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Shear bond strengths between different zirconia cores and veneering ceramics and their susceptibility to thermocycling

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ABSTRACT

Objectives. The purpose of this study was to evaluate the shear bond strength between various commercial zirconia core and veneering ceramics, and to investigate the effect of thermocycling.

Methods. The Schmitz–Schulmeyer test method was used to evaluate the core–veneer shear bond strength (SBS) of three zirconia core ceramics (Cercon Base, Vita In-Ceram YZ Cubes, DC-Zirkon) and their manufacturer recommended veneering ceramics (Cercon Ceram S, Vita VM9, IPS e.max Ceram). A metal ceramic system (Degudent U94, Vita VM13) was used as a control group for the three all-ceramic test groups ($n = 30$ specimens/group). Half of each group ($n = 15$) was thermocycled (5–55 °C, 20,000 cycles). Subsequently, all specimens were subjected to shear force in a universal testing machine. Fractured specimens were evaluated microscopically to determine the failure mode.

Results. The initial mean SBS values in MPa ± S.D. were 12.5 ± 3.2 for Vita In-Ceram YZ Cubes/Vita VM9, 11.5 ± 3.4 for DC-Zirkon/IPS e.max Ceram, and 9.4 ± 3.2 for Cercon Base/Cercon Ceram S. After thermocycling mean SBS values of 11.5 ± 1.7 MPa for DC-Zirkon/IPS e.max Ceram, 9.7 ± 4.2 MPa for Vita In-Ceram YZ Cubes/Vita VM9, and 9.6 ± 4.2 MPa for Cercon Base/Cercon Ceram S were observed. Neither the differences between the SBS values of the all-ceramic test groups nor the influence of thermocycling on all groups were statistically significant. Irrespective of thermocycling the metal ceramic control group (27.6 ± 12.1 MPa, 26.4 ± 13.4 MPa) exhibited significantly higher mean SBS than all three all-ceramic groups tested. The all-ceramic groups showed combined failure modes as cohesive in the veneering ceramic and adhesive at the interface, whereas the metal ceramic group showed predominately cohesive fractures.

Significance. The results indicated that the SBS between zirconia core and veneering ceramics was not affected by thermocycling. None of the zirconia core and veneering ceramics could attain the high bond strength values of the metal ceramic combination.

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1. Introduction

During the past 40 years the porcelain fused to metal technique has proven to be a reliable treatment option for fixed partial dentures (FPD) and therefore still represents the gold standard [1–3]. However, the rising interest in esthetic dentistry as well as the questionable biocompatibility of some dental metals and alloys has accelerated the development of alternatives to metallic ceramic dental restorations [4].

In the early 1990s yttrium oxide partially stabilized tetragonal zirconia polycrystal (Y-TZP) was introduced to dentistry as a core material for all-ceramic restorations and has been made available through the CAD/CAM technique. Due to a transformation toughening mechanism, Y-TZP has been shown to have superior mechanical properties compared to other all-ceramic systems [5,6]. In vitro studies demonstrated a flexural strength of 900–1200 MPa [6,7], and a fracture toughness of 9–10 MPa m$^{1/2}$ [5].

The Y-TZP framework materials Cercon Base (DeguDent, Hanau, Germany), Vita In-Ceram YZ Cubes (Vita Zahnfabrik, Bad Säckingen, Germany) and DC-Zirkon (DCS Dental AG, Allschwil, Switzerland) selected for the present study are commonly used for the application of FPD and are the subject of several in vitro and in vivo studies [8–11]. The Cercon Base and Vita In-Ceram YZ Cube frameworks are milled as enlarged constructions out of porously presintered zirconia ceramic blanks, then sintered to full density and shrunk to the desired final dimensions. The DCS system operates with highly dense sintered ceramics—the so-called hot isostatic pressed (HIPed) zirconia blanks. The DC-Zirkon cores are milled with final dimensions. According to manufacturers' recommendations the Y-TZP ceramic frameworks are veneered with suitable commonly used feldspathic veneering ceramics (Cercon Ceram S and Vita VM9) and a recently developed nano-fluorapatite glass veneering ceramic (IPS e.max Ceram).

Long-term clinical results for zirconia all-ceramic restorations are not available at the present time. In short [8] and medium-term studies [9,12–14] the Y-TZP core ceramic exhibited a high stability as a framework material. No fractures of the zirconia framework have been reported. However, some shortcomings such as marginal discrepancies and the resultant secondary caries remain to be improved [13]. The long-term success of veneered zirconia restorations seems to be determined by the weak performance of the veneering ceramics and its limited bond to the zirconia substrate. Delaminations with exposure of the zirconia core ceramic [9,13] and minor chip-off fractures [14] of the veneering ceramic were described as the most frequent reason for failures of zirconia FPDs. Chip-off fracture rates at 15% after 24 months [12] 25% after 31 months [14] and 8% and 13% after 36 and 38 months, respectively [9,13], were observed. A review of the literature for FPDs with metal framework, however, revealed either no fracture of the veneering ceramic [15] or substantially lower fracture rates ranging from 2.7% up to 5.5% for observation periods from 10 to 15 years [16,17].

The cause of fracture of veneering ceramics on zirconia all-ceramic cores was reported to be multifactorial in clinical application. Restoration geometry such as lack of proper veneering ceramic support, inadequate framework design and thickness of the ceramic layers seem to play a decisive role [13]. Moreover direction, magnitude and frequency of the applied load as well as size and location of occlusal contact areas can contribute to failures of the veneering ceramic [14].

Since the mechanical integrity and adhesion of the veneering ceramic to the ceramic substructure have proven to be key factors for the successful performance of veneer/core bilayered restorations, the initial bond strength and their reliability after thermocycling gained from in vitro investigations can provide useful information for the behavior and predictability of Y-TZP all-ceramic systems in clinical application [14].

The purpose of this study was to evaluate the shear bond strength of three commercial zirconia core ceramics and their corresponding veneering ceramics and to compare the results to the gold standard. Additionally the effect of thermocycling on the shear bond strength was investigated. Fractured surfaces were microscopically analyzed to determine the characteristics of bond failure. Due to the fact that metal ceramic FPDs have shown a reliable bond between metal core and veneering ceramic, these bond strength values served as a guideline.

