Influence of the coefficient of thermal expansion mismatch on the bond strength of bi-layered all ceramic system

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Dedicated To my beloved parents, wife and children for their constant support, patience, and encouragement

1 Introduction

Ceramic crowns have been used in dentistry since 1900s to restore teeth **(Powers & Wataha, 2007)**. Furthermore, from the 1980s onwards the use of ceramics has been extended to include veneers, inlays/onlays, crowns and short span bridges **(van Noort, 2007).** Ceramic originally referred to the art of fabrication of pottery. The term is derived from the Greek keramos which means potter or pottery. It is also related to a Sanskrit term means (burned earth) since the basic component were clays form the earth, that were heated to form pottery **(Touati, Miara, & Nathanson, 1999).**

Ceramics are compounds of metallic elements and nonmetallic substances such as oxides, nitrides and silicates **(van Noort, 2007).** There are two concepts helpful for our understanding of dental ceramics. First, ceramics fall into three main composition categories: Predominantly glass, Particle-filled glass and Polycrystalline (Figure 1). Second, ceramic can be virtually considered as a composite. It composed of two or more substances, in which the matrix is a glass filled lightly or heavily with particles. For Polycrystaline ceramics, the matrix is aluminum oxide or zircon oxide and the fillers are modified atoms called „dopants“ **(Kelly, 2008).**

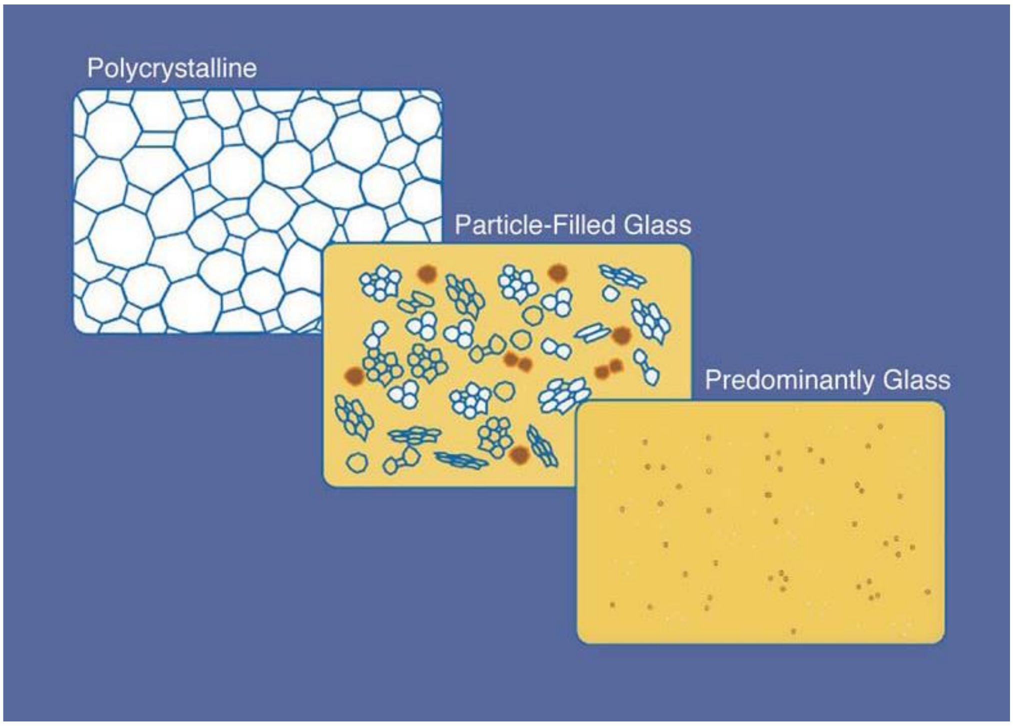


Figure 1. Schematic representation of three basic classes of dental ceramics **(Kelly, 2008).**

1.1 All-Ceramic system

Dental all ceramic systems are divided into 2 main groups based on their composition, Silicate ceramics and Oxide ceramics.

