The Effect of Coefficient of Thermal Expansion Differences on Bond Strength of Ceramic-Zirconia Interface

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ABSTRACT

Introduction: In all-ceramic bi-layered restorations, high occurrence of veneering ceramic chipping has been reported in clinical studies. The mismatch of Coefficient of thermal expansion (CTE) is one of the factors that may affect the interfacial residual stress and the veneer-core bond strength. In this study, the effect of CTE mismatch on the shear bond strength (SBS) of 5 veneering ceramics was evaluated. **Materials and Method:** Fifty rectangular zirconia plates (ZR) were divided equally and veneered with 5 different veneering ceramics; Zenoflex Dimension (ZD), Cercon Ceram Kiss (CCK), Vintage ZR 1 (V1), Vintage ZR 2 (V2) and Vintage ZR 3 (V3). SBS was performed according to ISO 10477. Fractured surfaces were examined to determine the fracture mode. **Results:** The mean SBS in MPa (±SD) were 27.61 (±5.56) for ZD, 26.44 (±5.5) for CCK, 30.96 (±7.82) for V1, 37.64 (±10.44) for V2, and 31.32 (±5.6) for V3. V2 had significantly higher mean SBS than ZD and CCK. All groups exhibited either combined or cohesive fracture modes. **Conclusion:** CTE mismatch in the range of 0.5 -1.5 10^{-6} K^{-1} does not have a major effect on the SBS between the veneering ceramic and ZR.
KEYWORDS: CTE mismatch, Fracture mode, Shear bond strength, Veneering Porcelain, Zirconia.

INTRODUCTION

The popularity of all-ceramic dental restorations has increased due to its superior esthetic and biocompatibility compared to metal-ceramic systems. The interest in zirconia as a core material has grown due to its mechano-visual properties, chemical stability and the predictability of production via CAD/CAM technology. Combining the zirconia core material with veneering ceramic can result in a reliable and compatible restoration.[1-3]

However, the failures of dental ceramic bi-layered structures are often multifactorial and can be associated with improper substructural design, thermal-related interfacial residual stresses. Moreover, the presence of critical structural flaws, and non-standardized processing techniques could result in structural failure as well.[4]

The major clinical drawback of zirconia-based restorations is the chipping of veneering porcelain.[5-8]

During manufacturing of bi-layered ceramic restorations, the cooling rate of the ceramic, the mismatch in coefficients of thermal expansion of substructure and the veneering ceramic as well as the thickness of the veneering ceramic are among the major triggers for unfavorable residual stresses.[9-11]

Several studies recommended that the coefficient of thermal expansion (CTE) of veneering ceramic should be lower than that of the substructure. This would result in establishing a strong bond between the core and the veneering ceramics.[4,12,13] Kim et al. reported that, if the CTE mismatch between the veneering ceramic and the zirconia-core material is about 5•10-6 K-1, the tensile stresses on the zirconia could be more than 150 MPa and that may lead to crack growth and spontaneous failure of the restoration.[14] Furthermore, DeHoff et al. concluded that, ceramic systems with CTE mismatch greater than 1.0•10-6 K-1 have a higher tendency to fail in

clinical use compared with systems having a smaller CTE mismatch.[1] In contrast, Fischer et al. investigated the shear bond strength of 12 veneering ceramics to zirconia by using the shear test, and they found that there was no correlation between the CTE mismatch and the shear bond strength.[13]

Therefore, the aims of this study were to compare the shear bond strength of five veneering ceramics with different values of CTE by using shear bond test according to DIN EN ISO 1047715, and to analyze the fracture mode visually and under electron microscope.

MATERIALS AND METHODS

ZIRCONIA SUBSTRUCTURE PREPARATION:
A total of 50 rectangular plates of tetragonal zirconia polycrystal (Y-TZP) were designed, manufactured by CAD/CAM, and provided by the company Wieland (Wieland Dental Technik, Pforzheim, Germany) in pre-sintered white zirconia blank. The specimens were then trimmed from the blocks, and the measurements were prepared by calculating the shrinkage factor to compensate for after sintering shrinkage process. After the final sintering, the specimens had a size of 20x10x1.5 mm respectively. Sintering protocols were performed according to the manufacturers' recommendations (Table 1) in Vita ZYrcomat furnace (Vita Zahnfabrik, Bad Säckingen, Germany). Then, all specimens were cleaned by a steam cleaner and divided equally and randomly into 5 groups.

Parameters	
Starting temperature (°C)	20
Heating time (h)	3:00
Sintering temperature (°C)	1450
Holding time (h)	2:00

Table 1.) Zirconia sintering schedule according to manufacturer's recommendations.