The null hypotheses were that the bond strength of the zirconia all-ceramic systems would be equal to metal ceramics, and would not be affected by thermocycling.

2. Materials and methods

The manufacturers, batch numbers, chemical compositions and mechanical properties of the three commercial zirconia core ceramics (Cercon Base, Vita In-Ceram YZ Cubes, DC-Zirkon) and respective veneering ceramics (Cercon Ceram S, Vita VM 9, IPS e.max Ceram) are listed in Table 1

. Ninety all-ceramic bilayered specimens were fabricated and divided into three test groups containing 30 specimens each. Thirty high gold alloy metal ceramic specimens (Degudent U94, Vita VM13) were prepared as a control group.

2.1. *Preparation of the zirconia core (test groups)*

Nominally identical bar shaped Y-TZP core specimens of 5.0 mm length, 5.4 mm width and 13.0 mm height were produced following the Schmitz–Schulmeyer method [18] (Fig. 1). All core specimens were supplied by the manufacturer.

2.1.1. *Cercon Base (DeguDent, Hanau, Germany)*

The porously presintered Y-TZP Cercon Base blanks were milled by the Cercon brain unit (DeguDent, Hanau, Germany) and thereafter sintered to full density in the Cercon heat furnace (DeguDent, Hanau, Germany).

2.1.2. *Vita In-Ceram YZ Cubes (Vita Zahnfabrik, Bad Säckingen, Germany)*

The porously presintered Y-TZP Vita In-Ceram YZ Cube blanks were milled in the Cerec InLAb unit (Sirona, Bensheim, Germany) and then sintered in the Vita ZYrcomat furnace (Vita Zahnfabrik, Bad Säckingen, Germany).

Table 1 – Chemical composition and mechanical properties of the core and veneering materials (CTE, coefficient of thermal expansion; *, 0.2% yield-strength) according to the manufacturers' instructions

Material manufacturer batch number	CAD CAM system manufacturer	Main components [mass %]	CTE [10^{-6} K^{-1}]	Flexural strength [MPa]	Modulus of elasticity [GPa]
Core material—test group					
Cercon base DeguDent, Hanau, Germany Batch: 20007715	Cercon Brain Degu Dent, Hanau, Germany	ZrO_2 (HfO_2) = 95 (<2 HfO_2); Y_2O_3 = 5; Al_2O_3+other oxides <1 (+SiO_2)	10.5	900	210
Vita In-Ceram 2000 YZ Cubes Vita Zahnfabrik, Bad Säckingen, Germany Batch: pilot charge A1	Cerec InLab Sirona, Bensheim, Germany	ZrO_2 (HfO_2) = 95 (<3 HfO_2); Y_2O_3 = 5; Al_2O_3+other oxides <1 (+SiO_2)	10.5	>900	210
DC-Zirkon DCS Dental AG, Allschwil, Switzerland Metoxit AG, Thayngen, Switzerland Batch: pilot charge Z001	Precident System DCS Dental AG, Allschwil, Switzerland	ZrO_2 (HfO_2) = 95; Y_2O_3<5; Al_2O_3+other oxides <1 (+Na_2O)	10	1200	210
Core material—control group					
Degudent U94 Degudent, Hanau, Germany Batch: 10013673		Au 76%, Pt 9.6%, Pd 8.9%, Ag 2.5%, In 1.5%, other <1%(Ir, Cu, Sn, Ta, Re)	13.8–14.1	470*	103
Veneering ceramic—test group					
Cercon Ceram S Liner: LC 3 DeguDent, Hanau, Germany Batch: 0011/1		SiO_2 60.0–70.0; Al_2O_3 7.5–12.5; K_2O 7.5–12.5; Na_2O 7.5–12.5	9.5	80–90	60–70
Cercon Ceram S Dentin (*feldspathic veneering ceramic*) Batch: 0011/1		SiO_2 60.0–70.0; Al_2O_3 7.5–12.5; K_2O 7.5–12.5; Na_2O 7.5–12.5	9.5	80–90	60–70
VitaVM9 Liner: Effect Bonder Vita Zahnfabrik, Bad Säckingen, Germany Batch: 7263		Confidential	9.1–9.2	106	/
Vita VM9 Dentin (*feldspathic veneering ceramic*) Batch: 7263		SiO_2 60–64; $Al_2O_3$13–15; K_2O 7–10; Na_2O 4–6; $B_2O_3$3–5	9 ± 0.2	100	65
IPS e.max Ceram Liner: Test material Liner 200 Ivoclar Vivadent AG, Schaan, Liechtenstein Batch: 620990		SiO_2 50–60; Al_2O_3 16–22; K_2O 4–8; Na_2O 6–11; CaO, P_2O_5 and F: 2.0–6.0; other oxides 1.5–8, pigments: 0.1–3	9.8 ± 0.25	90 ± 10	65 ± 10
IPS e.max Ceram		SiO_2 60–65; Al_2O_3 9–11; K_2O 7–8; Na_2O 7–8; ZnO_2 2–3; CaO, P_2O_5 and F 2.5–7.5	9.5 ± 0.25	90	65 ± 10

Dentin: Test Material (*flourapatite glass veneering ceramic*) Batch: 620990				
Veneering ceramic—control group				
Vita VM13 Opaque Vita Zahnfabrik, Bad Säckingen, Germany Batch: 7802	Opaque: SiO_2 40–44, Al_2O_3 11–14, K_2O 7–10, Na_2O 4–6, CeO_2 13–16	14.0	106	/
Vita VM13 Dentin (*feldspathic veneering ceramic*) Batch: 7733	Dentin: SiO_2 59–63, Al_2O_3 13–16, K_2O 9–11, Na_2O 4–6	13.1–13.6	121	63

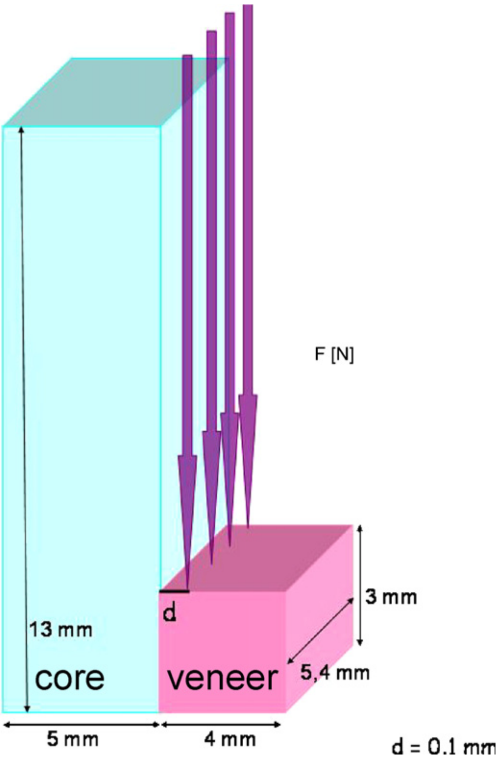

Fig. 1 – Design and dimensions of Schmitz Schulmeyer specimen. Arrows indicate load application during shear bond testing.