1.1.1 Silicate Ceramic

It is the oldest form of dental ceramics and is made from Materials that contain mainly silica. It consists of naturally or synthetic manufactured minerals such as feldspar, quartz, kaolin. Under silicate ceramic group, there are two types of silicate ceramics, feldspathic ceramic and glass ceramic.

Silicate ceramics has a high content of glass matrix in which crystalline particles distributed. This microstructural composition makes them the first choice when the aesthetic needs to be restored. On the other side, the high content of glass decreases the flexural strength property **(Kern et al., 2006; Strub, 1994)**

The reported flexural strength of the feldspathic ceramics ranges between 90-154 MPa, while the glass ceramic has flexural strength between 160-400 MPa. The increase of the flexural strength of the glass ceramic came from adding materials that help in improving the physical properties of the glass ceramic such as lithium-disilicate **(P. C. Guess et al., 2011).**

1.1.2 Oxide Ceramic

Under this group of all ceramic, there are two types of ceramic; Aluminium oxide ceramic and Zirconium oxide ceramic. They are used as core materials for silicate ceramic when the functional demand is high because of their high physical properties. In addition, the optical outcome is more pleasing when ceramic core is used instead of metal core (P. C. Guess et al., 2011).

In 1965, McLean and Hughes developed alumina-reinforced porcelain. They dispersed crystals of high-strength alumina in feldspathic matrix, which resulted in five times stronger porcelain than the regular porcelain. In 1993, Andersson and Oden developed the Procera All Ceramic Crown in cooperation with Nobel Biocare and Sandvik Hard Materials. This system consists of a densely sintered high-purity aluminous oxide core combined with low fusing porcelain**.** The flexural strength of Procera system is about 600MPa **(Andersson & Oden, 1993; May, Russell, Razzoog, & Lang, 1998).**

1.1.2.1Zirconia

Zirconium is a lustrous corrosion resistant metal element, discovered by Martin Heinrich Klaproth in 1789. Zirconium does not exist in nature in its pure state, but only as a free oxide (ZrO_2) or in conjunction with silicate (ZrO_2+SiO_2) so known as (Zirconia) **(Piconi & Maccauro, 1999; Vagkopoulou, Koutayas, Koidis, & Strub, 2009).**

Zirconia is a polymorphic material, which occurs in three crystallographic forms according to the temperature. Monoclinic structure exists at room temperature and upon heating up to 1170°C. Tetragonal phase occurs between 1170°C and 2370°C. Above 2370°C up to melting point, the structure is cubic. During cooling phase of the zirconia, the transformation from tetragonal to monoclinic structure occurs with 3-5% increase in volume (Figure 1.2). The volume expansion, associated with this transformation, leads to the development of internal stress that can break the zirconia into pieces at room temperature. Controlling the internal stress by adding of stabilizing oxides (e.g. MgO, CeO_2, Y_2O_3) helps to retain the tetragonal structure at room temperature, which leads to arresting crack propagation, which increases the fracture toughness of the zirconia (Figure 1.2). The toughening mechanism does not prevent the progression of a crack; it just makes it harder for the crack to propagate **(Denry & Kelly, 2008; Piconi & Maccauro, 1999)**.

