Influence of Thermal Expansion on the Bond Strength of Nanozirconia and Veneering Ceramics

Ahlam Almoualimy

Universiti Kebangsaan Malaysia

Hsu Zenn Yew (✉ hz_yew@ukm.edu.my)

Jasmina Qamaruz Zaman

Universiti Kebangsaan Malaysia

Norziha Yahaya

Universiti Kebangsaan Malaysia

Andanastuti Muchtar

Universiti Kebangsaan Malaysia

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Abstract

This study was aimed to determine the coefficient thermal expansion (CTE) of colloidal-processed slip-casted novel nanozirconia (Zir) and to elucidate the effect of the differences in CTE values between zirconia cores and veneering porcelains on core–veneer shear bond strength (SBS). CTE measurement was performed on core materials (CMs; Zir and Cercon® [Cer]) and veneering ceramics (VCs; Ceram Kiss® [CK], VITA VM9® [VM9], and IPS e.max Ceram® [e.max]). The SBS test was conducted on six groups of core–veneer samples (Zir–CK, Zir–e.max, Zir–VM9, Cer–CK, Cer–e.max, and Cer–VM9). The mean CTE values ($\times 10^{-6}/^{\circ}\text{C} \pm$ standard deviation) of Zir and Cer were 7.86 ± 0.47 and 7.82 ± 0.49 , respectively. CK, VM9, and e.max recorded mean CTE values of 7.20 ± 0.62 , 7.20 ± 0.29 , and 7.10 ± 0.46 , respectively. No statistically significant differences were found among the CTE values of CMs and between those of VCs. Zir–VM9 recorded the highest mean SBS value (MPa) of 149.48 ± 67.64 , whereas Cer–e.max produced the lowest SBS value of 44.07 ± 16.49 . Combinations with high CTE disparities produced low SBS values. CTE discrepancies affected the SBS of veneered zirconia restorations. The novel zirconia showed similar performance and can be used as an alternative to commercial zirconia.

1. Introduction

The use of nanozirconia as a dental restorative material has become increasingly popular due to its excellent mechanical, biological, and aesthetic properties [1, 2]. In restorative dentistry, zirconia is commonly used as a core or framework and laminated with feldspathic porcelains [3]. Clinical evaluations have indicated the good success rates of zirconia-based restorations [3, 4]. However, one of the major clinical setbacks of veneered zirconia restorations is the delamination of porcelain from the zirconia core [5]. The chipping of veneering porcelain in zirconia-based restorations occurs by as early as 24 months of function. The chipping rate in zirconia-based restorations is frequently higher than that in conventional metal–ceramic restorations [5]. Clinical studies suggest that despite the excellent physical properties of the zirconia core, the layering porcelain remains susceptible to chipping.

The actual cause of chipping is unknown, but literature suggests that it can be attributed to the bond failure between zirconia and the veneering porcelain [6]. The zirconia core–veneer bond strength is believed to be influenced by various factors, including the coefficient of thermal expansion (CTE) [7]. A slight CTE mismatch is beneficial for creating favorable compressive stresses in the veneering porcelain during cooling [7]. However, the ideal range of CTE mismatch has yet to be established.

In dentistry, a restorative framework/core is usually milled from a zirconia block produced with nanozirconia powders [8]. However, nanoparticles are susceptible to agglomeration, which results in inferior-quality blocks [9, 10]. By utilizing a special colloidal processing technique, zirconia powder with good particle homogeneity and a packing density of almost 100 wt% can be developed [11]. The combination of wet-processing and slip-casting techniques allows the fabrication of machinable zirconia blocks with good mechanical and aesthetic properties [12–15]. Furthermore, slip-casted zirconia blocks offer an economical alternative to the traditional compaction process [12] because zirconia slurry can be

poured manually into special porous molds (plaster mold) to form zirconia frameworks without the need for specialized machines [13, 14]. Novel nanozirconia achieves sufficient bond strength with the tested veneering porcelain [15].

These preliminary results suggest that experimental nanozirconia (Zir) holds a great potential for use in restorative dentistry. However, to the best of the authors' knowledge, the CTE of zirconia fabricated through this unique method remains undetermined. Therefore, this study aimed to evaluate the CTE values of colloidal-processed slip-casted Zir and to elucidate the effect of the differences between the CTE values of the zirconia core and veneering porcelains on their core–veneer shear bond strength (SBS).

2. Materials And Methods

The current study utilized two core materials (CM; Zir and Cercon® [Cer]) and three veneering ceramics (VCs) (Ceram Kiss® [CK], VITA VM9® [VM9], and IPS e.max Ceram® [e.max]). Five samples were used for CTE measurement, and six core–veneer assemblies with eight samples were utilized for the SBS test. Table 1 shows the materials used in this study.