2.1.3. *DC-Zirkon (DCS Dental AG, Allschwil, Switzerland)*

The densely sintered Y-TZP DC-Zirkon blocks could not be milled into the required dimensions by the Precimill unit (DCS Dental AG, Allschwil, Switzerland) and were therefore cut under water-cooling using a diamond saw.

2.2. *Preparation of the metal core (control group)*

2.2.1. *Degudent U94 (DeguDent, Hanau, Germany)*

The bars were cast in high gold metal ceramic gold–platinum–palladium alloy (Degudent U94) according to the manufacturers' instructions.

2.3. *Preparation of the core veneer specimens*

All core specimens were pretreated according to their manufacturers' recommendations (Table 2). The Cercon Base cores were sandblasted with 110 μm Al_2O_3 particles at 2.5 bar pressure (Rocatec-Pre, Espe, Seefeld, Germany) after sintering. Prior to the veneering all core specimens were steam-cleaned (Jaeger, Weimsheim, Germany) and air-dried (Table 2). After a thin liner layer was fired, the veneering ceramic was built up to final dimensions (4.0 mm length, 5.4 mm width, 3.0 mm height) using a metal mold according to the Schmitz–Schulmeyer method [18] (Fig. 1). Each veneering ceramic powder was mixed with the corresponding manufacturer's liquid and the slurry obtained applied to the core followed by blotting with tissue (Kimwipes Lite 200, Kimberly Clark, Koblenz, Germany) to draw off excess water. All firing steps followed the exact procedure recommended by the manufacturers (Table 2). The metal ceramic specimens were produced identically to the all-ceramic specimens, except a second layer of opaque porcelain applied as recommended by the manufacturer. Subsequently glaze-firing was applied to all specimens according to each manufacturer's recommendation.

Table 2 – Pretreatment of the core material, applied veneering materials and veneering procedures

Veneering ceramic	Pretreatment of the core material	Materials used for 1. firing	Materials used for 2. firing	Glaze firing	Furnace manufacturer
Cercon Ceram S	Sandblasting: Al_2O_3, 110 μm, 2.4 bar Steam cleaning Air-drying	Liner LC 3 modeling liquid OL	Chromadentin CD C3 and CD D3 modeling liquid SD	One layer	Multimat MCII (Biodent, Quebec, Canada)
Vita VM9	Steam cleaning Air-drying	Effect Bonder 1 Effect Bonder Fluid	Dentin 2M 3 Modeling Liquid Vita VM 7 Vita VM 9	One layer	Multimat MCII (Biodent, Quebec, Canada)
IPS e.max Ceram	Steam cleaning	Test Material Liner 200	Test Material Dentin A2 and A3,5	One layer	Programat P100 (Ivoclar Vivadent, Schaan, Liechtenstein)
	Air-drying	1/3 Empress glaze and painting liquid 2/3 Universalschicht EAM 462 liquid	Universalschicht EAM 462		
Vita VM13	Sandblasting Al_2O_3, 110 μm, 2.4 bar Tempering at 980 °C Sandblasting Al_2O_3, 110 μm; 2.4 bar Steam cleaning Air-drying	2 opaque firings: Vita VM13 Opaque Modeling Opaque Liquid VitaVM	Vita VM13 Dentin Modeling Liquid Vita VM	One layer	Programat P80 (Ivoclar Vivadent, Schaan, Liechtenstein)

2.4. *Thermocycling*

Prior to shear bond testing, half of each group ($n = 15$) was subjected to thermocycling for 20,000 cycles at temperatures alternating between 5 and 55 °C with an immersion time of 45 s (Sabri Enterprises, Illinois, USA). Transfer time between baths was 2 s. All specimens exposed to thermocycling were kept in deionized water at room temperature. The remaining specimens ($n = 15$) were stored dry at room temperature.

2.5. *Shear bond strength test*

Each specimen was tightened in a metal holder in a universal testing machine (Z010, Zwick, Ulm, Germany). Load was applied parallel to the long axis of the specimen through a wedge at the core–veneer interface at a crosshead speed of 5 mm/min until delamination of the veneering ceramic occurred (Fig. 1). The maximum load at failure of the veneering ceramic was recorded by the system's software (Test-Xpert, Zwick, Ulm, Germany). Shear bond strengths [MPa] were calculated by dividing the failure load [N] by the bonding area [mm^2].

2.6. *Intrinsic shear bond strength of the veneering ceramics*

To determine the intrinsic shear bond strength of the Cercon Ceram S, Vita VM9 and IPS e.max Ceram veneering ceramics, specimens ($n = 5$ per veneering ceramic) consisting of mere veneering ceramic (no core) were produced according to the Schmitz–Schulmeyer method [18] and tested in the universal testing machine, as described above.

2.7. *Microscopic examination*

After the shear bond test the fractured surfaces and their interfaces were visually analyzed with a microscope (Axioscop, Zeiss, Jena, Germany) at original magnification X 10 and a video camera (3CCD, AVT-Horn, Sony, Köln, Germany). The surface with remaining veneering ceramic was measured (analySIS 3.0 Soft Imaging System, Münster, Germany) and divided by the total bonded area to determine the failure mode in percentages. Sections of the core veneer interface and selected fractured surfaces were additionally evaluated by scanning electron microscopy (SEM: LEO, DSM 950 Zeiss, Oberkochen, Germany).