VENEERING CERAMIC PROCEDURES:
Five different veneering ceramics were used in this study. Each has a different CTE value as shown in Table 2.

Company	Ceramic	CTE (10⁻⁶ K⁻¹)	Δσ CTE (10⁻⁶ K⁻¹)	Ceramic Type
Wieland Dental+Technik, Pforzheim, Germany	Zenotec Zr Bridge	10.5	____	zirconia (Y-TZP)
Wieland Dental+Technik, Pforzheim, Germany	Zenoflex dimention (ZD)	9.4	1.1	Leucite reinforced
DeguDent, Hanau-Wolfgang, Germany	Cercon ceram kiss (CCK)	9.2	1.3	Glass ceramic
Shofu JNC , Kyoto, Japan	Vintage Zr 1 (V1)	9.0	1.5	unknown
Shofu JNC , Kyoto, Japan	Vintage Zr 2 (V2)	9.5	1.0	unknown
Shofu JNC , Kyoto, Japan	Vintage Zr 3 (V3)	10.0	0.5	unknown

Table 2.) CTE values of zirconia and veneering ceramics used in this study at 25-500°C.

The preparation process of the veneering layer was done through 5 steps: 1st and 2nd layers of the liner then, 1st and 2nd layers of dentin and finally, glazing with a firing cycle after each layer. The firing protocol for each step was carried out in a dental furnace Austromat 624 (DEKEMA, Freilassing, Germany) according to the manufacturer's recommendations (Table 3). For each group, a thin layer of the respective liner was applied to the zirconia plates and fired according to the manufacturer's recommendations. The second layer of the liner was applied and fired under the same condition to achieve a continuous thin layer of the liner. For the dentin layer, a stainless steel mold was placed on the zirconia plates where clearance of 2.5 mm height and 5 mm diameter was available above the zirconia plates. The inner surface of the mold was isolated with isolating fluid (Carat, Hager & Werken, Duisburg, Germany) to avoid the adhesion of the ceramic powder to the mold surface during dentin layering process. The veneering ceramic powder was mixed with an appropriate amount of respective liquid as in the common dental lab. The slurry mixture was then applied onto the liner layer through the mold (Figure 1 (a)), and the excess liquid was

Veneering ceramic	Temperature (°C)	Time (min)	Heating Rate (°C/min)	Firing Temp. (°C)	Holding Time (min)
1st/ 2nd Liner					
ZD	575	2	45	930	1
CCK	575	6	55	970/960	1
V 1,2,3	500	7:30	45	930	1
1st Dentin					
ZD	575	3	45	900	2
CCK	450	2	55	830	1:30
V 1,2,3	650	5:30	45	910	1
2nd Dentin					
ZD	575	2	45	890	1
CCK	450	2	55	820	1:30
V1,2,3	650	5:30	45	900	1
Glazing					
ZD	575	1	45	880	1
CCK	450	0	55	800	1
V1,2,3	600	6	55	860	0

Table 3.) Veneering ceramics firing schedule according to manufacturer's recommendations. Zenoflex Dimension Ceramic: ZD, Cercon Ceram Kiss Ceramic: CCK, Vintage ZR1 ceramic : V1, Vintage ZR2 ceramic : V2, Vintage ZR3 ceramic: V3

Figure 1.) (a) Veneering ceramic application.

absorbed with tissue paper. After applying the dentin layer, the mold was carefully removed, and the specimens were then fired according to the manufacturer's recommendations (Table 3). Under the same conditions, a second dentin layer was added and fired to compensate the porcelain shrinkage and to establish the correct diameter and thickness of the specimens. Finally, a glaze-layer firing was performed (Figure 1 (b)) according to the manufacturer's recommendations as well (Table 3).
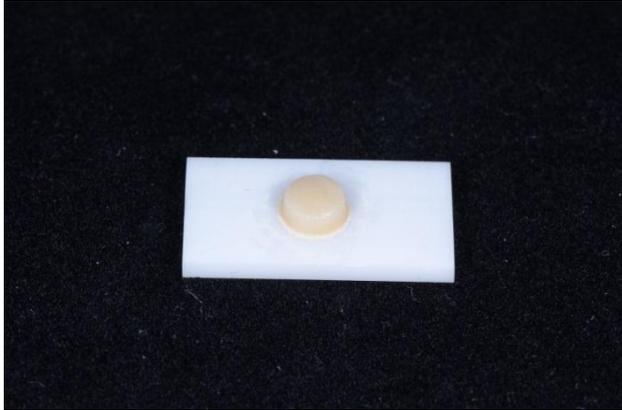

Figure 1.) (b) Final shape of the specimen before testing.