Table 1
Core and veneering ceramic materials and their chemical compositions

Materials	Brand/ Manufacturer	Contents wt%	Sintering Temp.	Duration
CM	Zir	99.9% pure Zir powder. 3 mol% yttrium oxidz	1500 °C	2 h
	Cer	Degudent®, GmbH, Germany 92% zirconium oxide 5% yttrium oxide 2% hafnium oxide 1% alumina and silica	1350 °C	90 min
VC	CK	Degodent GmbH, Germany SiO ₂ 60–70%; Al ₂ O ₃ 7.5–12.5%; K ₂ O 7.5–12.5%; Na ₂ O 7.5–12.5%	830 °C	6 min
	VM9	VITA Zahnfabrik H, Rauter GmbH & Co.KG,Bad Sackingen, Germany SiO ₂ 60–64%; Al ₂ O ₃ 13–15%; K ₂ O 7–10%; Na ₂ O 4–6%; B ₂ O ₃ 3–5%	910 °C	14 min
	e.max	Ceram/Ivoclar Vivadent, Schaan, Liechtenstein SiO ₂ 60–65%; Al ₂ O ₃ 8–12%; K ₂ O 6–8%; Na ₂ O 6–9%; ZnO 2–3%; CaO, P ₂ O ₅ and F 2–6%; other oxides 2–8.5%; pigments 0.1–1.5%	650 °C	4 min

2.1. Coefficient Thermal Expansion Measurement

2.1.1. Preparation of core materials

Five cylindrical (6 mm in diameter and 24 mm in length) samples of Zir were fabricated via the colloidal technique and slip-casting process. By using the dispersing technique parameters described by Amat et al. [14], a nanozirconia suspension was prepared by mixing nanozirconia powder with distilled water at 1:1.25 weight ratio. Then, 0.5 wt% polyethyleneimine was added to the mixture to disperse the agglomerated zirconia particles, and the suspension pH was adjusted to 2 (Five Easy F20, Mettler Toledo, Swiss) through the addition of hydrochloric solution. Under a magnetic vibrator, the mixture was stirred for 45 min before being transferred to an ultrasonic chamber (Ultrasonik 28X, NDI, California USA) at 50 Hz for 15 min. The dispersed zirconia slurry was consolidated through the slip-casting method into a cylindrical plaster of Paris mold. The samples were left for 3 days to ensure complete hardening before finally being sintered at 1500 °C for 2 h to obtain cylindrical samples with dimensions of 6 mm diameter and 24 mm length [16].

Five cylindrical samples were milled (Cer, Brain Expert, DeguDent GmbH, Germany) from Cer zirconia blanks. The samples were sintered at 1350 °C for 90 min (Cer, Heat Plus, DeguDent GmbH, Germany) to obtain the final cylindrical dimensions (6 mm diameter and 24 mm length) [15].

2.1.2. Preparation of veneering ceramics

The powder and liquid VCs were mixed and packed manually into a customized cylindrical rubber silicone mold. The surface of the fully packed mixture was blotted by using thin facial tissue, and the ceramics were transferred to the furnace for sintering in accordance with the manufacturers' instructions (Table 1). After sintering, the samples had the final dimensions of 6 mm diameter and 24 mm length [17]. Figure 1, illustrates the fabrication process of core and veneering materials.

2.1.3. Determination of coefficient of thermal expansion

The specimens were placed in a rod dilatometer device (L75/1550, LINSEIS, Germany) connected to a software (Rod alumina, software LINSEIS, Germany). Zirconia samples (Zir and Cer) were placed in the dilatometer and heated from room temperature to 1400 °C at a heating rate of 5 °C/min. After the temperature reached 1400 °C, the samples were kept for 1 min and then slowly cooled down to 500 °C. The CTE was determined at room temperature at a heating rate of 5 °C/min to 1400 °C.

The CTE of the VCs (CK, e.max, and VM9) was determined beginning from room temperature at a heating rate of 5 °C/min until the inversion of the heating curve (softening temperature) was reached. The CTE of VCs was determined up to the melting point.

2.2 *Measurement of Core–Veneer Shear Bond Strength*

A total of 24 discs of Zir and Cer (10 mm diameter and 3 mm thickness) were fabricated in accordance with the protocols explained in the previous section (2.1.1).

Sintered CM discs (Zir and Cer) were sandblasted with 50 μm Al_2O_3 at 0.2 MPa at a distance of 1.0 cm for 20 s and then cleaned with acetone for 10 min in an ultrasonic bath. Prior to the placement of the VCs, a piece of plastic tape was placed on each disc to define the bonding area. The powder and liquid VCs were mixed and packed onto the defined bonding area of the core discs by using a metal split mold. Packing was conducted incrementally, and manual vibration was performed to remove entrapped air bubbles. Water particles were removed through blotting with thin facial tissue prior to firing in a furnace. All samples received a layer of VC and fired to obtain 3 mm-thick veneers. The samples were cleaned by using steam, ultrasonicated with distilled water for 30 min, and stored for 24 h at 37 °C before the SBS test [18]. Samples were grouped as Zir-CK, Zir-e.max, Zir-VM9, Cer-CK, Cer-e.max, and Cer-VM9.