2.8. *Statistical analysis*

Statistical analysis of the shear bond strength was carried out using unpaired *t*-tests. The *p*-values were adjusted with the Holm post hoc test to determine significant differences.

$p < 0.05$ are considered to be statistically significant in all tests (R Foundation for Statistical Computing, Vienna, Austria). The means of the shear bond strength measurements with 95%-CI were used to illustrate the results. Using a graphical procedure justified the assumption that a normal distribution of the data can be assumed. Balanced analysis of variance (ANOVA) was used for testing the group and thermocycling effect.

3. Results

The shear bond strength results before and after thermocycling are shown in Table 3 and Fig. 2. During thermocycling one Vita In-Ceram YZ-Cubes/Vita VM9 and one Cercon Base/Cercon Ceram S specimen showed complete delamination of the veneering ceramic. The shear bond strength of these two specimens was therefore indicated 0 MPa. The difference between the all-ceramic test groups was irrespective of thermocycling not statistically significant (Table 3). The mean shear bond strength of the metal ceramic control group (Degudent U94/Vita VM13) was statistically significantly higher than any of the all all-ceramic test groups, before and after thermocycling (Fig. 2). The effect of thermocycling on the shear bond strength of the test groups as well as on the control group was not statistically significant (Table 3). The intrinsic shear bond strengths of the veneering ceramics are shown in Table 4 and were statistically significantly higher than the measured corresponding shear bond strengths. Cercon Base/Cercon Ceram S showed combined fracture modes: cohesive in the veneer and adhesive at the core veneer interface (Table 3, Fig. 3a). DC-Zirkon/IPS e.max Ceram and Vita In-Ceram YZ Cubes/Vita VM9 showed predominant adhesive fractures at the core veneer interface. None of the core veneer specimens failed cohesively in the core material. The metal ceramic control group Degudent U94/Vita VM13 mainly showed cohesive fractures of the veneering ceramic (Table 3). SEM analysis of the all-ceramic test groups revealed porosities in the veneering ceramic and structural defects at the zirconia veneer interface (Fig. 3b).

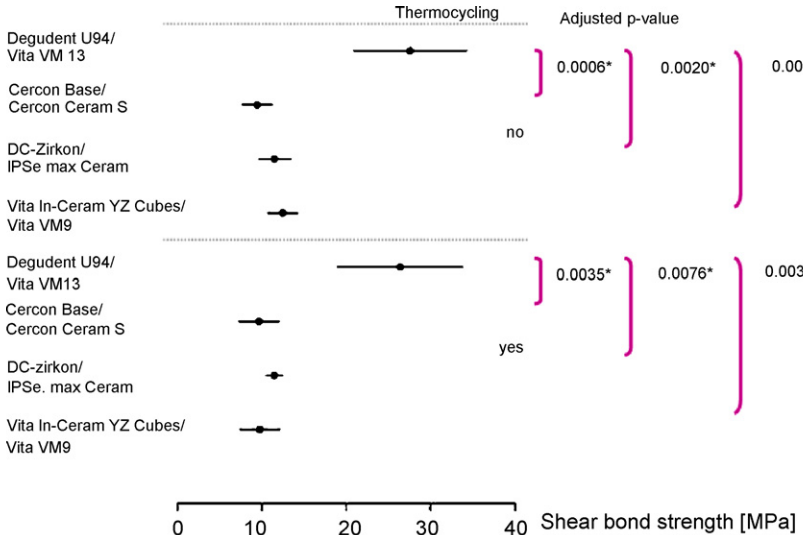

Fig. 2 – Estimated means of the shear bond strength measurements of zirconia ceramic test groups and metal ceramic control group with 95%-CI. Adjusted *p*-values show the statistical significance of the comparison, respectively (significant difference *$p < 0.05$).

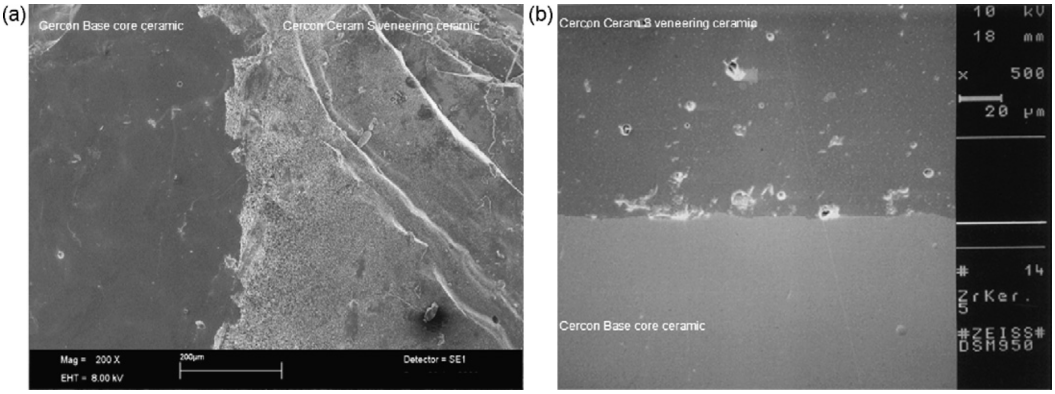

Fig. 3 – (a) Combined fracture mode as cohesive in the veneering ceramic (Cercon Ceram S) and adhesive at the interface with exposure of the zirconia core ceramic (Cercon Base), (b) Section through the interface: structural defects observed in the veneering ceramic (Cercon Ceram S) and at the interface to the zirconia core ceramic (Cercon Base).

