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Edge Chipping of Translucent Zirconia

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Abstract

Statement of problem

More translucent dental zirconias have been developed by incorporating the cubic phase and reducing the tetragonal phase content that undergoes transformation toughening, leading to reduced mechanical properties.

Whether the clinically relevant mechanical property of the edge chipping toughness of the material is also reduced is unclear.

Purpose

The purpose of this in vitro study was to evaluate the edge chipping toughness and translucency of translucent zirconia, 3mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP), and lithium disilicate.

Material and methods

Two translucent zirconia products, Katana and Lava Esthetic; one 3Y-TZP, Lava Plus; and one lithium disilicate, IPS e.max Press were prepared and tested for phase composition via X-ray diffraction (XRD) (n=3), translucency via a spectrophotometer (n=20), and edge chipping via a universal testing machine with a custom-machined specimen holder and diamond indenter (n=20). The 3Y-TZP and lithium disilicate served as the optimal control materials for edge chipping and translucency, respectively. Translucency was compared with 1-way ANOVA and edge toughness with ANCOVA ($\alpha=.05$).

Results

The XRD showed the 3Y-TZP to be almost completely tetragonal phase compared with the 2 translucent zirconia products that were predominantly cubic. Katana UTML and IPS e.max Press had a statistically similar ($P>.05$) translucency that was significantly ($P<.05$) greater than that of Lava Esthetic and Lava Plus. The edge toughness of Katana UTML was 304 N/mm, IPS e.max Press was 354 N/mm, Lava Esthetic was 394 N/mm, and Lava Plus was 717 N/mm, with significance rankings of Katana UTM<IPS e.max Press=Lava Esthetic<Lava Plus.

Conclusions

Some translucent zirconias had translucency similar to that of lithium disilicate; however, as translucency increased with increased cubic content, edge toughness decreased.

Clinical Implications

The term translucent zirconia may be too broad a category, as products exist with a significant range of optical properties. Zirconia can be made more translucent by incorporating the cubic phase, but mechanical properties decrease. The dentist must be aware that as translucency improves, the preparation design should match that for a lithium disilicate restoration more closely than for a traditional 3Y-TZP zirconia restoration.

With its strength, ability to better match color, and lower cost than noble alloys, zirconia is a popular ceramic for different fixed dental prostheses. Zirconia can take 3 different crystallographic forms, dependent on temperature and pressure. From room temperature and pressure to 1170 °C, a larger, monoclinic structure is observed. As temperature increases up to 2370 °C, it acquires a smaller but stable tetragonal form and higher still up to the melting point (2680 °C) a cubic form.^{1,2} When zirconia is doped with certain lower valence oxides such as CaO, MgO, or Y₂O₃, the crystallographic forms normally seen at higher temperatures can be stabilized at room temperature. Using yttria (Y₂O₃), one of the most common dopants, the tetragonal phase can be stabilized with 2 to 5 mol% dopant concentration, and the cubic phase with an 8 mol% minimal dopant concentration.^{3,4,5} Under loading, the stabilized tetragonal phase can convert to the larger monoclinic phase, and this expansion, called transformation toughening, induces a compressive stress that inhibits crack propagation and creates high fracture toughness.¹

Three-mol percent yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is one of the most common variants of zirconia used in dentistry. Although 3Y-TZP should be entirely tetragonal in structure, some cubic zirconia may be present depending upon sintering conditions.⁶ The presence of cubic zirconia can be detrimental to the

desired tetragonal structure because it eliminates transformation toughening. Another disadvantage of 3Y-TZP is its high opacity compared with the traditionally more esthetic lithium disilicate materials.^{7,8}

If the yttria used as a dopant is increased to 8 mol%, a stable cubic phase of zirconia is produced that is isotropic in different crystallographic directions, decreasing light scattering. Despite a smaller grain size, cubic zirconia appears translucent.^{9, 10, 11} Thus, to make zirconia more esthetic, manufacturers stabilize the cubic form over the tetragonal form by using a higher dopant concentration. However, transformation toughening is adversely affected, and edge chipping becomes a problem for dental restorations made from high-cubic content translucent zirconias.