MEASURING THE VENEERED AREA: Prior to the shear bond strength test (SBS) the area to be veneered of all specimens were examined with Photomacroscope M400 (Wild Heerbrugg, Gais, Switzerland), and measured in (mm^2) using software Image Pro Plus (Media Cybernetics, Washington, USA).

After the SBS test, the specimens were examined, and the area of remaining veneering porcelain was measured in (mm^2) to determine the fracture mode.

SHEAR BOND STRENGTH: The completed specimens were attached to a special sample holder and placed in a universal testing machine Z010 (Zwick, Ulm, Germany). The shear force was applied to specimens with a distance of 0.5 mm to the interface between the veneering ceramic and the zirconia. The shear force was applied with a crosshead speed of 1 mm/min until the fracture occurred. The forces at fracture were recorded in Newton (N). For each group, the mean bond strength (MPa) was calculated by dividing the force at fracture (N) by the bonding area (mm^2).

DETERMINING THE FRACTURE MODE: After measuring the remaining veneering ceramic area, the percentage of the remaining veneering ceramic was calculated and every specimen was classified, according to the percentage of the remaining veneering ceramic, into one of the following fracture modes:

- Less than 20% → adhesive fracture within the veneering ceramic.
- Between 20% and 80% → mixed fracture.
- More than 80% → cohesive fracture.

Analysis of the fractured area under scanning electron microscope (SEM)

After calculating the bond strength of all the specimens in each group, 3 specimens were selected from each group. The main selection criterion was the bond strength value of the specimens. The specimens with the highest, middle, and lowest bond strength value in each group, were inspected under SEM (Leo 1430, Zeiss, Oberkochen, Germany) at 30x, 100x, and 1000x magnification, and imaged to analyze the fractured surface.

STATISTICAL ANALYSIS: Statistical analysis was done using JMP software (SAS Institute Inc. NC, USA). The data were analyzed using one-way analysis of variance test (ANOVA) to determine the differences between the shear bond strengths of the 5 groups at ($p<0.05$). A Tukey multiple comparisons test at ($p<0.05$) was used to assess the differences among the groups.

RESULTS

The mean shear bond strengths of all 5 veneering ceramics are presented in Table 4 and graphically in Figure 2.

Veneering Ceramic	SBS mean (MPa)	SD	Sign.*	N
ZD	27.61	± 5.56	B	10
CCK	26.44	± 5.5	B	10
V1	30.96	± 7.82	A/B	10
V2	37.64	± 10.44	A	10
V3	31.32	± 5.6	A/B	10

Table 4.) Mean shear bond strength, standard deviation(SD), and significance of all tested veneering ceramics * Groups with the same letter are not statistically different using Tukey test at P<0.05). Zenoflex Dimension Ceramic: ZD, Cercon Ceram Kiss Ceramic : CCK, Vintage ZR1 ceramic : V1, Vintage ZR2 ceramic : V2, Vintage ZR3 ceramic : V3
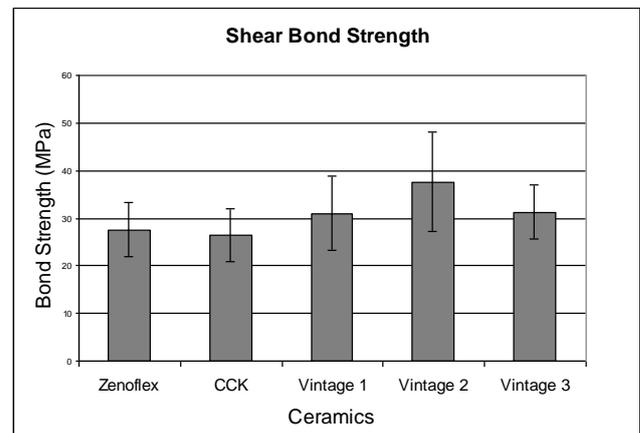

Figure 2.) Mean shear bond strength of the 5 tested veneering ceramics (n=10). Error bar shows the standard deviation (SD) (p<0.05).

The highest mean shear bond strength was recorded for V2 (37.6±10.4 MPa) followed by V3 (31.3±5.6 MPa). The mean shear bond strength of V1 and ZD were (31.0±7.8 MPa) and (27.6±5.6 MPa) respectively. The lowest mean shear bond strength was recorded for CCK (26.4±5.5 MPa).