Table 3 – Results (TC, thermocycling; SBS, shear bond strength; 95%-CI, 95% confidence interval; S.D., standard deviation, △ SBS, shear bond strength changes after thermocycling in %, *p*-values adjusted by method Holm ($p < 0.05$); SV mean surface with remaining veneering ceramic in percentage)

Group	Core material	Veneering ceramic	TC/storage	SBS [MPa] mean	95%-CI	S.D.	Δ SBS [%] after TC	Comparison dry/TC adjusted *p*-value	SV [%] mean	Comparison of test groups before/after TC	Adjusted *p*-value
A Test	Cercon base	Cercon Ceram S	Dry	9.4	7.6–11.2	3.2	−2.1	1	54.6	Cercon Base-Ceram S/Vita In-Ceram YZ Cubes-Vita VM9 dry	0.1572
			TC	9.6	7.3–12.0	4.2			51.4	Cercon Base-Ceram S/Vita In-Ceram YZ Cubes-Vita VM9 TC	1.000
B Test	Vita In-Ceram YZ Cubes	Vita VM9	Dry	12.5	10.7–14.2	3.2	28.9	0.55	42.2	Vita In-Ceram YZ Cubes-Vita VM9/DC-Zirkon-IPS e.max Ceram dry	1.000
			TC	9.7	7.4–12.1	4.2			38.1	Vita In-Ceram YZ Cubes-Vita VM9/DC-Zirkon-IPS e.max Ceram TC	0.9920
C Test	DC-Zirkon	IPS e.max Ceram	Dry	11.5	9.6–13.4	3.4	0	1	31.8	DC-Zirkon-IPS e.max Ceram/Cercon Base-Ceram S dry	0.8187
			TC	11.5	10.5–12.4	1.7			29.5	DC-Zirkon-IPS e.max Ceram/Cercon Base-Ceram S TC	0.9802
D Control	Degudent U94	Vita VM13	Dry	27.6	19.0–30.7	12.1	4.5	1	89.5	–	–
			TC	26.4	15.9–32.7	13.4			77.6	–	–

Table 4 – Results (ISBS, intrinsic shear bond strength of veneering ceramics, group comparison, comparison of ISBS and SBS before and after TC, *p*-values adjusted by Holm method ($p < 0.05$))

Veneering ceramic	ISBS [MPa] mean	95%-CI	S.D.	Comparison of ISBS	Adjusted *p*-value	Comparison of ISBS and SBS before/after TC	Adjusted *p*-value
Cercon Ceram S	33.6	14.0–53.1	15.8	Cercon Ceram S/IPS e.max Ceram	0.8564	Cercon Ceram S/Cercon Ceram S TC	0.1813
						Cercon Ceram S/Cercon Ceram S dry	0.1813
Vita VM9	25.5	3.7–47.65	17.8	Vita VM9/Cercon Ceram S	0.8564	Vita VM9/Vita VM9 TC	0.0792
						Vita VM9/Vita VM9 dry	0.0792
IPS e.max Ceram	38.2	22.3–54.0	12.8	IPS e.max Ceram/Vita VM9	0.7094	IPS e.max Ceram/IPS e.max Ceram TC	0.7092
						IPS e.max Ceram/IPS e.max Ceram dry	0.596

4. Discussion

Bond strength measurement of metal ceramic systems was standardized by the Organization of Standardization through the Schwickerath crack initiation test (three point bending test). A minimum bond strength of 25 MPa for metal ceramic combinations was established [19]. Due to the brittleness of all-ceramic core materials this test setup cannot be applied to all-ceramic multilayered systems [20]. In a survey of the literature few articles address the bond strength of all-ceramic core and veneering ceramics (Table 5). To date an adequate standardized test setup and a minimum required bond strength for bilayered all-ceramic materials has not been determined [21–23].

The Schmitz–Schulmeyer test [18], a planar interface shear bond test, is based on minimal experimental variables and has proven to be a reliably well-suited test set up for metal ceramic bond strength measurements [24]. The stresses during the shear tests were reported to be directed mainly at the interface resulting in a relatively uniform distribution of interfacial stresses [25]. The method was therefore transferred for the application of all-ceramic systems [26] and chosen for the present study.

In order to reject the approach that shear tests may not be measuring the strength of the adherence zone but rather the mechanical properties of the veneering ceramic [25] the authors determined the intrinsic shear bond strength of the veneering ceramics by using the Schmitz–Schulmeyer test method and compared them to the shear bond strength data of bilayered composites. The intrinsic shear bond strength of the veneering ceramics were in the range of 25.5 and 38.2 MPa and were significantly higher than the measured shear bond strengths between the core and veneering ceramics. It can be concluded that the Schmitz–Schulmeyer test can be considered as an applicative test to measure the effective shear bond strength of bilayered all-ceramic systems.

The zirconia systems Vita In-Ceram YZ Cubes/Vita VM9, DC-Zirkon/IPS e.max Ceram and Cercon Base/Cercon Ceram S showed bond strength values in the range of 9.4–12.5 MPa that did not differ significantly. The metal ceramic bond strengths (26.6 and 26.4 MPa) were irrespective of exposure to thermocycling significantly higher than those of all zirconia test groups.

The interpretation of these shear bond strength data requires consideration of three factors: (1) bonding of the core and veneering ceramic materials, (2) coefficient of thermal expansion (CTE) of the core and veneering ceramics, and (3) cooling rate and geometry of the bilayered ceramic composite.

(1) The highly divergent results of the all-ceramic test and metal ceramic control group can primarily be attributed to the different adhesion mechanism of metal and zirconia core materials to veneering ceramics. Whereas mechanical interlocking and primarily the chemical bond resulting from suitable metal oxidation and interdiffusion of ions seem to play the most prominent role in the metal ceramic interface [27,28], the bonding mechanisms of veneering ceramics to Y-TZP surfaces are up to now unclear. Based on investigations on the wettability of zirconia core with veneering ceramics, micromechanical interactions were merely assumed [29]. Following the manufacturers' recommendation the surface of Cercon Base zirconia core prior to veneering (Cercon Ceram S) was sandblasted to promote a mechanical interlock.
Surface roughening with 110 μm Al_2O_3 (2.4 bar) sandblasting had no significant influence on the core veneer bond strength in comparison to the non-sandblasted Vita In-Ceram YZ Cubes/Vita VM9 and DC-Zirkon/IPS e.max Ceram groups. These results could be confirmed in a related study, where the effect of sandblasting on the shear bond strength for all zirconia core and corresponding veneering ceramics investigated, was systematically analyzed (unpublished data from the same author group).