Zirconia doped with 5 mol% yttria (5Y-ZP) has been said to provide the esthetics of cubic zirconias and the strength of 3Y-TZP, although these claims lack independent verification. One such zirconia has been reported to have approximately 50% cubic structure.¹² Initial findings have indicated that the mechanical properties and esthetics of this material fall between 3Y-TZP and lithium disilicate.¹¹ While the mechanical properties may be better than those of lithium disilicate, the esthetic properties do not surpass the more translucent lithium disilicate.^{13,14} The transformation from tetragonal to monoclinic is desirable under stresses to limit crack propagation but is less desirable when it happens spontaneously at low temperatures. The zirconia doped with 5 mol% yttria has been reported not to be susceptible to low temperature transformation.^{15,16} The presence and percentage of each crystallographic structure can be determined through X-ray diffraction (XRD).^{17, 18, 19}

Since brittleness is characteristic of ceramic restorations, determining their chipping resistance is important as ceramic chipping is a common clinical and technical complication.²⁰ However, clinical chipping rates of monolithic restorations have rarely been reported²¹ compared with those of veneered restorations. An in vitro edge chipping test advances an indenter to near the material's edge until the force causes a fracture. Metal-ceramic and veneered zirconia specimens have been reported to have similar chipping patterns²² and are more susceptible to chipping than monolithic ceramic restorations.²³

The objective of this research was to compare the edge chipping toughness, translucency, and crystalline phases of translucent zirconia with those of 3Y-TZP and lithium disilicate. The null hypotheses were that no difference would be found in edge chipping toughness and translucency among 2 translucent zirconias, 1 3Y-TZP, and 1 lithium disilicate, with the 3 zirconia formulations having similar phase distributions.

Material and methods

Four materials were investigated: 2 translucent zirconia products (Katana UTML; Kuraray Noritake Dental Inc and Lava Esthetic; 3M ESPE), 1 traditional 3Y-TZP zirconia (Lava Plus; 3M ESPE), and 1 lithium disilicate (IPS e.max Press; Ivoclar AG). All products were Vita shade A2 with no glaze applied so that the base material could be evaluated.

Each zirconia product was soft milled and fully sintered according to the manufacturer's specifications into 10×10×1.5-mm specimens (n=25) for testing. The lithium disilicate was pressed to create material with greater mechanical properties than the milled, again, according to the manufacturer's recommendations. For translucency and edge chipping, a single individual (J.D.F.) hand ground the surface to be tested for 30 seconds by creating small circles on each silicon carbide abrasive disk with water and progressing from 240, 320, 600, and finally to 1000 grit (BuehlerMet; Buehler Ltd). The final polish was done with a polishing cloth (PoliCloth; Buehler Ltd), a 1.0-µm polishing paste (MicroPolish Alumina Suspension; Buehler Ltd), and water for an additional 30 seconds. This same process was used for each specimen to create a uniform, standardized, smooth surface.

An unground and unpolished specimen of each zirconia material (n=3) was analyzed via XRD (MiniFlex II; Rigaku Americas) from a 2θ of 10 to 90 degrees at 0.03 degrees/s by using Cu-Kα radiation of 1.54 Å with an excitation voltage of 30 kV and tube current of 15 mA. XRD patterns were qualitatively compared, and phase analysis was accomplished by using established methods.^{24, 25, 26} Integrated peaks for the monoclinic phase were quantified as between 27 and 32 degrees and for the cubic and tetragonal peaks between 72 and 76 degrees via Lorentz curve fitting.

To evaluate translucency, a spectrophotometer (CM-700d; Konica Minolta, Inc) was placed in a holding device. The polished surface of each ceramic specimen was placed over the orifice of the spectrophotometer so that the entire opening was covered. A standard white tile (Lucideon) was then placed over the specimen, and the L*a*b* values were recorded as the average of 3 measurements. D65 was used as the standard illuminant. Without moving the ceramic specimen, the white tile was removed and replaced with a standard black tile (Lucideon), and another reading was made. This was performed for each specimen (n=20).

From the L*a*b* values for both tile backgrounds, translucency was calculated by using:

$$TP = \sqrt{((Lb - Lw)^2 + (ab - aw)^2 + (bb - bw)^2)},$$

where TP is the translucency parameter and the “b” and “w” after L, a, and b values refer to black and white, respectively.²⁷ The TP was compared among the 4 materials via 1-way ANOVA and the Tukey honestly significant difference (HSD) post hoc test (α=.05) by using a statistical software program (SAS; SAS Institute Inc).

