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THE IMPACT OF PRE-SINTERING COLORING TECHNIQUES AND SINTERING PROTOCOLS ON TRANSLUCENCY OF MONOLITHIC ZIRCONIA WITH TWO THICKNESSES

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ABSTRACT

Aim: This study aims to evaluate the impact of different pre-sintering coloring techniques and different sintering protocols on the translucency of monolithic zirconia with two thicknesses.

Materials and Method: Sixty disc-shaped samples (12 mm x 12 mm) of high translucent zirconia (BSM Aconia: Chengdu Besmile Biotechnology Co. China) were fabricated and divided into 2 groups (n=30) according to thickness; T1 (0.5mm), T2 (1mm). Before sintering, each group was subdivided into 3 subgroups (n=10) according to coloring techniques; T1C1 and T2C1 (immersion only for 15 secs in A1), T1C2 and T2C2 (application by brush twice in T01 then immersion in A1 for 15 sec), T1C3 and T2C3 (application by brush three times in T01 then immersion in A1 for 15 sec). Each subgroup was furtherly classified into 2 classes (n=5) according to sintering protocols; S (Standard), F (Fast). A spectrophotometer was used to measure translucency. A three-way ANOVA and Bonferroni's post-hoc tests were utilized for statistical analysis. The significant level was set at ($P \leq 0.05$).

Results: T1 showed a higher mean value of TP (16.28) than T2 (10.25). C3 showed the highest mean value (14.2) followed by C2 (13.19) whereas; C1 showed the lowest mean value of TP (12.41). CL S recorded higher mean value of TP (13.51) than CL F (13.03).

Conclusions: For more natural appearance and translucent zirconia, it's preferable to reduce their thickness. Using brushing coloring technique with multiple applications significantly affected the translucency of zirconia. Standard sintering protocol ensures that zirconia restorations exhibit desired optical properties.

KEYWORDS High translucent zirconia, translucency, thickness, coloring, sintering.

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INTRODUCTION

The interest in natural-looking smiles has greatly increased among dental patients in recent years, fueled in part by a rising interest in cosmetic dentistry. The aesthetic appeal of zirconia restorations largely depends on their optical properties, including color, translucency, contrast ratio, light transmission, and opalescence.

Translucency is crucial for achieving a natural look, as it allows light to pass through. However, zirconia's natural opacity can hinder its aesthetic performance, leading to the use of layering materials to improve appearance. Despite this, issues with the chipping of layered porcelain have been noted. To address this, 5% mol Yttria was added to zirconia, creating translucent zirconia (5Y-TZP), which effectively resolves the problem [1-6].

Several translucent zirconia brands are currently available in the market, including Katana, Cercon, XTCERA, and BSM Aconia, all of which claim high translucency, flexural strength, and fracture toughness. Translucency of zirconia is affected by multiple factors, such as the crystal component configuration, the type and number of additives, the heating methods, the presence of light-scattering pores, the specific brand of zirconia-based all-ceramic systems, and the coloring agents used [7-11]. Also, both sintering time and temperature influence the grain size, thereby affecting zirconia's optical properties [12-18]. Moreover, these factors also determine the desired aesthetic outcomes. To enhance translucency, many zirconia manufacturers often increase the final sintering temperature.

Many advancements in monolithic zirconia have made it possible to use it in reduced thicknesses, which is essential for the success of all ceramic restorations. Thickness is a key factor in achieving aesthetic restorations, as decreasing the thickness can affect the translucency of the ceramic restoration. [19-24]

Despite advancements in manufacturing zirconia ceramics, achieving the desired translucency and shade reproduction remains challenging. Furthermore, the variety of coloring and sintering techniques offered by manufacturers adds to the confusion [25-30]. This study aims to clarify how pre-sintering coloring procedures and different sintering protocols impact the translucency of monolithic zirconia at two thicknesses.

Null hypothesis was that pre-sintering coloring techniques and sintering techniques may have an impact on the translucency of monolithic zirconia with two thicknesses.

MATERIALS AND METHODS

CAD/CAM white zirconia disc was used in this study. Brand name, shade, and volume of the used materials are shown in ***Table 1.***

TABLE (1) Brand name, shade, and volume of the used materials.