(2) The effect of the CTE mismatch on veneer and core bonding has been frequently discussed in the dental literature [30,31]. The bond strength can be compromised by residual stresses from veneer and core CTE mismatch [32]. To generate acceptable levels of residual stress within a multilayer all-ceramic composite, efforts have been made by dental manufacturers to develop ceramic cores and low fusing veneering ceramics with similar CTE. In the present study the CTE mismatch varied from

Table 5 – Bond strength results of metal ceramic and all-ceramic systems and applied test methods (TC, Thermocycling; SBS, shear bond test; MTBS, microtensile bond test; n.s., not specified)

Reference	Core material/veneering ceramic	Bond strength [MPa] (S.D.)		Storage/TC	Test method
[18]	Metal ceramic: n.s.	34.9 (21.3)		n.s.	Schmitz–Schulmeyer
[46]	Metal ceramic			n.s.	Schmitz–Schulmeyer
	Cameo/Ceramco Porcelain (Jelenko, USA; Ceramco, USA)	15.2 (2.1)			
	Degudent U/Ceramco Porcelain (Degussa, Germany)	13.6 (3.2)			
	Microbond Hi-Life/Ceramco Porcelain (Howmedica Inc, USA)	13.1 (2.3)			
	V-Delta/Ceramco Porcelain (Metaux Precieux, Switzerland)	13.3 (1.4)			
[47]	Metal ceramic			n.s.	Schmitz–Schulmeyer
	Armator 2/Biodent (UGDO, Geneva, Switzerland, DeTrey, Wiesbaden, Germany)	13.5 (15.0)			
[26]	Zirconia TZP/Veneering ceramic for Zirconia (Metoxit, Thayngen, Switzerland; Vita Zahnfabrik, Bad Säckingen, Germany)	36.2 (12.1)		n.s.	Schmitz–Schulmeyer
[21]	IPS Empress 2/Eris (Ivoclar Vivadent, Amherst, USA)	30.86 (6.47)		n.s.	Shear bond test
	Procera All Zirkon/Cerabien CZR (Procera Sandvik, Gothenberg, Sweden; Noritakekizai, Nagoya, Japan)	28.03 (5.03)			
	DC-Zirkon/Vita D (Metoxit AG, Thanygen, Switzerland; Vita Zahnfabrik, Bad Säckingen, Germany)	27.9 (4.79)			
	Procera AllCeram/Degussa Ney AllCeram (Procera Sandvik, Gothenberg, Sweden, Dentsply, New York USA)	22.40 (2.40)			
	metal ceramic:				
	Lodestar/Noritake (Ivoclar Vivadent, Amherst, USA; Noritakekizai, Nagoya, Japan)	30.16 (5.89)			
[22]		Dry storage	TC	TC	Shear bond test
	IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein)	41 (8)	31 (4)	5 cycles	
	Finesse (Ceramco, NJ, USA)	28 (4)	n.s	5 and 55 °C	
	In-Ceram Alumina (Vita Zahnfabrik, Bad Säckingen, Germany)	26 (4)	n.s	30 s Dwell time	
	Evopress (Wegold, Wendelstein, Germany)	23 (3)	n.s	Dry storage	
[23]		SBS	MTBS	Distilled water	Shear bond test
	IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein)	41 (8)	9 (1)	37 °C	Micro tensile bond test
	Finesse (Ceramco, NJ, USA)	28 (4)	15 (2)	1 Week	
	In-Ceram Alumina (Vita Zahnfabrik, Bad Säckingen, Germany)	26 (4)	9 (1)		
	IPS Empress (Ivoclar Vivadent, Schaan, Liechtenstein)	23 (3)	12 (2)		
[32]	Cercon Base/Cercon Ceram S (DeguDent, Hanau, Germany)	29.1 (13.7)		n.s.	Micro tensile bond test
	IPS Empress 2/Eris (Ivoclar Vivadent, Schaan, Liechtenstein)	44.6 (9.1)			
	IPS Empress 2/IPS Empress 2 Veneer (Ivoclar Vivadent, Schaan, Liechtenstein)	37.2 (10.8)			
	Vita Mark II/Vitadur Alpha (Vita Zahnfabrik, Bad Säckingen, Germany)	32.2 (7.8)			
[43]	Cercon Base (DeguDent, Hanau, Germany)	Without	With liner	n.s.	Micro tensile bond test
	Ceram S	17.2 (4.1)	26.3 (8.6)		
	Ceram Express	38.6 (6.4)	29.7 (8.9)		
	Rondo Dentin (Nobel Biocare, Göteborg, Sweden)	41.1 (11.1)	30.8 (14.5)		
	Rondo Shoulder	39.3 (9.6)	–		
	Lava Dentin (3M Espe, Seefeld, Germany)	30.9 (7.2)	34.3 (7.0)		
	Sakura Interaction (Elephant Dental, Hoorn, Netherland)	19.9 (9.2)	23.8 (7.8)		
	Experimental pressable (ACTA, Netherland)	25.2 (7.4)	–		

0.75 to 1.7 × $10^{-6x} \times K^{-1}$ for the three all-ceramic systems (Table 1), but the measured bond strengths showed no difference. The fact that the CTE of veneering ceramics is nonlinear and varies, depending on the temperature interval studied, the time of heat soak at peak firing temperature and particle size should also be taken into consideration. Therefore, the question arises if the CTE measured between 25 and 500 °C is adequate to characterize the thermal compatibility of all-ceramic core and veneer systems. In addition, dental ceramics show phase changes as a result of thermal history (i.e. number of firings) [33]. Whereas this change in thermal dimensional behavior does not result in significant problems in metal ceramic systems, it may affect the thermal compatibility of the ceramic core and veneering materials [34]. The chemical compatibility of metal ceramic systems implies a bond strong enough to resist both transient and residual thermal stresses during veneer ceramic firing and could be taken into consideration as explanation for the superior bond strength values in comparison to the all-ceramic combinations [35].