A custom specimen holder milled out of steel was used to hold each specimen for the edge chipping evaluation. The holder had an 11×11×4-mm central recessed area with a 2-mm round channel through the middle to allow dislodgement of the specimens. The recessed area was filled with composite resin (Z100; 3M ESPE) to simulate dentin. A glass slab was placed over the material to ensure a flat surface, and the material was polymerized for 40 seconds by using a light-polymerizing unit (BluePhase II; Ivoclar AG). Each ceramic specimen had the side to be bonded prepared according to the manufacturer’s recommendations: Lava Plus, Lava Esthetic, and Katana UTML were airborne-particle abraded with 50-µm aluminum oxide and thoroughly cleaned with alcohol; IPS e.max Press was etched with hydrofluoric acid (IPS Ceramic Etching Gel; Ivoclar AG) for 20 seconds and rinsed with water for 15 seconds.

The specimens were then silanated (Monobond Plus; Ivoclar AG) for 60 seconds. After each material was prepared, a self-adhesive universal resin cement (RelyX Unicem; 3M ESPE) was hand mixed and applied to the resin. The specimen was seated in a standardized manner and any excess cement was removed; the specimen was then light polymerized for 40 seconds.

The holder and specimen were fixed in a universal testing machine (Model 5500R; Instron) beneath a sharp conical head indenter with a diamond tip at 120 degrees and a tip sharpness under 5 µm (Gilmore Diamond Tools, Inc). The specimen and tip were positioned for the force to be applied between 0.1 and 1.0 mm from the edge of the specimen.²⁸ Two edge chipping tests were performed on opposite edges of the specimen by using a crosshead speed of 0.1 mm/min, and the maximum load (N) causing a chip was recorded (n=40). The distance from the specimen’s test edge to the indenter’s point of contact was measured by using a traveling microscope (TM-505 Microscope; Mitutoyo Corp).

Different methods have been used to evaluate edge chipping data with different forces and distances applied.²⁹ For the present research, 2 methods were used. The first was fitting the data to a straight line with an intercept of zero. By calculating the force over distance from the edge, the edge toughness can be found by using the equation: $F/d=T_E$, where F is the chipping force in N, d is the distance of the chip from the edge in mm, and T_E is the edge toughness in N/mm. The second method used a quadratic equation ($F=a_1d+a_2d^2$), where the

a_1 term is associated with surface phenomena and fracture energies while the a_2 term refers more closely to deformation.²⁹ With this equation, the 2 terms provide evidence of a brittle materials blending of deformation and fracture values that make up the indentation energy needed to produce the fracture.²⁹ The data points were graphed and fitted to the linear and quadratic equations. ANCOVA with Bonferroni corrections were used to compare the 4 materials with a statistical software program (SAS; SAS Institute Inc) ($\alpha=.05$).

Results

The XRD patterns for the 3 zirconia products are shown in Figure 1. Cubic and tetragonal zirconias possess a similar structure, and thus, the XRD patterns and peaks are close as evidenced by the 3 patterns in Figure 1. However, Lava Plus displayed the characteristic tetragonal peak splitting,³⁰ whereas Lava Esthetic and Katana UTML did not, indicative of an increased cubic structure. The 3 most intense tetragonal/cubic peaks were at approximately 30, 50, and 60 degrees, corresponding to the (111), (220), and (311) planes. The intensities of the monoclinic (-111) and (111) peaks at approximately 28.2 degrees and 31.5 degrees relative to the tetragonal and cubic peak at 30 degrees are often used for quantifying the amount of monoclinic phase,^{24,25} but there was no evidence of the monoclinic phase being present in the Lava Esthetic and Katana UTML zirconias. A small amount of monoclinic phase was observed in Lava Plus, however. Integrated intensities of tetragonal peaks for planes (004) and (400) at approximately 73.4 degrees and 74.7 degrees and a cubic peak (004) at approximately 74.2 degrees were used to estimate the phase composition of these respective phases via Lorentz curve fitting.²⁶ Overall, the estimated monoclinic/tetragonal/cubic phase content (%) was approximately 1.5/96.5/2 for Lava Plus, 0/41/59 for Lava Esthetic, and 0/27/73 for Katana UTML.