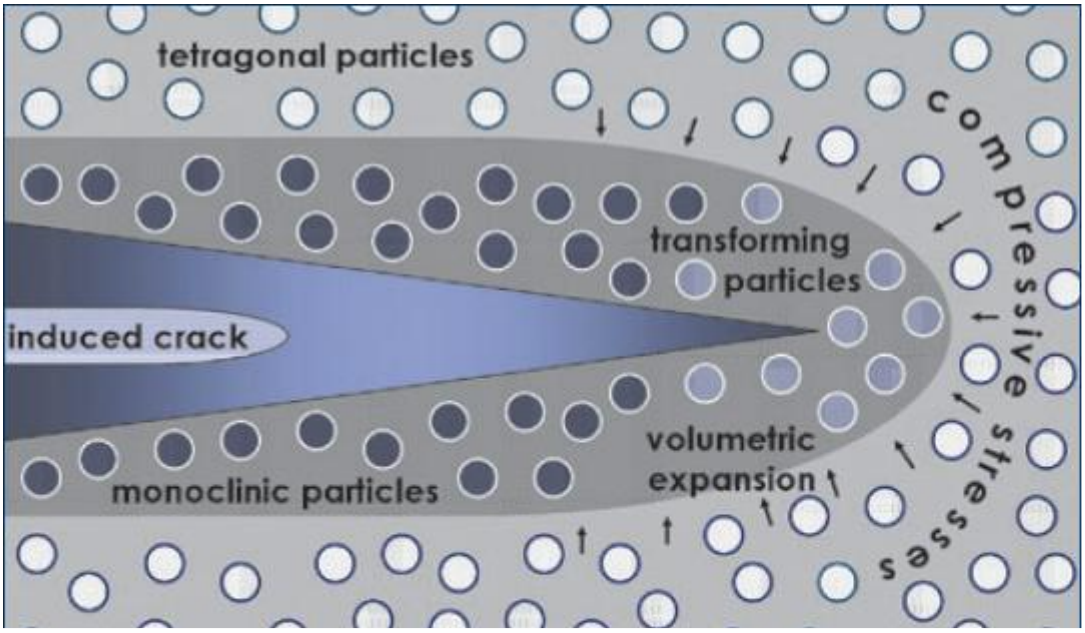


Figure 1.2: Toughening mechanism of the zirconia when the crack is induced **(Vagkopoulou et al., 2009)**

In addition to the transformation property, zirconia has other physical properties such as biocompatibility, dimensional stability, mechanical strength and toughness; these properties increased the interest in using zirconia as a ceramic biomaterial **(Denry & Kelly, 2008; Piconi & Maccauro, 1999)**.

In the medical field, zirconia has been used in the biomedical application since 1969 **(Piconi & Maccauro, 1999)**. In 1988, Christel et al. published the first paper that introduced zirconia as a material for total hip replacement **(Christel et al., 1988).** In the early 1990's zirconia entered the dental field and since then has been used for orthodontic brackets **(Winchester, 1991)**, and endodontic posts **(Meyenberg, Lüthy, & Schärer, 1995)**. After the huge development of the CAD/CAM technology, zirconia became one of the most interesting materials to be examined and used in almost the entire dental field **(Komine, Blatz, & Matsumura, 2010; Vagkopoulou et al., 2009).**

Moreover, there are two types of zirconia blocks used to produce zirconia core material by CAD/CAM technology. The first type is the fully sintered zirconia block, which is extremely difficult to be milled, time consuming and expensive due to the increased hardness of the zirconia as well as the wear of the cutting tools. On the other hand, this type of zirconia block has an advantage of superior fit because of the dimensional stability after milling process. The second type is the pre-sintered block, which has an advantage of fast milling process and lower costs. The main disadvantage of the pre-sintered blocks is the shrinkage that takes place during the final sintering stage (20-25%). To compensate that problem, the original framework must be enlarged during the milling process to compensate the shrinkage after the final sintering process **(Komine et al., 2010; Miyazaki, Hotta, Kunii, Kuriyama, & Tamaki, 2009; Raigrodski, 2004).**

1.2 Veneering Porcelain

Veneering porcelain is a term referred to the porcelain used to mask the metal or all-ceramic core in order to optimize form and aesthetic of the restoration. Zirconia has an opaque white color; therefore it has to be veneered with silicate or glass ceramic **(P. C. Guess et al., 2011).** The coefficient thermal expansion (CTE) of the zirconia is between 10.5-10.8x10^{-6} K^{-1} **(Yasuda & Hishinuma, 2000).** For establishing a strong bond between the core and the veneering ceramic, the CTE of the veneering ceramic must be adjusted to be lower than that of the zirconia **(Anusavice, DeHoff, Hojjatie, & Gray, 1989; Coffey, Anusavice, DeHoff, Lee, & Hojjatie, 1988; Fischer, Stawarzcyk, Trottmann, & Hämmerle, 2009).** The flexural strength of a commonly used veneering porcelain is about 90-120 MPa **(P. C. Guess et al., 2011).**

