

SHEAR BOND STRENGTHS BETWEEN CERAMIC CORES AND VENEERING CERAMICS OF DENTAL BI-LAYERED CERAMIC SYSTEMS AND THE SENSITIVITY TO THERMOCYCLING

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The purpose of this study was to investigate the bond strength between various commercial ceramic core materials and veneering ceramics of dental bi-layered ceramic combinations and the effect of thermocycling. The shear bond strength of four dental bi-layered ceramic combinations (white Cercon, yellow Cercon, white Lava, yellow Lava, IPS E.max) were tested. Metal ceramic combinations were conducted as a control group. Half of each group was subjected to thermocycling. All specimens were thereafter subjected to a shear force.

The initial mean shear bond strength values in MPa \pm S.D were 28.02 \pm 3.04 for White Cercon Base/Cercon Ceram Kiss, 27.54 \pm 2.20 for Yellow Cercon Base/Cercon Ceram Kiss, 28.43 \pm 2.13 for White Lava Frame/Lava Ceram, 27.36 \pm 2.25 for Yellow Lava Frame/Lava Ceram, 47.10 \pm 3.77 for IPS E.max Press/IPS E.max Ceram and 30.11 \pm 2.15 for metal ceramic control. The highest shear strength was recorded for IPS E.max Press/IPS E.max Ceram before and after thermocycling. The mean shear bond strength values of five other combinations were not significantly different ($P < 0.05$). Lithium-disilicate based combinations produced the highest core-veneer bonds that overwhelmed the metal ceramic combinations. Thermocycling had no effect on the core-veneer bonds. The core-veneer bonds of zirconia based combinations were not weakened by the addition of coloring pigments.

INTRODUCTION

An ideal all ceramic restoration is expected to exhibit superior aesthetics, like translucency, natural tooth color, outstanding light transmission, at the same time, optimal mechanical properties, like flexural strength, fracture toughness and limited crack propagation [1]. Porcelain fused to metal technique has been a golden standard for fixed restorations since 1960s [2,3]. The technical procedures in producing metal frameworks were highly technique-sensitive. Moreover, the layer of opaque is likely to affect the translucency resulting in limitation for the aesthetics [4]. Furthermore, metal allergy was reported [5, 6].

Metal ceramic restorations have been increasingly replaced by all ceramic restorations in recent years because of their superior aesthetics, inertness and biocompatibility [7]. However, the inherent brittleness of all-ceramic systems may lead to premature failure, especially under repeated contact loading in moist environments [8]. Yttrium oxide partially stabilized tetragonal zirconia polycrystal (Y-TZP) framework manufactured through CAD/CAM process are of desirable mechanical properties such as chemical and dimensional stability, high mechanical strength, and fracture-toughness [9]. Some recent studies showed

that zirconia offered sufficient stability as a framework material [10-13]. Y-TZP can form the framework for multi-unit posterior fixed partial dentures (FPDs) [9, 14, 15]. Zirconia based restorations have been assessed in numerous clinical studies. In previous studies that the success rates of zirconia based restorations were 100 % [16] after 28 months, 100 % [17] after 36 months, and 97.8 % [18] after 60 months that showed the high stability of zirconia frameworks. In another clinical study Y-TZP crowns even showed comparable fatigue life to metal ceramic crowns [19].

Failures have also been reported for Y-TZP frameworks because of secondary caries, fracture of the framework and chipping of the veneering ceramic [13, 20, 21]. For FPDs fabricated with Y-TZP frameworks the chipping rates of the veneering porcelain was found in 15 % [22] after 24 months and 6 % [23] after 36 months. However, clinical studies on metal ceramic restorations indicated substantially lower chipping rates 0 % after 3 years [24], 2.5 % after 5 years [25] and between 5 % and 10 % over 10 years [26]. Moreover, Aboushelib [27] reported the addition of coloring pigments to zirconia frameworks resulted in structural changes that significantly decreased the microtensile bond strength of frameworks and veneering ceramics.