Material	Zirconia Disc	Coloring liquid	Special color
Brand name	BSM aconia	BSM aconia	BSM aconia
Shade	HT^{+} white	A1	T01
Volume	-	50 mm	20 mm

Samples Grouping

Sixty disc-shaped samples were tested in the current study and divided into two groups based on the tested thickness (n=30); group T_1 & T_2 (0.5 mm and 1 mm thick samples) respectively. Each group was subdivided into three subgroups (n= 10) based on the technique of coloring used before the sintering stage into; subgroups T_1C_1& T_2C_1 (Immersion in A1 coloring liquid for 15 seconds), T_1C_2& T_2C_2 (Application by brush twice using T01 special liquid then immersion in A1 coloring liquid for 15 sec) and T_1C_3 & T_2C_3 (Application by brush three times using T01 special liquid then immersion in A1 coloring liquid for 15 sec). Each subgroup was further divided into two classes (5 samples each) according to the used sintering protocol. Class T_1C_1S, T_1C_2S, T_1C_3S, T_2C_1S, T_2C_2S& T_2C_3S representing standard sintering protocol and T_1C_1 F, T_1C_2F, T_1C_3 F, T_2C_1F, T_2C_2F& T_2C_3F representing fast sintering protocol.

Samples Preparation

Twelve zirconia cylinders (12 x 12 x 10 mm) were designed using inLab CAD software and then milled from HT^+ zirconia blanks using a milling machine (inLab MC X5; Dentsply Sirona Co. U.S.A.). To account for sintering volumetric shrinkage, all cylinders were cut 20% larger than the desired dimensions and then finished by Profi (Q Profi ST; Schick Gmbh. Schemmerhofen. Germany).

Sixty disc-shaped samples were prepared from the pre-sintered zirconia cylinders. The cylinders were cut by a linear microtome (Isomet 4000; Buehler Co. U.S.A.) with a 20 cm diameter, 0.6 mm thick diamond disk running at 2500 rpm under continuous water coolant. The integrated cooling system ensured that samples were flooded from both sides of the cutting blade.

In their pre-sintered state, 30 disc-shaped samples measured (12 x 0.6 mm) and were designed to achieve a final thickness measuring 0.5 mm, and another 30 discs measuring (12 x 1.2 mm) to reach a final thickness of 1 mm after sintering, representing Group T_1 and T_2, respectively. The dimensions of the samples were verified using a digital caliper (TOTAL Bcoolyz Caliper; China) immediately after sectioning in their pre-sintered state. After the sintering process, these dimensions were rechecked to achieve the desired final thickness.

Samples coloring

White pre-sintered HT^+ zirconia samples of each group were colored before sintering and subdivided into three subgroups (n= 10) based on the technique of coloring used before sintering into; T C_1 (Immersion in A1 coloring liquid for 15 seconds); T C_2 (T01 special liquid brushed twice on sample surfaces, then immersion in A1 coloring liquid for 15 seconds), and T C_3 (T01 special liquid brushed three times, then immersion in A1 coloring liquid for 15 seconds).

After applying each coloring technique, the samples were left to dry on paper tissues to remove excess coloring liquid, following the manufacturers' guidelines. Once the samples were dried, each subgroup's samples were placed in coded sterilization pouches indicating their coloring technique ready for sintering.

Sintering of samples

Each subgroup was further divided into two classes (n=5) based on the sintering protocol used; CL S (standard sintering) and CL F (fast sintering). A zirconia sintering furnace (Tabeo sintering oven zirconium; MIHM-VOGT, Inc. MI, U.S.A.) was employed for both standard and fast sintering processes.

Colored samples from each subgroup were placed on the sintering plate and sintered according to the specified protocol, cut coded parts of a zirconia disc indicating the number of each subgroup and the sample thickness were used as separators.