One-way ANOVA showed a significant difference in the shear bond strength among the five tested

veneering ceramics (p<0.05). The Tukey HSD comparison test was used to make all pair comparison of the mean shear bond strength of the 5 groups. The results of this comparison are presented in Table 4. The P values of the different comparison showed that ZD, CCK, V1, and V3 had no statistically significant difference. V2 had a significantly higher mean bond strength compared to ZD and CCK groups.

All groups demonstrated either cohesive or mixed fracture mode within the veneering ceramic. In V1 and V2 groups, 9 specimens from each group demonstrated a mixed failure mode while one specimen failed cohesively from each group. In V3 and CCK groups, 6 specimens from each group demonstrated mixed fracture mode and 4 specimens failed cohesively. In ZD group, 8 specimens exhibited mixed fracture mode, and 2 specimens failed cohesively (Table 5).

Ceramic Group	Cohesive	Mixed	Adhesive
ZD	2	8	0
CCK	4	6	0
V1	1	9	0
V2	1	9	0
V3	4	6	0

Table 5.) Fracture mode incidence of all tested veneering ceramics. Zenoflex Dimension Ceramic: ZD, Cercon Ceram Kiss Ceramic : CCK, Vintage ZR1 ceramic : V1, Vintage ZR2 ceramic : V2, Vintage ZR3 ceramic : V3
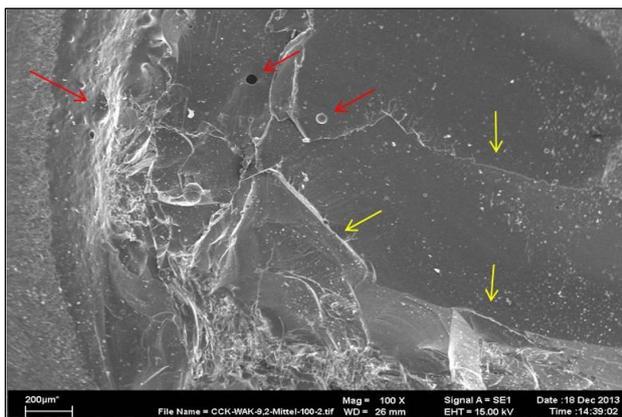

Figure 3.) (100 magnification) SEM image of a fractured surface, yellow arrows point at the fracture lines, red arrows point at defects in the veneering ceramic. (CCK group).
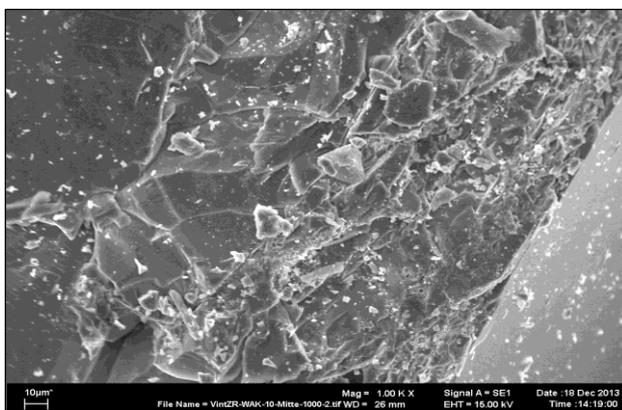

Figure 4.) (1000 magnification) SEM image shows irregular fracture lines. (V3 group).

Examination of SEM images of different specimens revealed several inherent defects (porosity) (Figure 3) irregular crack lines (Figure 4) and detached veneering ceramic crystalline (Figure 5).
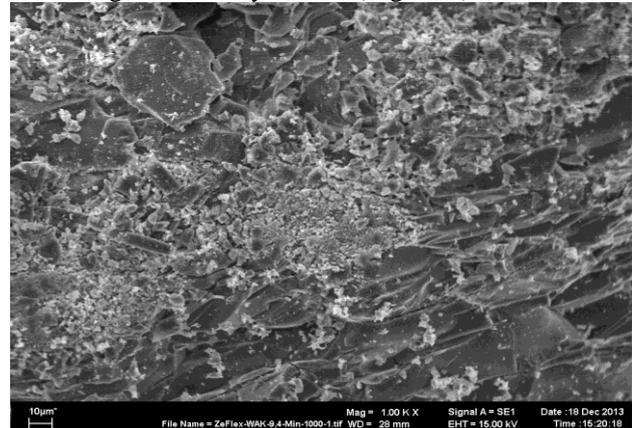

Figure 5.) (1000 magnification) SEM image of fracture surface shows detached crystalline of the veneering ceramic. (ZD group).

DISCUSSION

The present study investigated the effect of CTE mismatch between the veneering ceramic and zirconia substrate.