The SBS test was performed by using a universal testing machine (AGS-1000D, Shimadzu® Co., Japan) with a 5 kN load cell. A customized acrylic resin jig was fabricated to facilitate holding of the samples during the test Fig. 2. The shearing process was completed at a crosshead speed of 0.5 mm/min until all samples debonded. The bonding strength was calculated by using the formula [load (N)/area (mm^2)] and recorded in megapascals (MPa) [19, 20]. Prior to each procedure, calibration was performed, and the jig position was set to zero.

Data analysis was performed by using SPSS version 24 (IBM, USA). One-way analysis of variance (ANOVA) and Tukey's post-hoc test were used to determine significant differences in the CTE values of CMs and VCs at $p < 0.05$. Pearson's correlation coefficient test was used to determine the correlation between mean SBS values and mean CTE values of core/veneer assemblies.

3. Results And Discussion

- *Determination of the coefficient of thermal expansion of core materials (Zir and Cer), and veneering ceramics (CK, VM9, and max)*

Figure 3 depicts the materials with their CTE values. All materials behaved similarly at the beginning of temperature application. From room temperature up to 100 °C, the CTE values exponentially increased before decreasing. The CTE values of the materials stabilized at 100 °C. However, a further increase in temperature resulted in a gradual increase in CTE. Both CMs exhibited linear expansion with a small increment between each point of temperature degrees. At each 20 °C interval, the expansion was approximately $0.1 \times 10^{-6}/^\circ\text{C}$. The maximum expansion of $8.6 \times 10^{-6}/^\circ\text{C}$ for Zir and Cer occurred at 1400 °C. Both CMs showed comparable CTE values at the same temperatures. The resemblance between the results might be attributed to the similarity in the compositions of Cer and Zir (Table 1).

The thermal expansion of VCs was similar to that of the CMs to a certain extent. The thermal expansion of the VCs began with a drastic expansion which then dropped, followed by a gradual increase with the increase in temperature until the VCs reached

their melting points. The melting points of CK, VM9, and e.max are 615 °C, 686 °C, and 572 °C, respectively. Beyond their melting points, the VC stopped expanding and shrunk.

Table 2 shows the differences in the mean CTE values ($\pm$ standard deviation (SD)) of veneering and CMs. The mean CTE of Cer was slightly lower than that of Zir. However, these results were not statistically significant. The mean CTEs of CK and VM9 were almost the same. Although the mean CTE value of e.max was slightly lower than that of CK and VM9, no significant differences were observed between the CTE values of all the VCs. The difference between the CTE of e.max and that of CK and VM9 may be attributed to the similarity in the chemical composition of CK and VM9. By contrast, e.max contains additional chemical contents as shown in Table 1. The results of this study are supported by those reported by Juntavee and Dangsuwan [17]. They observed that the CTE values of CK ($10.03 \times 10^{-6}/^{\circ}\text{C}$) and VM9 ($9.93 \times 10^{-6}/^{\circ}\text{C}$) were higher than that of e.max ($9.86 \times 10^{-6}/^{\circ}\text{C}$). Similarly, the CTE of CK and VM9 in the present study was $7.19 \times 10^{-6}/^{\circ}\text{C}$, which was higher than that of e.max ($7.08 \times 10^{-6}/^{\circ}\text{C}$).

One-way ANOVA and Tukey's post-hoc test confirmed that the CTE values of CMs significantly differed from those of VCs ($p < 0.05$) as depicted in Table 2.

Table 2 Mean coefficient of thermal expansion values of core materials and veneering ceramics

		Mean CTE values ($10^{-6}/^{\circ}\text{C}$)	SD
Materials			
CMs	Zir	7.86 ^a	0.47
	Cer	7.82 ^a	0.49
VCs			
	CK	7.19 ^b	0.61
	VM9	7.19 ^b	0.29
	e.max	7.08 ^b	0.45

^{a-b} groups with the same superscript letters are not significantly different $p \geq 0.05$

a-b groups with the different superscript letters are significantly different $p < 0.05$

- *Determination of Core-Veneer Shear Bond Strength*

Table 3 shows the mean SBS values of six core-veneer groups. The highest mean SBS value was recorded for Zir-VM9 and Cer-VM9, whereas the lowest was obtained for Cer-e.max and Zir-e.max.