The IPS E.max Press castable glass ceramic, composed mainly of a modified lithium disilicate, was introduced recently. Lithium disilicate frameworks are recommended to apply a single crown. At present, there is a little clinical performance data available to confirm the application of frameworks of FPDs. Medium [24] and long-term [12] clinical studies of short-span lithium disilicate based FDPs in the anterior and posterior segments were reported in a prospective. One clinical study [11] even showed a up to 100 % success rate for 3-unit lithium disilicate based FDPs after 4 years observation period. It was reported the survival rates for inlay-retained lithium-disilicate based FDPs were 57 % after 5 years and 38 % after 8 years, while for hybrid-retained FDPs was 100 % after 5 and 60 % after 8 years [28]. An in vitro study showed that fracture resistance of lithium disilicate based crowns for molars was comparable with that of natural unprepared teeth [10]. The fracture rates of the veneering porcelain of lithium disilicate based restorations were found in 3.3 % after 3 years [24] and 6 % after 8 years [12] which were comparable to the golden standard.

The before-mentioned studies showed the application of framework materials like zirconia and lithium disilicate are prospective. While, chipping and delamination of the veneering ceramic was reported as the most frequent reason for failures of zirconia and lithium disilicate based restorations [13, 20, 24].

The aim of present study was to evaluate the core-veneer bond strength of bi-layered all-ceramic systems and compare to the golden standard. The effect of thermocycling on core-veneer bond was also investigated.

EXPERIMENTAL

The properties and manufacturers of core materials tested and their respective veneering ceramics are listed in Table 1. A hundred and twenty commercial core ceramics (white Cercon Base, yellow Cercon Base, white Lava Frame, yellow Lava Frame, IPS E.max Press) and their respective veneering ceramics (Cercon Ceram Kiss, Lava Ceram, IPS e.max Ceram) were fabricated and divided into four groups containing 30 specimens each. Cobalt chromium alloy metal ceramic specimens (Wirobond 280, Ceramco3) were served as a control group (n = 30).

Specimen preparation

White and yellow Cercon Base, Lava Frame blanks were milled in Cercon brain unit (Densply, Hanau, Hesse-Darmstadt, Germany) then sintered in Cercon heat furnace (Densply, Hanau, Hesse-Darmstadt, Germany). White and yellow Lava Frame blanks were milled in Lava CNC 500 (3M ESPE, AG, Seefeld, Bavaria, Germany) thereafter sintered in Lava Furnace (3M ESPE, AG, Seefeld, Bavaria, Germany). IPS E.max blanks were hot pressed and sintered to full density in the furnace (Ivoclar Vivadent AG, Schaan, Liechtenstein). Wirobond 280 cobalt chromium alloy were cast in a vacuum pressure casting machine (Nautilus T, Bego, Bremen, Freie Hansestadt Bremen, Germany). The framework specimens of each group were made into microbars (5 × 5.4 × 13 mm) using a

Table 1. The properties and manufacturers of the core materials and their respective veneering ceramics.

| | Material | Manufacturer | Main components (mass %) | CTE (10 ⁻⁶ k ⁻¹) |
|----------------------|--|--|--|---|
| Frame work materials | White Cercon Base | Densply, Hanau, Hesse-Darmstadt, Germany | ZrO ₂ (HfO ₂) = 95(<2HfO ₂); Y ₂ O ₃ = 5; Al ₂ O ₃ + other oxides <1 (+SiO ₂) | 10.5 |
| | Yellow Cercon Base | | | 10.5 |
| | White Lava Frame | 3M ESPE, AG, Seefeld, Bavaria, Germany | | 10.0 |
| | Yellow Lava Frame | | | 10.0 |
| | IPS E.max Press | Ivoclar Vivadent AG, Schaan, Liechtenstein | | SiO ₂ , Li ₂ O, K ₂ O, P ₂ O ₅ , ZrO ₂ , ZnO, Al ₂ O ₃ , MgO, La ₂ O, pigments >57 |
| Bego Wirobond 280 | Bego, Bremen, Freie Hansestadt Bremen, Germany | Co 60.2, Cr 25, W 6.2, Mo 4.8, Ga 2.9, other <1 (Si, Mn) | 14.0-14.2 | |
| Veneering ceramics | Cercon Ceram Kiss | DeguDent, Hanau, Hesse-Darmstadt, Germany | SiO ₂ 60.0-70.0; Al ₂ O ₃ 7.5-12.5; K ₂ O 7.5-12.5; Na ₂ O 7.5-12.5 | 9.2 |
| | Densply Ceramco3 | Densply, Burlington, USA | Sodium potassium aluminosilicate: 80-100, thin oxide 0-20 | 12.8-13.9 |
| | IPS E.max Ceram | Ivoclar Vivadent AG, Schaan, Liechtenstein | SiO ₂ 5 0-60; Al ₂ O ₃ 16-22; K ₂ O ₄ 8; Na ₂ O ₆ 11; CaO, P ₂ O ₅ and F: 2.0-6.0; other oxides: 1.5-8, pigments: 0.1-3 | 9.5 |
| | Lava Ceram | 3M, ESPE, AG, Seefeld, Bavaria, Germany | | 10.0 |

