significance level was set at p < 0.05. Statistical analyses were performed using SPSS version 22 (SPSS Inc., Chicago, IL, USA).

Results

Translucency analysis

Translucency at 550 nm

Among the three Y-TZP crowns, LavaTM Plus was the most translucent (average translucency: $61.80\% \pm 1.49\%$), followed by NexxZr T ($60.24\% \pm 2.06\%$) and VITA YZ®HT ($59.02\% \pm 1.15\%$). Repeated-measure ANOVA showed no significant difference between these groups (Table 1). However, the Y-TZP groups were significantly less translucent compared with e.max CAD® ($78.83\% \pm .74\%$) (p < 0.001, Table 1).

According to the transparency performance at the most sensitive light wavelength of the human eye (i.e., 550 nm), $^{3,7,10,19-22}$ VITA YZ®HT and e.max CAD® showed a drop in translucency curve at two fluctuation wavelengths of 520 and 655 nm. LavaTM Plus was the most transparent, followed by NexxZr T and VITA YZ®HT (Fig. 2).

Translucency at 380-780 nm

The translucency performance at the wavelength range of visible light (380–780 nm) was transformed through linear regression by using the formula y (translucency) = ax (wavelength) + b (material constant). The formulas for 95% confidence interval for R^2 were as follows:

Lava TM Plus : $y = 0.0088x + 55.242$,	$R^2 = 0.9237.$ ((1))
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VITA YZ®HT : y = 0.0308x + 29.339, $R^2 = 0.9237$. (2)

NexxZr T: y = 0.3013x - 101.45, $R^2 = 0.9119$. (3)

$$e.\max CAD$$
[®] : $y = 0.0191x + 61.528$, $R^2 = 0.8466$. (4)

No significant difference was found in the absolute translucency performance of the three Y-TZP within 380–780 nm in terms of R^2 When the 95% confidence interval range was compared, the translucency of VITA YZ®HT at 380–544 nm wavelength was significantly different from that of the other two ceramics. At long wavelengths between 544 and 780 nm, the translucency values of the three Y-TZP were not statistically significant (Fig. 2). Regardless of whether R^2 or 95% confidence intervals overlapped, the glass-ceramic e.max CAD® consistently showed significantly higher translucency compared with the three Y-TZP ceramics (p < 0.001).

X-ray diffraction

Quantitative analysis of crystal preferential orientations and sizes

The obtained data were compared to the standard pattern of the Joint Committee on Powder Diffraction





Figure 1 Equipment setup (A) Simulation and configuration of absolute translucency measurement showing the relationship of the optical framework, testing probe, optic fiber, and integrating sphere (B) Absolute translucency measurement and typical screen on the SpectraSuite program.

Standards (JCPDS) cards #30-1468 by selecting four major intensity diffraction peaks of the Y-TZP and labeling them to the tetragonal phase transformation apparent during specimen sintering. The preferences at the respective tetragonal (h, k, l) corresponding to (111) (200) (220), and (311) planes shown at diffractionable peaks 2θ values of 30.1° , 34.9° , 50.2° , and 59.6° were indicated. Quantitative analysis was conducted on the Y-TZP corresponding to each crystal phase of the Y-TZP as related by the intensity at the planes (111) (200) (220), and (311). The results suggested that the preferential orientation of the tetragonal phase Y-TZP occurred after sintering. When the Y-TZP preferential orientations of (111) (200) (220), and (311) were calculated in descending order, the sequence order was Lava™ Plus > VITA YZ®HT > NexxZr T.

Scherrer's equation was used to calculate the crystal grain sizes (mean \pm standard deviation) of the three Y-TZP.¹⁷ LavaTM Plus showed the largest grain size (66.26 \pm 1.82 nm), followed by NexxZr T (65.84 \pm 4.77 nm) and VITA YZ® HT (64.30 \pm 3.73 nm).

Translucency and phasic quantitation for the Y-TZP ceramics

The relationship between the four major predominant preferential orientations of the tetragonal phase in the Y-TZP and the translucency was determined through linear mixed analysis, and the result is shown in Fig. 3. The diffraction plane of (111) was the main diffraction peak of the three Y-TZP ceramics. The phasic quantification and translucency of the Y-TZP positively correlated with each

Table 1 Comparison of translucency at 550 nm of all four ceramic materials by using the optical fiber spectrome						
	Lava™ Plus ^a	VITA YZ®HT ^b	NexxZr T ^c	e.max CAD® ^d		
Lava™ Plus	_	.151#	.977#	.000***		
VITA YZ®HT	.151#	_	1.000#	.000***		
NexxZr T	.977#	1.000#	_	.000***		
e.max CAD®	.000***	.000***	.000***	-		

^aLava™ Plus: #LOT 636535 of Y-TZP, 3M™ ESPE™; ^bVITA YZ® HT: #LOT 57200 of Y-TZP, VITA; ^cNexxZr T: #LOT WETCS of Y-TZP, Sagemax; ^de.max CAD®: #LOT S12302 of lithium disilicate glass-ceramic, Ivoclar.