(3) Whereas dental metal alloys have a high thermal conductivity (in the range of 300 $\mathrm{W\,m^{-1}\,K^{-1}}$ for noble alloys) zirconia core materials are thermal insulators [36]. Based on various manufacturers' data Y-TZP core materials exhibit a thermal conductivity of 2–2.2 $\mathrm{W\,m^{-1}\,K^{-1}}$. Feldspathic veneering ceramics are in the same range with a thermal conductivity of 2.39 $\mathrm{W\,m^{-1}\,K^{-1}}$ [36]. The combined low thermal conductivities of core and veneering ceramics greatly retard the porcelain cooling rate at the interface as compared to the metal configuration potentially changing the CTE and introducing residual thermal stresses [37,38]. These residual interfacial stresses are a possible explanation for the thermal cycling delaminations of the veneering ceramic of one Vita In-Ceram YZ-Cubes/Vita VM9 and one Cercon Base/Cercon Ceram S specimen and could contribute to the lower shear bond values for these systems. Clinically, additional residual stresses may also result from place to place variation in thermal properties owing to irregular veneering ceramic thickness and the relative core veneer layer thickness ratio [39].

Oral fluids are known to facilitate stress corrosion of ceramic materials, resulting in slow crack growth and finally leading to failure of ceramic restorations in the complex situation of the oral cavity [40,41]. The in vitro aging sensitivity of the shear bond strength of bilayered specimens was therefore assessed by exposure to a standardized thermocycling test setup [22]. In the present study the application of 20,000 cycles of thermocycling had no influence on the shear bond strength of all groups investigated. Comparative studies on the bond strength of zirconia core and veneering ceramics after exposure to thermocycling are not available up to now. The stable bond strength of the metal ceramic combination is in agreement with the literature [42].

The failure mode observed for the Cercon Base/Cercon Ceram S, Vita In-Ceram YZ Cubes/Vita VM9 and DC Zircon/IPS e.max Ceram all-ceramic systems was mainly combined as adhesive at the interface and cohesive in the veneering ceramic. The described failure modes with delamination of the veneer from intact zirconia core structure were comparable to the results of other laboratory studies, where crack deflection has been identified at the core/veneer-interface [11,43,44]. This can be interpreted in two ways: First, crack deflection could be a consequence of the superior ability of Y-TZP to resist crack propagation. Second, the interlaminar crack deflection could also correlate with the relatively poor bond of the zirconia core to veneering ceramic. The clinical implication of this finding is that the investigated all-ceramic systems could have a tendency to produce chip-off fractures of the veneering ceramic and delaminations rather than catastrophic failure of the core structure [45]. The exact mechanism of apparent interfacial bond failure in the current study is unknown and needs further investigation. The microscopic observations of the fractured metal ceramic specimens showed that a rim of porcelain remained on the metal part of all specimens and that the veneering ceramic adhered to the alloy. The predominately cohesive mode of failure of the metal ceramic control group is in agreement with previous reports in the literature [46].

The authors acknowledge that depending on the technical skills, particularly required for the sophisticated layering technique production of all-ceramic composites, porosities and micro-gap formations at the interface could be observed in the present study and may be another factor that weakens the interfacial bond [23,26,43].

As a limitation of this study the authors admit that the layered all-ceramic specimens investigated do not represent clinical shape conditions of dental restorations, but provide a geometry that permits shear bond strength measurement.

Considerable refinements are required to obtain quantitative estimates of interfacial stress in the complex-shaped multicomponent dental restoration. Predictive models such as finite element analysis and investigations on the effects of residual stresses and cooling rates could be most enlightening.

In comparison to the gold standard, adequate shear bond strength values between Y-TZP core and their corresponding veneering ceramics investigated could not be attained. The low bond strength values of all Y-TZP ceramic systems investigated can be considered as a possible explanation for the high fracture rates of the veneering ceramics observed in clinical studies. Further investigations, development and refinement of the Y-TZP core and veneering ceramic interface are necessary for clinical long-term success.

5. Conclusions

The Schmitz–Schulmeyer test can be considered a simple and reliable screening method to evaluate shear bond strengths of metal and all-ceramic systems.

Based on the shear bond strength results of the present study the interceramic bond between zirconia core and veneering ceramics requires considerable refinements in order to overcome existing thermal incompatibilities between zirconia core and veneering ceramics, and to match the values set by the metal ceramic gold standard.

REFERENCES

[1] Tan K, Pjetursson BE, Lang NP, Chan ES. A systematic review of the survival and complication rates of fixed partial dentures (FPDs) after an observation period of at least 5 years. Clin Oral Implants Res 2004;15:654–66.

[2] Scurria MS, Bader JD, Shugars DA. Meta-analysis of fixed partial denture survival: prostheses and abutments. J Prosthet Dent 1998;79:459–64.

[3] Creugers NH, Kayser AF, van't Hof MA. A meta-analysis of durability data on conventional fixed bridges. Commun Dent Oral Epidemiol 1994;22:448–52.

[4] Raigrodski AJ. Contemporary materials and technologies for all-ceramic fixed partial dentures: a review of the literature. J Prosthet Dent 2004;92:557–62.

[5] Christel P, Meunier A, Heller M, Torre JP, Peille CN. Mechanical properties and short-term in vivo evaluation of yttrium-oxide-partially-stabilized zirconia. J Biomed Mater Res 1989;23:45–61.

[6] Tinschert J, Zwez D, Marx R, Anusavice KJ. Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics. J Dent 2000;28:529–35.

[7] Filser F, Kocher P, Weibel F. Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM). Int J Comput Dent 2001;4:89–106.

[8] Sturzenegger BFA, Luthy H, Schumacher M, Loeffel O, Filser F, Kocher P, et al. Clinical evaluation of zirconium oxide bridges in the posterior segments fabricated with the DCM system. Acta Med Dent Helv 2000;5:131–9.

[9] Tinschert J, Natt G, Latzke P. [All-ceramic FPDs made of DC-Zirkon—a clinical concept with success?]. DZZ 2005;60:435–45.

[10] Studart AR, Filser F, Kocher P, Luthy H, Gauckler LJ. Cyclic fatigue in water of veneer-framework composites for all-ceramic dental bridges. Dent Mater 2007;23:177–85.

[11] Studart AR, Filser F, Kocher P, Luthy H, Gauckler LJ. Mechanical and fracture behavior of veneer-framework composites for all-ceramic dental bridges. Dent Mater 2007;23:115–23.

[12] Vult von Steyern P, Carlson P, Nilner K. All-ceramic fixed partial dentures designed according to the DC-Zirkon technique. A 2-year clinical study. J Oral Rehabil 2005;32:180–7.

[13] Sailer I, Feher A, Filser F. Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow up. Quintessence Int 2006;37:685–93.