metal mold following the Schmitz-Schulmeyer method [29] (Figure 1). The sintered specimens were sandblasted thereafter ultrasonically and steam cleaned. For white and yellow Cercon Base, Lava Frame, a layer of liner was applied, for the IPS E.max Press, a thin wash layer of the veneering porcelain was applied, for the metal cores a layer of liner and a second layer of opaque was applied. The veneering ceramics were added to the core specimens and built up to a final dimension ($4 \times 5.4 \times 3$ mm) according to the Schmitz-Schulmeyer method (Figure 1). The powder of each veneering ceramic was mixed with the corresponding manufacturer's liquid and the obtained slurry was plotted with tissue to draw excess water. The core veneer specimens were then fired to full density. Subsequently glaze-firing was applied to all specimens. Each step was applied according to each the manufacturers' recommendations.

Thermocycling

Prior to shear bond testing, half of each group ($n = 15$) was subjected to thermocycling ($20\ 000\times$) in water ($5/55^\circ\text{C}$) with a transfer time of 2 s (DEYI, Enterprise, Xiamen, Fujian, China). All specimens underwent thermocycling were kept in deionized water at room temperature. At the same time the remaining specimens ($n = 15$) were stored dry at room temperature.

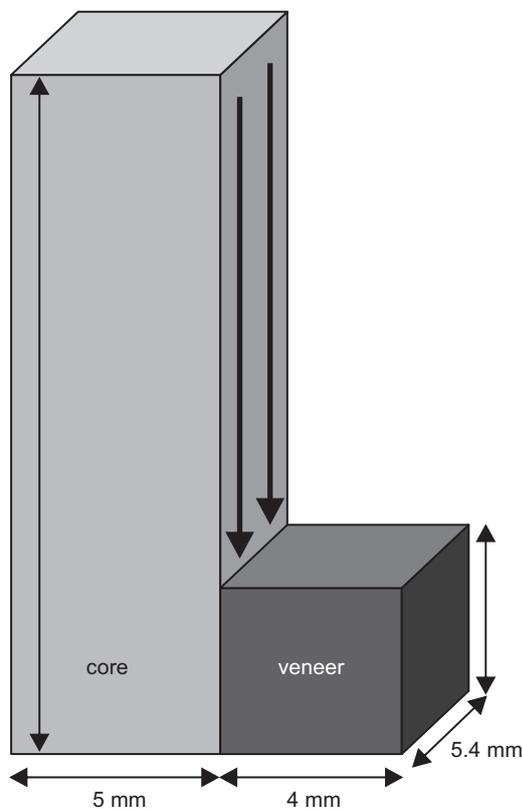


Figure 1. Design and dimension of Schmitz-Schulmeyer specimens. Arrows indicate load application during shear bond strength test.