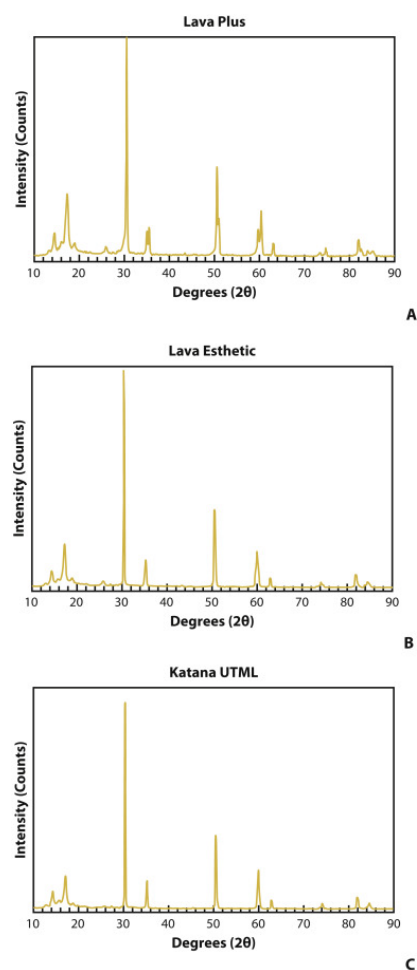


Figure 1. XRD patterns. A, Lava Plus. B, Lava Esthetic. C, Katana UTML.

The L*a*b* values on the white and black backgrounds, and the calculated translucency parameter are listed in Table 1. Table 2 details the ANOVA results, and Table 3 displays the Bonferroni pairwise comparisons. Changes in all 3 color coordinates (L*a*b*) were observed, not just the lightness (L*); specifically, both a* and b* became less positive/more negative, indicating a shift toward green and blue, respectively, when measured against the black background versus the white. Katana UTML and IPS e.max Press were significantly ($P<.05$) more translucent than Lava Plus and Lava Esthetic; otherwise, no statistically significant differences were found between pairings.

Table 1. Mean \pm standard deviation color measurements and translucency of IPS e.max Press, Lava Plus, Lava Esthetic, and Katana UTML

Material	White Background			Black Background			Translucency Parameter
	L*	a*	b*	L*	a*	b*	
IPS e.max Press	73.49 \pm 0.54	2.28 \pm 0.17	12.12 \pm 0.49	66.58 \pm 0.62	0.12 \pm 0.08	5.71 \pm 0.38	9.67 \pm 0.69 ^A
Lava Plus	87.43 \pm 0.16	-0.20 \pm 0.05	1.57 \pm 0.11	80.17 \pm 0.21	-0.80 \pm 0.03	-2.15 \pm 0.10	8.18 \pm 0.15 ^B
Lava Esthetic	75.88 \pm 0.34	2.84 \pm 0.11	14.89 \pm 0.61	71.45 \pm 0.27	0.35 \pm 0.07	8.73 \pm 0.73	7.99 \pm 0.48 ^B
Katana UTML	75.89 \pm 0.48	0.24 \pm 0.26	14.89 \pm 0.90	69.14 \pm 0.40	-1.69 \pm 0.10	7.87 \pm 0.92	9.96 \pm 0.52 ^A

Different superscript letters indicate statistically significant differences for translucency ($P<.05$).

Table 2. Descriptive statistics and ANOVA table for translucency of IPS e.max Press, Lava Plus, Lava Esthetic, and Katana UTML

Parameters	N	Mean \pm Standard Deviation	F Value	P	Partial Eta-Square (95%CI)		
Translucency	—	—	81.63	<.001	0.76	0.66	0.81
IPS e.max Press	20	9.67 \pm 0.69	—	—	—	—	—
Lava Plus	20	8.18 \pm 0.15	—	—	—	—	—
Lava Esthetic	20	7.99 \pm 0.48	—	—	—	—	—
Katana UTML	20	9.96 \pm 0.52	—	—	—	—	—

R-Square=0.76 (Adjusted R-Square=0.75).

Table 3. Bonferroni pairwise comparison for IPS e.max Press, Lava Plus, Lava Esthetic, and Katana UTML

(I) Group	(J) Group	Mean Difference	P	95% CI (Lower, Upper)	
IPS e.max Press	Lava Plus	1.50	<.001	1.07	1.93
IPS e.max Press	Lava Esthetic	1.68	<.001	1.25	2.11
IPS e.max Press	Katana UTML	-0.29	.458	-0.71	0.14
Lava Plus	Lava Esthetic	0.19	>.999	-2.45	0.61
Lava Plus	Katana UTML	-1.78	<.001	-2.21	-1.35
Lava Esthetic	Katana UTML	-1.97	<.001	-2.39	-1.54

CI, confidence interval.