[14] Raigrodski AJ, Chiche GJ, Potiket N. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. J Prosthet Dent 2006;96:237–44.

[15] Walter M, Reppel PD, Boning K, Freesmeyer WB. Six-year follow-up of titanium and high-gold porcelain-fused-to-metal fixed partial dentures. J Oral Rehabil 1999;26:91–6.

[16] Coornaert J, Adriaens P, De Boever J. Long-term clinical study of porcelain-fused-to-gold restorations. J Prosthet Dent 1984;51:338–42.

[17] Valderhaug J. A 15-year clinical evaluation of fixed prosthodontics. Acta Odontol Scand 1991;49:35–40.

[18] Schmitz K, Schulmeyer H. [Determination of the adhesion of dental metal-porcelain bonding systems]. Dental Labor 1975;23:1416–20.

[19] ISO 9693. Metal-ceramic bond characterization (Schwickerath crack initiation test). Geneva, Switzerland: International Organization for Standardization; 1999.

[20] Albakry M, Guazzato M, Swain MV. Fracture toughness and hardness evaluation of three pressable all-ceramic dental materials. J Dent 2003;31:181–8.

[21] Al-Dohan HM, Yaman P, Dennison JB, Razzoog ME, Lang BR. Shear strength of core–veneer interface in bi-layered ceramics. J Prosthet Dent 2004;91:349–55.

[22] Dundar M, Ozcan M, Comlekoglu E, Gungor MA, Artunc C. Bond strengths of veneering ceramics to reinforced ceramic core materials. Int J Prosthodont 2005;18:71–2.

[23] Dundar M, Ozcan M, Gokce B, et al. Comparison of two bond strength testing methodologies for bilayered all-ceramics. Dent Mater 2007;23:630–6.

[24] Hammad IA, Talic YF. Designs of bond strength tests for metal-ceramic complexes: review of the literature. J Prosthet Dent 1996;75:602–8.

[25] Anusavice KJ, Dehoff PH, Fairhurst CW. Comparative evaluation of ceramic-metal bond tests using finite element stress analysis. J Dent Res 1980;59:608–13.

[26] Luthardt RG, Sandkuhl O, Reitz B. Zirconia-TZP and alumina-advanced technologies for the manufacturing of single crowns. Eur J Prosthodont Restor Dent 1999;7: 113–9.

[27] Mackert Jr JR, Ringle RD, Parry EE, Evans AL, Fairhurst CW. The relationship between oxide adherence and porcelain-metal bonding. J Dent Res 1988;67:474–8.

[28] Schweitzer DM, Goldstein GR, Ricci JL, Silva NR, Hittelman EL. Comparison of bond strength of a pressed ceramic fused to metal versus feldspathic porcelain fused to metal. J Prosthodont 2005;14:239–47.

[29] Stephan M. Beschichtungsverhalten von Verblendmaterialien auf Dentalkeramik. Tübingen: Diplomarbeit der Geowissenschaftlichen Fakultät; 1996.

[30] De Kler M, De Jager N, Meegdes M, Van Der Zel JM. Influence of thermal expansion mismatch and fatigue loading on phase changes in porcelain veneered Y-TZP zirconia discs. J Oral Rehabil 2007 [online early articles].

[31] Fairhurst CW, Anusavice KJ, Ringle RD, Twiggs SW. Porcelain-metal thermal compatibility. J Dent Res 1981;60:815–9.

[32] Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Dent Mater 2005;21:984–91.

[33] Mackert Jr JR, Butts MB, Fairhurst CW. The effect of the leucite transformation on dental porcelain expansion. Dent Mater 1986;2:32–6.

[34] Isgro G, Wang H, Kleverlaan CJ, Feilzer AJ. The effects of thermal mismatch and fabrication procedures on the deflection of layered all-ceramic discs. Dent Mater 2005;21:649–55.

[35] Bagby M, Marshall SJ, Marshall Jr GW. Metal ceramic compatibility: a review of the literature. J Prosthet Dent 1990;63:21–5.

[36] Biomaterials Properties Online Database. University of Michigan. Quintessence Publishing 1996; www.lib.umich.edu/dentlib/Dental_tables/Thermcond.html.

[37] Hermann I, Bhowmick S, Zhang Y, Lawn BR. Competing fracture modes in brittle materials subject to concentrated cyclic loading in liquid environments: Trilayer structures. J Mater Res 2006;21:512–21.

[38] Mora GP, O'Brien WJ. Thermal shock resistance of core reinforced all-ceramic crown systems. J Biomed Mater Res 1994;28:189–94.

[39] Hojjatie B, Anusavice KJ. Effects of initial temperature and tempering medium on thermal tempering of dental porcelains. J Dent Res 1993;72:566–71.

[40] Peterson IM, Wuttiphan S, Lawn BR, Chyung K. Role of microstructure on contact damage and strength degradation of micaceous glass-ceramics. Dent Mater 1998;14:80–9.

[41] Zhang Y, Song JK, Lawn BR. Deep-penetrating conical cracks in brittle layers from hydraulic cyclic contact. J Biomed Mater Res B Appl Biomater 2005;73:186–93.

[42] Shimoe S, Tanoue N, Yanagida H, et al. Comparative strength of metal-ceramic and metal-composite bonds after extended thermocycling. J Oral Rehabil 2004;31: 689–94.

[43] Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: zirconia veneering ceramics. Dent Mater 2006;22:857–63.

[44] Kim B, Zhang Y, Pines M, Thompson VP. Fracture of porcelain-veneered structures in fatigue. J Dent Res 2007;86:142–6.

[45] White SN, Miklus VG, McLaren EA, Lang LA, Caputo AA. Flexural strength of a layered zirconia and porcelain dental all-ceramic system. J Prosthet Dent 2005;94:125–31.

[46] Oilo G, Johansson B, Syverud M. Bond strength of porcelain to dental alloys—an evaluation of two test methods. Scand J Dent Res 1981;89:289–96.

[47] Susz CP, Meyer JM, Payan J, Stoian M, Sanchez J. [Effect of the treatments preceding porcelain baking on the strength of the ceramic-metal bond]. Schweiz Monatsschr Zahnmed 1980;90:393–404.