Shear bond strength test

Each specimen was mounted in a metal holder on the universal testing machine (AG-IC, Shimadzu, Kyoto, Kyoutofu, Japan). Load was applied parallel to the long axis of the specimen through a wedge at the core-veneer interface at a crosshead speed of 1 mm/min until failure (Figure 1). The ultimate load to failure was recorded by the system's software (Trapezium X, Shimadzu, Kyoto, Kyoutofu, Japan). Average shear bond strengths [MPa] were calculated by dividing the failure load [N] by the bonding area [mm^2]. Shear stress (MPa) = load (N)/Area (mm^2)

Scanning electron microscopy (SEM)

The fractured surfaces were visually analyzed with a microscope (LSM 700, Carl Zeiss, Jena, Germany) at original magnification $20\times$. The surface with remaining veneering ceramic was measured by a software (AnalySIS 3.0 Soft Imaging System, Münster, North Rhine-Westphalia, Germany) and divided by the total bonded area to determine the failure mode in percentage. This was done by tracing the borders of the cohesive veneer/core fracture that remained within the bonded interface. The fracture patterns were classified as cohesive in the veneer (V), adhesive at the core-veneer interface (C/V), and cohesive in the core (C). A part of the selected fractured surfaces were ultrasonically cleaned and gold sputter-coated for SEM examination (SSX-550, SHIMADZU, Kyoto, Kyoutofu, Japan). The distribution of the elements remained on the fractured surface of the frameworks were investigated using EDS (SSX-550, SHIMADZU, Kyoto, Kyoutofu, Japan).

Statistical analysis

The SBS was statistically analyzed by the SPSS 13.0 Program (SPSS Inc. Chicago Illinois, USA program). The data was analyzed by One-way ANOVA to assess the group effect ($\alpha = 0.05$). Also, a Tukey post hoc test ($\alpha = 0.05$) was used for testing the differences among the specified materials.

RESULTS

Table 2 demonstrates the mean shear strength values between the core and the veneer of four all-ceramic test groups and the metal ceramic control group before and after thermocycling. The highest mean shear strength was recorded for IPS E.max Press bonded to IPS E.max Ceram before and after thermocycling. There was a significant difference for the shear strengths among different groups at $P < 0.05$ irrespective of thermocycling (Table 3, 4). The effect of thermocycling on the shear bond strength was not statistically significant ($P < 0.05$).

The mean shear bond strength of IPS E.max Press bonded to IPS E.max Ceram was statistically significantly higher than other all-ceramic groups and the metal ceramic group ($P < 0.05$). The mean values of white Cercon, colored Cercon test groups and the metal ceramic control group were not significantly different ($P < 0.05$). IPS E.max Press/IPS E.max Ceram showed predominantly cohesive fractures in the core and the veneering ceramic (Table 5, Figure 2a). The main failure mode for white Cercon Base /Cercon Ceram Kiss, yellow Cercon Base /Cercon Ceram Kiss, White Lava Frame/Lava Ceram, yellow Lava Frame/Lava Ceram and the metal ceramic control group was cohesive at the core-veneer interface (Table 5, Figure 2b).

DISCUSSION

A bond strength of 25 MPa was accepted as the minimum for metal ceramic systems [30]. A minimum required bond strength for all-ceramic multilayered systems has not been established [7]. Bond strength measurement of all-ceramic restorations has not been standardized. The shear bond strength (SBS) test was used most frequently in studies and reported to be relatively simple and easily performed [7, 31]. The Schmitz-Schulmeyer test has been proved to be a reliable test for metal ceramic bond strength measurements with minimal experimental variables [29]. In a recent study, the Schmitz-Schulmeyer test was reported to be a applicative test for measurement of the core-veneer bond strength rather than the mechanical properties of the veneering ceramic [32, 33, 34]. Shear bond strength test has not been standardized. Various factors might have effect on the result of shear bond strength test, such as geometry shape of specimens, type of substrates, storage conditions and cross-head speed [35, 36]. The ISO standards recommend that the rate of loading for a bonded specimen should be $0.75 (\pm 0.30)$ mm/min [37]. A crosshead speed of 1.0 mm/min was employed to evaluate shear bond strengths in several studies [7, 38, 39].