For standard sintering, samples were placed in a sintering furnace that was calibrated according to the manufacturer's instructions for 9 hours and 40 minutes (580 minutes). ***Table 2***

TABLE (2). Standard sintering steps

Step	Initial temperature (°C)	Final temperature (°C)	Time (min)	Heating rate (°C /min)
1	50	300	90	2.8
2	300	1520	240	5
3	1520	1520	90	Holding
4	1520	800	160	-4.5
5	800	Natural cooling		

For fast sintering, samples were placed in a sintering furnace calibrated according to the manufacturer's instructions for 1 hour and 50 minutes (110 minutes). ***Table 3***

TABLE (3) Fast sintering steps

Step	Initial temperature (°C)	Final temperature (°C)	Time (min)	Heating rate (°C /min)
1	50	200	15	10
2	200	1000	20	40
3	1000	1550	25	22
4	1550	1550	30	Holding
5	1550	1000	20	-27.5
6	1000	Auto cooling		

Translucency Measurement

A spectrophotometer (Cary 5000 UV-Vis-NIR, Agilent, U.S.) was used to assess the color parameters L*, a*, and b* of each specimen. Measurements were performed at the center of each specimen over a standardized white (CIE L*=95.35, a*=-1.31, b*= -0.27) and black (CIE L*=0.01, a*=-0.02. b*=0.01) backgrounds.

Samples were positioned at the center of the measuring port and maintained in the same position for both backgrounds. Each specimen was measured three times on each background, and an average of the three measurements was calculated to obtain a single value for each of the color coordinates for each specimen on each background. Translucency was assessed using the Translucency Parameter (TP), calculated based on the following equation: TP $= [(L^*_B - L^*_W)^2+(a^*_B - a^*_W)^2+(b^*_B - b^*_W)^2]^{1/2}$, where subscript B corresponds to color parameter when using a black background and subscript W for white background. L* represents lightness which ranges between 0&100, a* represents the red-green axis, and b* represents the yellow-blue axis. Each sample had its mean TP value recorded and tabulated on computer software (Microsoft Excel spreadsheets, Microsoft) ready for statistical analysis.

Statistical Analysis

A three-way ANOVA test was used to study the effect of thickness, coloring technique, sintering technique, and their interactions on mean values of (TP). Bonferroni's post-hoc test was used for pair-wise comparisons when the ANOVA test was significant. The significance level was set at $P \le 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

RESULTS

Effect of thickness regardless of coloring technique and sintering technique

Samples of Gp T_1 (0.5 mm thick) showed statistically significant higher mean values of TP than those of Gp T_2 (1 mm thick). (*P*-value <0.001, Effect size = 0.998) as shown in ***Table 4 and Figure 1***.

TABLE (4) Translucency parameter means, and standard deviation (SD) values of the two tested thicknesses regardless of coloring technique and sintering technique.

0.5 mm thickness		1 mm thickness		*P*-value	*Effect size (Partial Eta squared)*
Mean	SD	Mean	SD		
16.28	1.01	10.25	0.6	<0.001*	0.998

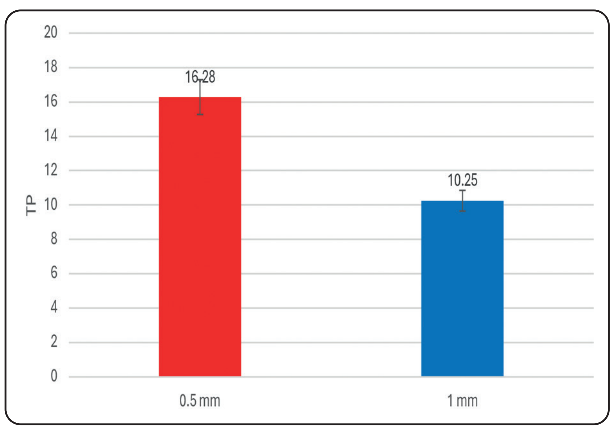

Fig. (1). Mean and standard deviation values of (TP) of the tested thicknesses regardless of coloring and sintering technique.

Effect of coloring technique regardless of thickness and sintering technique

Table 5 and Figure 2 showed that there was a statistically significant difference between the tested coloring techniques (*P*-value <0.001, Effect size = 0.969). Pair-wise comparisons between coloring techniques revealed that subgpC $_3$; Application by brush three times followed by immersion (14.2) recorded the highest statistically significant mean values of (TP), followed by subgp C $_2$; Application by brush twice followed by immersion (13.19). Whereas subgp C $_1$; Immersion only showed the lowest statistically significant mean values of (TP).