Chipping force versus distance of the chip from the edge is plotted in Figure 2. Table 4 lists the linear regression analysis results. From the slopes formed by the linear regression with the intercept set at 0, the edge toughness of the materials were Katana UTML at 304 N/mm, IPS e.max Press at 354 N/mm, Lava Esthetic at 394 N/mm, and finally Lava Plus at 717 N/mm with significance rankings of Katana UTML < IPS e.max Press = Lava Esthetic <

Lava Plus ($P < .05$). Similar significance rankings were obtained with the quadratic equation method. Lava Plus possessed the greatest a_1 term associated with the fracture process, whereas Lava Esthetic displayed the greater a_2 term associated with deformation. Overall, IPS e.max Press exhibited more scatter in data compared with the 3 zirconia products.

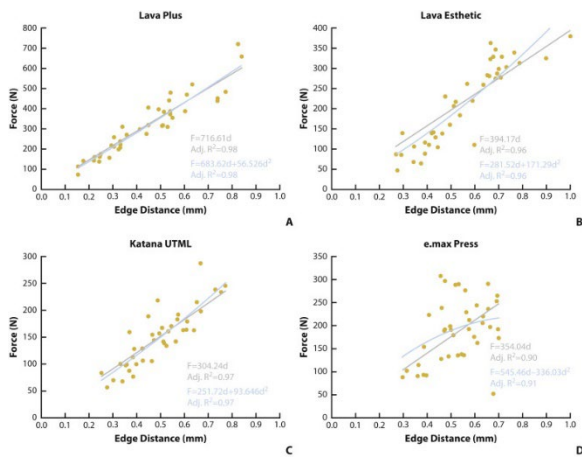


Figure 2. Chipping force versus edge distance. A, Lava Plus. B, Lava Esthetic. C, Katana UTML. D, IPS e.max Press. For each graph, red line fit to linear equation and blue line fit to quadratic equation via linear regression.

Table 4. Regression parameter estimates for IPS e.max Press, Lava Plus, Lava Esthetic, and Katana UTML

Group	Predictor of Force	Degrees of Freedom	R ²	Adjusted R ²	Parameter Estimate	95% CI for Parameter	P (t Test)
IPS e.max Press	d	1	0.90	0.90	354.04	316.23-391.23	<.001
	d ²	1	0.91	0.91	545.46 -336.03	334.79-756.12 -700.39-28 362	<.001 .070
Lava Plus	d	1	0.98	0.98	716.61	683.71-749.50	<.001
	d ²	1	0.98	0.98	683.62 56.53	567.86-799.39 -113.53-246.58	<.001 .551
Lava Esthetic	d	1	0.96	0.96	394.17	368.16-420.17	<.001
	d ²	1	0.96	0.96	281.52 171.29	181.90-381.15 24.50-318.07	<.001 .023
Katana UTML	d	1	0.97	0.97	304.24	287.02-321.45	<.001
	d ²	1	0.97	0.97	251.72 93.64	175.04-328.40 -0.39.67- 226.97	<.001 .163

CI, confidence interval; d, distance; d², distance squared.

Discussion

The null hypotheses were all rejected as significant differences were observed in the translucency and edge chipping toughness of the 4 ceramic products, as well as the phase distribution in the 3 zirconia products. Discriminating between tetragonal and cubic zirconia via XRD is challenging because of similarities in unit cells and overlapping peaks. Nevertheless, it was determined that Lava Plus was nearly all tetragonal and Lava Esthetic and Katana UTML were predominantly cubic phase (approximately 59% and 73%, respectively). These results were expected as Lava Plus has been reported to be 3Y-TZP and the 2 other zirconia products are

marketed as translucent, which would necessitate the inclusion of the cubic phase. The calculated phase analyses are generally consistent with those of previous research, where the amount of monoclinic phase in as-sintered Lava Plus has been determined to range from 0% to <1.5%^{31, 32, 33, 34} and the cubic content in as-sintered Katana UTML ranged from 69% to 75%.^{35, 36, 37} The authors are unaware of published data on the zirconia phase distribution of Lava Esthetic.