The data (Table 2) showed that IPS E.max Ceram applied to IPS E.max Press produced the highest values

of SBS and were significantly different from other bi-layered systems before and after thermocycling. This might be attributed to good micromechanical interlocking or chemical bond between the core and the veneering ceramic. The result was in agreement with findings of some previous studies [31, 35] that the shear bond strength of lithium disilicate framework to the corresponding veneering ceramic was significantly higher than metal and zirconia frameworks to their frameworks. However, compared with the previous studies the different shear bond strength values of the current study could be attributed to the difference in methodologies. The core-veneer interface of lithium disilicate glass based combinations presented with higher shear strength with press technique than layered technique [36]. The higher bond strength can be explained by compression of the veneer over the core during cooling in fabrication. Aboushelib [27] found the addition of coloring pigments, resulted in structural changes, reduced the bond strength of zirconia frameworks and the corresponding veneering porcelains. The data (Table 2) demonstrated the core-veneer bond strength of white zirconia based combinations and yellow zirconia based combinations were not significantly different indicating the addition

Table 3. One-way ANOVA of shear strength of five all-ceramic groups and the control group before thermocycling (TC).

| Source | Sum of squares | df | Mean square | F | P |
|-----------|----------------|----|-------------|--------|-------|
| Materials | 4497.48 | 5 | 899.50 | 126.80 | 0.000 |
| Error | 595.90 | 84 | 7.09 | – | – |

*Significant at 95 % CI

Table 4. One-way ANOVA of shear strength of five all-ceramic groups and the control group after thermocycling (TC).

| Source | Sum of squares | df | Mean square | F | P |
|-----------|----------------|----|-------------|--------|-------|
| Materials | 3928.41 | 5 | 785.68 | 109.67 | 0.000 |
| Error | 601.76 | 84 | 7.16 | – | – |

*Significant at 95 % CI

Table 2. Mean shear strengths (MPa) of five all-ceramic groups and the control group before and after thermocycling (TC).

| Group | SBS [Mpa] mean (dry) | Statistical category (dry)* | SBS [Mpa] mean (TC) | Statistical category (TC)* | Comparison dry/TC P-value |
|---------------|----------------------|-----------------------------|---------------------|----------------------------|---------------------------|
| White Cercon | 28.02 ± 3.04 | a,b | 27.71 ± 3.21 | a,b | 0.79 |
| Yellow Cercon | 27.54 ± 2.20 | a,c | 27.15 ± 2.39 | a,c | 0.64 |
| White Lava | 28.43 ± 2.13 | a,d | 27.17 ± 2.60 | a,d | 0.58 |
| Yellow Lava | 27.36 ± 2.25 | a,e | 28.85 ± 2.00 | a,e | 0.83 |
| IPS E.max | 47.10 ± 3.77 | g,h | 45.68 ± 3.40 | g,h | 0.29 |
| Control group | 30.11 ± 2.15 | a,f | 29.80 ± 2.14 | a,f | 0.71 |

*Different uppercase letters indicate statistical difference ($P < 0.05$).

of coloring pigments had no effect on the core-veneer bond strength of zirconia based combinations. EDX analysis of the fractured framework surface showed minor chemical differences between white zirconia framework and yellow zirconia framework were found [27]. Table 2 demonstrated the mean bond strength of the control group were higher than recommended and not significantly different from zirconia based all-ceramic combinations that was consistent with the results of the previous studies [31, 35]. MC could not attain the high bond strength of IPS E.max Press/IPS E.max Ceram that might be attributed to the shrink of the thin layered opaque after firing.

Table 5. Failed bonded surfaces divided by percentage: (V) cohesive within veneering ceramic, (C/V) combined surface failure, (C) cohesive within core.