Effect of sintering technique regardless of thickness and coloring technique

Samples of CL S (standard sintering) recorded higher statistically significant mean values of TP than those of CL F (fast sintering). (*P*-value <0.001, Effect size =0.765). as shown in ***Table 6 and Figure 3.***

TABLE (5). Translucency mean and SD values of the tested coloring techniques regardless of thickness and sintering technique.

Immersion		Application by brush twice then immersion		Application by brush three times then immersion		*P*-value	*Effect size (Partial Eta squared)*
Mean	SD	Mean	SD	Mean	SD		
12.41 ^{C}	2.88	13.19 ^{B}	3.14	14.2 ^{A}	3.32	<0.001*	0.969

TABLE (6). TP mean and SD values of the tested sintering techniques regardless of thickness and coloring technique.

Standard sintering		Fast sintering		*P*-value	*Effect size (Partial Eta squared)*
Mean	SD	Mean	SD		
13.51	3.26	13.03	3.07	<0.001*	0.765

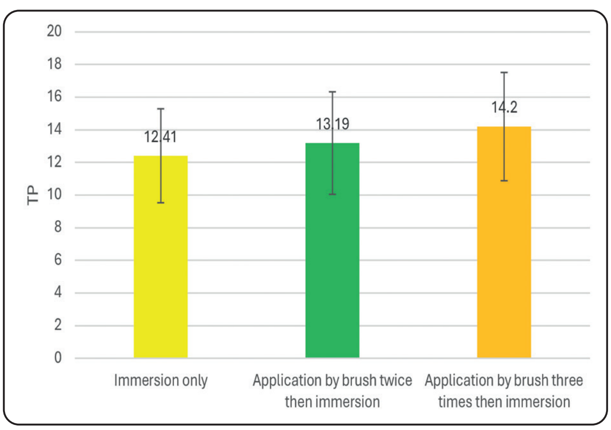

Fig. (2). Mean and SD values of (TP) of the tested coloring techniques regardless of thickness and sintering technique.

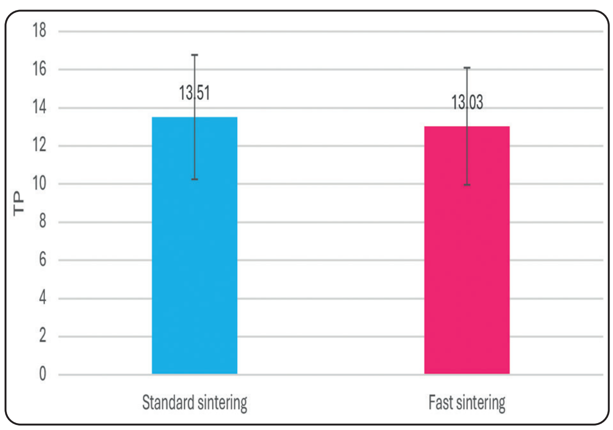

Fig. (3). Mean and SD values for TP of the tested sintering techniques regardless of thickness and coloring technique.

TABLE (7) Shows the Mean and SD values of TP of sintering techniques with different variables.

Coloring technique	Thickness	Standard sintering		Fast sintering		*P*-value	Effect size *(Partial eta squared)*
		Mean	SD	Mean	SD		
Immersion	0.5 mm	15.65	0.15	14.7	0.14	<0.001*	0.698
	1 mm	9.73	0.07	9.54	0.14	0.039*	0.086
Application by brush twice then immersion	0.5 mm	16.46	0.18	16.02	0.14	<0.001*	0.312
	1 mm	10.27	0.13	10.01	0.17	0.007*	0.141
Application by brush three times then immersion	0.5 mm	17.73	0.17	17.11	0.18	<0.001*	0.479
	1 mm	11.17	0.14	10.8	0.14	<0.001*	0.239