One-way ANOVA test with post-hoc Tukey's test was used to determine the significant differences between the six core-veneer groups as illustrated in Table 3. Statistically significant differences were observed among the Zir-CK, Zir-VM9, and Zir-e.max groups. In addition, statistically significant differences were observed among the Cer-CK, Cer-VM9, and Cer-e.max groups with $p \leq 0.05$. The mean SBS values of Zir/veneer groups were comparable with those of Cer/veneer groups. No significant differences were observed between both groups Fig. 4.

Table 3 Mean shear bond strength of the six core-veneer groups

Core/Veneer	Mean SBS values	SD	
	(MPa)	N	
Zir-CK	93.22 ^{a,d}	8	38.70
Zir-e.max	71.56 ^{b,d}	8	37.46
Zir-VM9	149.48 ^{c,d}	8	67.64
Cer-CK	85.32 ^{a,e}	8	41.41
Cer-e.max	44.07 ^{b,e}	8	16.49
Cer-VM9	123.38 ^{c,e}	8	38.16

a-b-c groups with the same superscript letters are not significantly different $p \leq 0.05$

d-e groups with the same superscript letters are significantly different $p < 0.05$

- *Effect of Coefficient of Thermal Expansion Mismatch on Bond Strength of Core/Veneer Groups (Zir-CK, Zir-e.max, Zir-VM9, Cer-CK, Cer-e.max, and Cer-VM9)*

The mismatch between Zir-CK (0.67) was higher than that between Cer-CK (0.63). CK originated from the same manufacturer and is fabricated specifically for use with Cer. VM9 also behaved akin to CK. The similarity between the chemical compositions of VM9 and CK might explain the likeness between the CTE mismatch values of the VCs and the tested CMs (Table 1). In the case of e.max, its CTE mismatch with Zir (0.78) and Cer (0.74) was higher than that with the other tested veneering porcelains. Large discrepancies might be due to the fact that e.max was originally designed for use with lithium disilicate ceramics and not zirconia.

As illustrated in Fig. 3, the CTE values of CMs showed a slightly positive mismatch (the CTE of CMs was higher than that of overlaying ceramics) with the CTE of each VC. In both groups, the CMs consistently generated higher values than the veneers. The positive CTE discrepancies may explain the SBS results obtained in this study. Literature shows that residual interfacial stresses occur as a result of CTE disparity between two layered materials during the firing and cooling processes of ceramic layering [21,22]. Ideally, a positive CTE deviation is desirable to produce compressive stress on the veneers at room temperature [23,24]. However, adverse tensile stresses will develop on the ceramic interface if a negative CTE discrepancy exists [15]. In the present study, all six core-veneer combinations recorded positive CTE mismatches.

Although statistical analysis showed no substantial correlation between the CTE mismatch and SBS of veneered zirconias, groups with high CTE differences produced low SBS as explained in Table 4. The results of previous studies showed that veneered all-ceramic restorations spontaneously delaminate if CTE dissimilarity exceeds $2.0 \times 10^{-6}/^{\circ}\text{C}$ [15,24,25]. In this study, the mean CTE mismatches of all combinations were within the range of $0.63\text{--}0.78 \times 10^{-6}/^{\circ}\text{C}$ Fig. 5. The results of Alsulami et al. (2015) concur with the present findings. The authors showed no notable changes in the SBS of zirconia with VCs if the CTE is within $0.5\text{--}1.5 \times 10^{-6}/^{\circ}\text{C}$ [26].

Table 4   Effect of coefficient of thermal expansion mismatch on bond strength between core and veneer materials

CMs	Statistical Test	VC		
		CK	e.max	VM9
Zir	Mean SBS value (MPa)	93.22	71.56	149.48
	Mean CTE Differences ($10^{-6}/^{\circ}\text{C}$)	0.672	0.78	0.67
Cer	Mean SBS value (MPa)	85.32	44.07	123.38
	Mean CTE Differences ($10^{-6}/^{\circ}\text{C}$)	0.631	0.74	0.630

4. Conclusion

The mean CTE values of experimental zirconia were similar to those of commercial zirconia and within the limitations of this study. The CTE mismatch between Zir and VC was comparable with that between commercial zirconia and the same VC. The SBS between Zir and VC was affected by their CTE mismatch. The assembly with high CTE mismatch recorded the lowest SBS value. Conversely, the assemblies that registered low CTE mismatches recorded high SBS values. When veneered with the same VC, the SBS results of Zir were identical to those of commercial zirconia. Overall, the results of this study suggest the feasibility of using Zir as an alternative material to commercial zirconia.

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Figures

Figure 1

Fabrication process of sample materials for coefficient of thermal expansion measurement

Figure 2

Mounted shear blade and sample on a universal testing machine (AGS-1000D, Shimadzu® Co., Japan)

Figure 3

CTE values of core materials (Cer and Zir) and veneering ceramics (CK, VM9, and e.max)

Figure 4

Mean shear bond strength values of the six core-veneer groups

Figure 5

Relationship between the coefficient of thermal expansion and shear bond strength of veneered restorative materials