Currently for all ceramic bi-layered systems, there is no standardized test for bond strength measurement. Several bond strength test methods are used to evaluate the bond strength between core and veneering ceramics in all ceramic systems[16-20]. Each test has advantages and disadvantages. Ease of specimens' preparation, clear protocol, and rapid results collection are the main advantages of shear bond strength test. The main disadvantages include high standard deviation, occurrence of non-uniform interfacial stresses, and the influence of specimen geometry.[21,22] Therefore, the standardization of SBS methodology is important for improving the clinical relevance. Standardization of the methodology should include storage conditions, type of substrate, specimen preparation, the rate of load application, cross-sectional surface area, and experience of the researcher.[20,22,23] In the present study, the shear bond strength test was prepared and performed according to the ISO 10477 standards of the SBS for metal-resin.[15]

In the present study, the bond strength of 5 veneering ceramics with different CTE values were tested. Generally, all the 5 veneering ceramics exhibited mean bond strength in the range of 26.4 - 37.6 MPa, which is consistent with most of the previous shear bond strength studies.[2,20,24-27] The mean bond strength of CCK obtained from this study is 26.4 MPa. This value is higher than that reported by Özkurt et al. and Saito et al. which were 20.19 MPa, and 22.0 MPa respectively.[21,25] The difference between the present study and the other two studies could be explained by the effect of thermocycling. According to most bond strength studies, the bond strength would decrease

further with thermocycling.[22] In the present study the specimens were tested in a dry environment, while in Özkurt et al. study and Saito et al. study they stored all the specimens in distilled water at 37 °C for 24 hours prior to testing.[21,25] In a study by Aboushelib et al.[28], CCK exhibited mean bond strength of 36.6 MPa, which was higher than the 26.4 MPa obtained in the present study. Two reasons could explain the high mean bond strength obtained in Aboushelib et al. study. First, they used a different bond strength test, second; they used double veneering ceramic layer, one with press-on technique and the second with layering technique.[28] No study could be found to evaluate the bond strength of ZD or the experimental veneering ceramics V1, V2, and V3. However, the three experimental veneering ceramics showed higher mean bond strength than ZD and CCK veneering ceramics. The CTE mismatch of V2, ZD, and CCK are 1.0, 1.1, 1.3·10^{-6} K^{-1} respectively. Although the difference in CTE mismatch of those veneering ceramics was not significantly different, the mean bond strength of the V2 showed significantly higher mean bond strength than ZD and CCK veneering ceramics. Therefore, we can state that the CTE mismatch in the range of 0.5 -1.5·10^{-6} K^{-1} does not have a major effect on the SBS between the veneering ceramic and zirconia substrate which is consistent with the other studies.[13,25,29] The other properties of the veneering ceramic, such as modulus of elasticity and Poisson's ratio, may have an effect on the bond strength. It should be considered that the CTE of ceramics is nonlinear, so, the best way to describe the thermal behavior of ceramics is a combination of quadratic relation, linear relation and by ISO standard.[30]

The specimens mainly showed a combined fracture mode as adhesive at the interface and cohesive within the veneering ceramics (Table 5). Combined fracture mode was seen in 66% of the specimens, and these findings are consistent with previous studies.[2,20,21,29] Such findings could be explained by the deflection of the crack due to the superior resistance of zirconia to crack propagation. Moreover, these combined fracture modes could be a result of poor bonding between veneering ceramic and zirconia substrate.[29] In addition, cohesive fracture mode was seen in 34% of the specimens. In those specimens, cohesive fracture mode indicated that the bond strength between the veneering ceramic and core substrate is higher than the cohesive strength of the veneering ceramic itself.[13]

SEM evaluation revealed that the fracture is originated in the veneering ceramic layer through multiple porosities. Irregular fracture lines indicated that the fracture was caused by the porosities within the veneering ceramic that acted as stress stem points. Aboshelib et al. found porosities-free veneer after SEM examination of zirconia press-on veneer interface. They found that the mean bond strength of CCK is 36.6 MPa, which is higher than that obtained in the present study.[31] Therefore, veneering technique may play an integral role in the presence of pores within veneering ceramic and affect the mean bond strength of tested specimens.

CONCLUSION

Within the limitations of this in vitro study, it can be concluded that CTE mismatch in the range of 0.5 -1.5•10-6 K-1 does not have a major effect on the bond strength between the ceramic and zirconia substrate. The experimental veneering ceramics used (Vintage ZR 1, 2, 3), revealed higher bond strength than Zenoflex and Cercon Ceram Kiss veneering ceramics. Surface analysis of the fractured structures revealed mainly combined, and cohesive fracture mode but no adhesive fracture were observed.

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