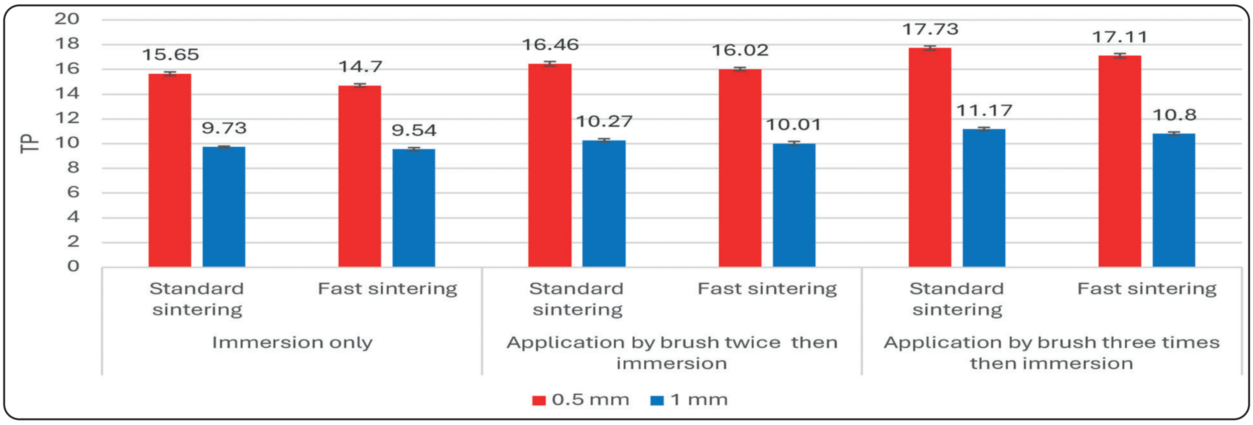

Fig. (4). Translucency mean values and SD of different variables interactions.

Effect of Variables Interactions on Translucency Parameter

Figure 4 showed that Gp T_1 0.5 mm thickness samples of all tested coloring techniques whether using conventional or rapid sintering, showed statistically significant higher TP mean values than those of Gp T_2 1 mm thickness.

DISCUSSION

Advancements in technology and procedures have elevated patients' expectations of receiving highly aesthetic restorations with superior mechanical properties. The use of ceramic restorations in routine dental care has significantly increased due to the rising standards and demand for aesthetics. The aesthetic appeal of a metal-free restoration is determined by how closely it replicates the original tooth in shape, surface texture, ideal color, and translucency.

Zirconia's high reflectivity and low light penetration give it a whitish, opaque appearance, diminishing its aesthetic appeal. To address this, a ceramic veneer should be used to cover the zirconia base in visible areas. Bilayer restorations consist of a durable zirconia core and an aesthetically pleasing ceramic veneer, both of which are biologically compatible. However, due to the differing thermal expansion coefficients of the two ceramic materials, the ceramic veneer typically has low fracture toughness when subjected to tensile forces. Consequently, separation or chipping of ceramic veneers is common, leading to significant challenges in dentistry. [1, 5, 31] [32, 33]

The translucency of zirconia restorations is a crucial factor in choosing the desired aesthetic restorations to achieve a natural-looking appearance. This translucency is determined by the amount of light transmission. The light scattering in zirconia is influenced by the ceramic's microstructure, including its grain size, porosity, and contaminants. Because coloring solutions contain metal oxides that cause light scattering, monolithic zirconia may become less translucent. Additionally, the translucency of zirconia is affected by the ceramic brands, thickness, shade, grain size, refractive index, environmental conditions, sintering process, surface roughness, contrast ratio, acidic environments, and coloring technique. [6, 34, 35] [36] [37]

Due to its enhanced mechanical properties, even at reduced thicknesses, monolithic zirconia restorations can serve as an alternative to traditional veneered zirconia. [21]

This study aimed to evaluate the effect of different pre-sintering coloring techniques, and different sintering protocols on the translucency of monolithic zirconia aesthetic restoration with two different thicknesses to enhance the aesthetic properties of zirconia restorations and provide an aesthetic restoration with a natural appearance that combines both the mechanical and aesthetic properties of zirconia. Bsm Aconia was used in this study; a high-translucent white yttria-stabilized tetragonal zirconia material that includes 3% mol of yttria in its composition (3Y-TZP), which is currently available on the market.

According to the results presented in this study, the null hypothesis was confirmed. The Three-way ANOVA test revealed that thickness (regardless of coloring and sintering techniques), coloring technique (regardless of thickness and sintering technique), and sintering technique (regardless of thickness and coloring technique) each had a statistically significant effect on mean TP. Moreover, the interactions between these factors also had a statistically significant effect on mean TP, indicating that the factors are dependent upon each other.