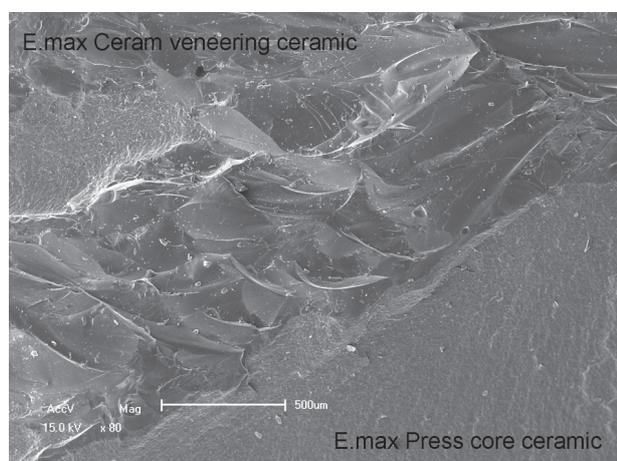
| Material group | V | V/C | C |
|-------------------------------------|------|------|------|
| White Cercon Base/Cecon Ceram Kiss | 34.2 | 65.8 | 0 |
| Yellow Cercon Base/Cecon Ceram Kiss | 34.5 | 66.5 | 0 |
| White Lava Frame/Lava Ceram | 38.4 | 61.6 | 0 |
| Yellow Lava Frame/Lava Ceram | 37.9 | 62.1 | 0 |
| IPS E.max Press/IPS E.max Ceram | 19.5 | 0 | 80.5 |
| MC (control) | 39.1 | 60.9 | 0 |

In assessment of the findings in this study, failure mode should also be taken into consideration. Table 5 illustrated the fracture modes were cohesive both in the core and the veneering ceramic for IPS E.max Press/IPS E.max Ceram. No adhesive fracture at the interface was observed. This could be interpreted as a good bond of the core-veneer interface of IPS E.max combinations which result in higher SBS. The flexural strength of IPS E.max Press framework is much lower than zirconia and metal frameworks according to the data provided by

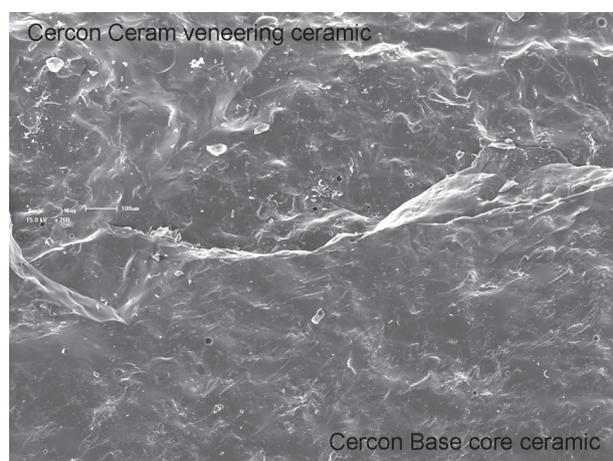
the manufacturers. The data showed failures occurred primarily at the core-veneer interface for the veneering ceramic bonded to zirconia framework that was comparable to the results of other laboratory studies [32, 33, 34]. This could be interpreted as the superior ability of Y-TZP framework on resistance of crack propagation. While, the interlaminar crack deflection could also result from the relatively low bond strength of the veneering ceramic to zirconia framework. Metal ceramic group showed failure mainly occurred at the interface (Table 5). However, the predominantly failure mode of metal ceramic group were reported to be cohesive in the veneering ceramic in the previous studies [18, 31]. EDS analysis showed the elements of the opaque residue were traced on the core surface indicating fractures occurred between the opaque and the metal core.

Figure 2a showed a combined failure mode: cohesive in the core material and adhesive at the core-veneer interface (IPS E.max Press/IPS E.max Ceram). Figure 2b also showed a combined failure mode: cohesive in the veneering ceramic and adhesive at the core-veneer interface (white Cercon Base /Cercon Ceram Kiss). A combined failure mode: cohesive in the veneering ceramic and adhesive at the core-veneer interface was observed in Figure 3 (MC). Porosities could be found in both the core material and the veneering ceramic that might weaken the interfacial bond (Figures 2, 3). The porosities and micro-gap formations are related to technical skills. The imperfections of the veneering ceramic and core material might be one of the impacts for the high chipping rate reported in the clinical studies [40].