All pairwise comparisons were set for $\alpha = 0.05$, # indicates no significant difference (p > 0.05); *** indicates significant difference (p < 0.0005).



Light Transmittance Between Different Materials



Figure 2 Absolute translucency at 380–780 nm of all four materials. (A) Scattering in the light color area represents the 95% confidence interval of the material's translucency and in the solid-colored line represents the average translucency of the material. (B) Light transmittance and related statistical analysis results at 550 nm are presented. (*p < 0.05, ***p < 0.0005).

other in LavaTM Plus and negatively correlated with each other in VITA YZ®HT and NexxZr T. The peaks diffracted at a 20 of 34.9° referring to the minor diffraction plane of (200) were positively correlated with the translucency of LavaTM Plus, VITA YZ®HT, and NexxZr T. The tetragonal crystal's preferential orientation of (220) and (311) planes showed that the phasic quantitation and translucency of Y-TZP were positively correlated in LavaTM Plus and VITA YZ®HT but negatively correlated in NexxZr T (Fig. 3).

Relationship between the translucency and grain sizes of Y-TZP

The relationship between grain size and translucency was analyzed with a linear mixed model. The grain size range of 57–70 nm was negatively correlated with translucency. In

Lava[™] Plus and VITA YZ®HT, grain size and translucency were significantly related. Therefore, the null hypothesis was rejected (Table 2 and Fig. 4).

Discussion

The results of the present study revealed a significant relationship between the translucency and grain sizes of Y-TZP ceramics; thus, the null hypothesis was rejected. Furthermore, translucency was statistically correlated with the preferential orientations of the Y-TZP's crystal tetragonal lattice. In terms of the influence of the predominated tetragonal planes in the three commercial types of Y-TZP, no significant difference was observed in the preferential orientations of the tetragonal phase. Furthermore, only the specific (200) plane can increase the translucency of the



Figure 3 Typical XRD patterns referring to the tetragonal planes of zirconia in the three commercial Y-TZP ceramics and the relationship between the quantitation of different predominant tetragonal phases with translucency.

Table 2	Relationship between the translucency at 550 nm and particle sizes of yttria-stabilized tetragonal zirconia polycrystal
(Y-TZP).	

	Lava™ Plus	VITA YZ®HT	NexxZr T	Multiple t tests
Coefficient	397	794	076	215
Significance	.000***	.000***	.789#	.157#

 $^{\#}$ indicates no significant difference (p > 0.05); *** indicates significant difference (p < 0.0005).

three commercially available zirconia of Y-TZP (Fig. 3). Future research in this direction is necessary.

Grain size within 54-70 nm (Fig. 4) was negatively correlated with translucency, and this finding is consistent with previous studies.^{14,23} The influence of translucency on grain size has been previously described.²⁴⁻²⁶

The current trend correlations showed that when translucency increased, grain size also increased to more than 1000 nm due to the decreased grain boundaries. Therefore, scattering, refraction, and reflection of light were reduced. On the contrary, limiting the grain size under 100 nm could increase the crystals'



Figure 4 Statistical analysis of the relationship between translucency at 550 nm and grain sizes of the three Y-TZP ceramics.

density and decrease the grain boundaries to increase translucency.

Brodbelt measured dentin as well as enamel and found that under the visible light range (380-780 nm),⁷ the translucency of a 1 mm-thickness enamel was similar to that of a .5 mm-thickness Y-TZP. In the current study, the translucency of a .5 mm-thickness e.max CAD® glassceramic was $78.83\% \pm .74\%$. Based on the results from this experiment, the transparency of e.max CAD® glass-ceramic was similar to that of human and bovine teeth. $^{25-27}$ Considering the difference in the restoration of tooth volume, the Y-TZP used in the present study could provide comparable translucency to reduce tooth volume.²⁷ On the contrary, using a ceramic material with higher translucency compared with those in the current study is recommended for bulky esthetic restorations. In clinical scenarios, the tooth is a multi-layered structure,²⁸ sometimes including ceramic materials, adhesive resin, enamel, dentin, pulp cavity-filling materials, and a hollow of pulp. The angle, curvature, and uneven thickness may cause difficulties in further designing similar experiments. Given that commercial Y-TZP are less transparent than tooth enamel, their application in replacing tooth enamel may be limited and requires further investigation.

Within the limitations of the study and based on the results presented, the following conclusions are drawn. The translucency of the three commercial highly translucent Y-TZP ceramics used in this study was significantly lower than that of lithium disilicate glass-ceramic material. The predominant phase of the Y-TZP ceramics was tetragonal polycrystal zirconia. No relationship was found between the preferred orientations and translucency of the three Y-TZP ceramics. However, the specific (200) plane was the only plane noted to increase the translucency of all three Y-TZP ceramics. At the grain size of 54–70 nm, the translucency of the Y-TZP ceramics was significantly negatively correlated with their grain size. Finally, the translucency of the three Y-TZP ceramics was lower than that of enamel, thus limiting their application as enamel replacement.

Declaration of competing interest

All authors declare no conflicts of interest.

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