Regarding thickness, the findings showed that group T_1 showed statistically significantly greater mean values of TP than those of group T_2. This was in agreement with Fonseca Y. R., et al. (2019)[10], Alameldin, A.M.L., et al. (2020)[21], Hajhamid, B., et al. (2024)[38] and Kang, C.M., et al. (2024)[37] who reported that the material's translucency is affected by its thickness because, as thickness increases, crystallinity increases and the interaction between elements that scatter the incoming electromagnetic wave also increases, leading to a reduction in the material's translucency. They concluded that decreasing the thickness elevated the translucent properties of the zirconia material and related that to the polycrystalline nature of zirconia.

Also, these findings were in accordance with Supornpun, N., et al. (2023)[23], who stated that the kind, thickness, and shade of ceramic substance all had an impact on the translucency parameters as well as the degree of light transmission. It was also in accordance with Steven, C.D., et al. (2024) [39] who reported that there is a significantly strong negative association between thickness and TP of zirconia.

Regarding coloring, the results revealed that the coloring technique affected the translucent properties of monolithic zirconia showing that the highest statistically significant mean TP values were observed in subgroup C_3, followed by subgroup C_2. In contrast, subgroup C_1 exhibited the lowest statistically significant mean TP values. This was in agreement with Yılmaz Savaş, T., & Akın, C. (2022)[40] who found that the amount of shading solution applied during brushing may influence the translucent properties of zirconia restorations and Chen, Z., et al. (2024)[36] who reported that the color of the shading solution and the duration of immersion affected the translucent properties of monolithic zirconia, potentially causing undesirable or noticeable variations in both color and translucency.

However, there is a lack of agreement in some dental studies as Sen, N., et al. (2018)[14] who revealed that both the biaxial strength and translucency of monolithic zirconia were not significantly affected by the independent factor of the coloring procedure. The variations in results could be attributed to differences in methodology, including the coloring procedures, test parameters, sample size, zirconia manufacturers, type of coloring solution, and timing.

Regarding the findings of sintering protocols; samples of **class S** (standard sintering) showed higher statistically significant mean values of (TP) than those of **class F** (fast sintering). this was in agreement with Juntaveem, N., & Attashu, S. (2018) [41] and Al-Zordk, W., & Saker, S.[2] who stated that the sintering procedure had an impact on the translucent properties of the translucent zirconia. Translucent properties are enhanced upon raising the sintering temperature and extending the sintering duration. This is also in accordance with Rezeika, Y.I., et al. (2023)[42] who reported that the best results of the study were achieved after completing both the speed sintering cycle and the traditional sintering period. As a result, aesthetic zirconia restorations still require extended sintering times, limiting chair-side procedures to non-aesthetic areas. Salah, K., et al. (2023)[43] and Alshahrani, A.M., et al. (2024)[44] revealed that translucency of cubic and tetragonal zirconia decreased after being subjected to either speed or super speed sintering. Steven, C.D., et al. (2024)[39] stated that there is a positive association between sintering time and translucency parameters of zirconia.

On the other hand, these findings were against Ibrahem, A.S., et al. (2022)[45] who claimed that the shade and translucency of the final restoration would remain unaffected by the ZirCAD Prime speed sintering process. However, this assertion was disputed due to differences in the methodologies used and the various brands of monolithic zirconia selected, which likely led to contrasting results and opinions on the process's effect on zirconia's aesthetic qualities.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

1. Zirconia's translucency and natural look are better achieved by reducing its thickness.
2. Brushing coloring technique and increased number of applications by brush followed by immersion produce monolithic zirconia restorations that are more translucent than immersion in coloring liquid only.
3. Standard sintering protocol produces monolithic zirconia restorations with enhanced translucency as compared to fast sintering.

RECOMMENDATIONS

- It is recommended to use thin-thickness restorations for anterior teeth to produce natural-looking translucent monolithic zirconia aesthetic restorations.
- In cases where high translucency is required, it is advisable to use standard rather than fast sintering protocol.
- Further investigations regarding other optical properties and different monolithic zirconia brands available in the market are advised.

LIST OF ABBREVIATIONS

HT**:** High translucent; YSZ: Yetteria-stabilized zirconia.

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