The impact of the CTE mismatch on bond strength between the veneering porcelain and the core material has been widely discussed. Exposure to direct contact with saliva and cyclic loading lead to degradation of ceramic materials where slow crack originated and spread that resulted in failure of ceramic restorations in oral cavity



a) IPS E.max Press/IPS E.max Ceram



b) white Cercon Base/Cercon Ceram Kiss

Figure 2. A combined failure mode: cohesive in a) the core material and adhesive at the core-veneer interface, b) the veneering ceramic and adhesive at the core-veneer interface.

[41, 42]. The bond strength can be weakened by residual stresses as a result of veneer and core CTE mismatch [36]. Ceramic cores and veneering ceramics with similar CTE are recommended that generate compressive stresses in the weaker veneering ceramics, therefore reinforcing the overall strength of the restorations. The shear bond strengths of five tested ceramic composites showed no difference within the CTE mismatch of 0 to $1.3 \times 10^{-6}/^{\circ}\text{C}$ which was in accordance with the finding of Guess et al [19]. Mackert [43] reported the CTE of veneering ceramic was nonlinear and varied, related to the temperature interval, the time of heat soak at peak firing changes resulting from thermal history. On contrary, other studies [14, 38] demonstrated strong CTE mismatch between the veneering ceramics significantly affect the shear bond strength.

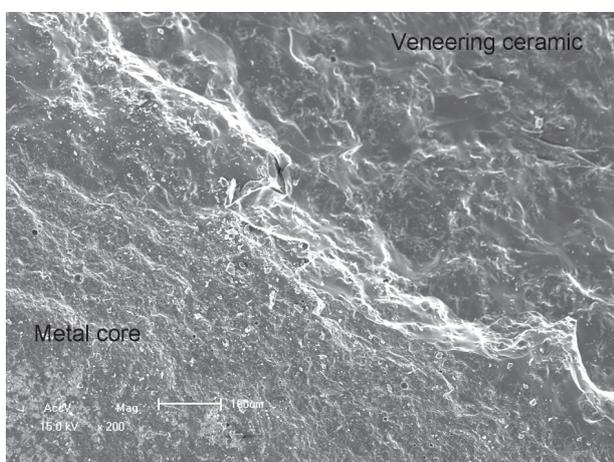


Figure 3. A combined failure mode: cohesive in the veneering ceramic and adhesive at the core-veneer interface (MC).

Some studies proposed the application of thermal cycling induces repeated load on the cover-veneer interface that result in low bond strength between the core and the veneering ceramic [44]. The aging sensitivity on the shear bond strength of all-ceramic bi-layered systems was thereby tested by exposure to a standardized thermocycling test where the sample was moved between high and low temperature surroundings for a predetermined number of cycles [22]. The predetermined temperatures ranging from 5-55°C was proposed in ISO 11405 recommendations (ISO, 1994). Moreover, a dwell time of 2 s and the application of 20,000 cycles at either peak temperature were also proposed. The average number of thermal cycles would normally occur in the oral cavity that was estimated approximately 4000 [45] to 10,000 [46] per year. In the current study the application of 20,000 cycles of thermocycling had no effect on the shear bond strength of all test groups that was in consistency with findings of Guess et al. [19].

The bi-layered all-ceramic combinations investigated in this study do not represent the shape conditions of dental restorations clinically, but provide a geometry

that permits shear bond strength measurement, and this could be considered as a limitation. The exact mechanism of bond failure of the core-veneer interface, for instance, the origination and the spread of the cracks and effective methods to improve core-veneer bond of all-ceramic systems need to be further investigated.

CONCLUSIONS

Within the limitations of this study, the following conclusions are made:

- Lithium disilicate porcelain bonded to the corresponding veneer porcelain showed the highest shear strength values before (47.10 ± 3.77) and after (5.68 ± 3.40) thermocycling.
- The core-veneer bond strength of zirconia based combinations was not weakened by the addition of coloring pigments.
- Thermocycling had no effect on veneering-core ceramics bonds.

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