In the current research, the 3Y-TZP Lava Plus was comparatively opaque, having minimal difference between a white and a black background. The translucency of the translucent zirconia products, however, was brand dependent. While the lithium disilicate was more translucent than Lava Esthetic, the Kuraray product (Katana UTML) was statistically similar to IPS e.max Press ($P>.05$). This finding demonstrates that not all products marketed as translucent zirconias have the same optical properties. Katana UTML, with its greater translucency compared with Lava Esthetic, also had an increased cubic zirconia content, consistent with research that reported a linear relationship between translucency and cubic content.³⁶ More surprising was that the >96% tetragonal Lava Plus and the approximately 59% cubic Lava Esthetic zirconia products were not significantly different from one another in terms of translucency, suggesting that other factors such as grain size also influenced the relative translucency of zirconia. Previous research investigating the translucency of Katana UTML compared with IPS e.max has been conflicting. Baldissara et al⁷ reported Katana UTML to be more translucent, whereas others reported the opposite.^{11,13} Different specimen shapes and dimensions and translucency measurement methodologies may explain the conflicting results. However, the research that reported greater translucency for IPS e.max tested specimen thicknesses from 0.5 to 1.0 mm, but Katana UTML was more translucent at a thickness of 1.5 mm.⁷ The current study found similar translucencies between the 2 products using a thickness of 1.5 mm; this thickness was chosen to more closely replicate the thickness that would be seen in a clinical situation but may demonstrate a turning point where the translucent zirconia (Katana UTML) matches the translucency of the lithium disilicate.^{38,39}

Lava Plus had the highest edge chipping resistance, which was expected given its tetragonal structure, and reported high strength and fracture toughness.¹⁴ Lava Esthetic and IPS e.max Press exhibited a statistically similar ($P>.05$) edge toughness but were significantly greater ($P<.05$) than Katana UTML. As with the translucency results, the 2 translucent zirconia products differed in their edge chipping response. Interestingly, fitting the data of the zirconia products produced slightly more promising and consistent coefficients of determination (adjusted R^2) than the lithium disilicate. The Lava Plus produced the highest R^2 at 98%. Perhaps the transformation toughening characteristic of zirconia allows for a more predictable fracture as it would always have to overcome the toughening before a crack could propagate, and therefore, a more predictable chip. Lithium disilicate, with a heterogenous structure consisting of crystals within a glassy matrix and without the characteristic of transformation toughening, behaved more spontaneously and therefore less predictably. Authors noted that the lithium disilicate behaved anisotropically.⁴⁰ They theorized that this was because of the material's microstructure. The arrangement of the needle-like crystals embedded in the glassy matrix was determined by the pressing direction. The fractures would grow along this direction but would be resisted perpendicular to the crystals.⁴⁰

Limitations of the edge chipping test included that the chips were not always at a perfect 90-degree edge. Some of the chips were also difficult to measure because of a large portion of the material fracturing away. This was particularly problematic with the IPS e.max Press, where almost the entire edge to be measured fractured away, leaving a minimal point of reference to be used as a starting point for measuring. The lithium disilicate stands out in that it was pressed instead of milled. This process was chosen to produce a potentially high-strength alternative to the zirconia products.^{41, 42, 43} Despite this fact, the material was constructed in a thickness similar to that of a clinical crown and was cemented to a resin similar in physical properties to dentin. Finally, though many variations of the edge chipping test have been proposed, the process is yet to be standardized.⁴⁴

An increase in the cubic phase produces higher translucency zirconia but lower edge chipping resistance. Based only upon translucency and edge chipping toughness, the indication for the translucent products over lithium disilicate may be questioned. One exhibited equivalent translucency but less edge toughness, whereas the other was equal in edge toughness but more opaque. Further, the terminology “translucent zirconia” may be too broad when describing the category. Perhaps the material should be referred to by the percentage of each phase present so that a clinician can better predict its optical and mechanical properties. Additionally, zirconia has a reputation for being a strong material, but this implication is detrimental when that strength is significantly reduced for esthetic purposes. Clinically, traditional 3Y-TZP zirconia allows the practitioner to use a more conservative preparation design by maintaining outstanding mechanical properties at a thinner dimension. Based on the above results, a preparation design as used with a lithium disilicate may be indicated if a translucent zirconia is to be used. It is possible clinicians are under-reducing their preparations, believing the needed thickness for their material is less than it should be.

Conclusions

Based on the findings of this in vitro study, the following conclusions were drawn:

1. XRD analysis demonstrated that 3Y-TZP zirconia was 96.5% tetragonal, whereas translucent zirconias were primarily cubic in phase: 59% for Lava Esthetic and 73% for Katana UTML.
2. Certain translucent zirconia products have translucency similar to that of lithium disilicate.
3. The conventional 3Y-TZP product maintains a clear advantage in edge toughness, while the translucent products show an edge toughness closer to that of lithium disilicate.
4. “Translucent zirconia” may be too broad a term for a class of materials with a wide range of properties that depend on fabrication and phase composition.

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