1.3 Bond strength

Bond strength is determined by several factors including, the strength of chemical bonds, mechanical interlocking, type and concentration of defects at the interface, wetting properties, and the degree of compressive stress in the veneering layer due to a difference in the coefficients of thermal expansion between zirconia and the veneering ceramic **(Fischer, Grohmann, & Stawarczyk, 2008)**. The bond between the weaker ceramic and the stronger framework must be of a certain minimum value and toughness to allow proper transfer of loading stresses between the two materials **(Aboushelib, De Kler, Van Der Zel, & Feilzer, 2009)**

The International Organization of Standardization has standardized the bond strength measurement for metal ceramics systems, through Schwickerath initiation crack test and determined that the mean bond strength of metal ceramic systems should be greater than 25 MPa to meet the ISO requirements **(ISO 9693).** For all ceramic bi-layered systems, there is no standardized test for bond strength measurement have been introduced yet**,** though several tests have been used to evaluate the adhesion of veneering porcelain to zirconia core material. These include the microtensile bond strength test **(Aboushelib, De Jager, Kleverlaan, & Feilzer, 2005)** Schmitz Schulmayer test **(Petra C Guess et al., 2008)** Schwickerath test **(Kosyfaki, Swain, Fischer, Witkowski, & Strub, 2013; Schille, Wieland, & Geis-Gerstorfer, 2012)**, biaxial flexural strength test **(Yilmaz, Nemli, Aydin, Bal, & Tıraş, 2011)**, and shear bond strength tests **(Fischer et al., 2008; Fischer et al., 2009)**. However, the adequate bond strength test for all-ceramic materials has not been determined yet.

In several studies, Aboushelib et al. have used the microtensile bond strength test method to evaluate the bond strength of a variety of zirconia-porcelain combinations **(Aboushelib et al., 2005; Aboushelib, de Kler,**

van der Zel, & Feilzer, 2008; Aboushelib et al., 2009; Aboushelib, Kleverlaan, & Feilzer, 2006, 2008a, 2008b).

Aboushelib et al, 2005, investigated the effect of finishing the core surface and CTE mismatch between the core and the veneering ceramic materials. The study showed that finishing of the core surface did not affect the bond strength between core and veneering ceramic materials. The study also showed that an experimental veneering ceramic with higher CTE compared to the CTE of the core, resulted in massive fractures in both the core and the veneering ceramics **(Aboushelib et al., 2005)**.

Guess et al. 2008, used Schmitz-Schulmeyer test to study the bond strength of three all ceramic systems and investigate the effect of thermocycling. Cercon Base, DC-Zirkon, and Vita InCeram and their manufacturer recommended veneering ceramic (Cercon CeramS, IPS e.max Ceram, Vita VM9), have been tested using Schmitz-Schulmeyer test. Half of each group was subjected to the thermocycling process prior to the test. The result showed that the effect of thermocycling on the shear bond strength of the test groups as well as on the control group was not statistically significant. Cercon Base/Cercon Ceram S showed combined fracture modes: cohesive in the veneer and adhesive at the core veneer interface 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. SEM analysis of the all-ceramic test groups revealed porosities in the veneering ceramic and structural defects at the zirconia veneer interface **(Petra C Guess et al., 2008)**.

Furthermore, Nakamura et al. 2009 used tensile bond strength test to examine the core-veneer bond strength of porcelain to sandblasted zirconia. Lava zirconia was sintered and then sandblasted with 70µ alumina powder at pressure of 0.2, 0.4 or 0.6 MPa. The zirconia then, was

veneered with 3 different veneering ceramic. The study resulted in that the specimens had a higher bond strength when sandblasted at 0.4 or 0.6 MPa than when blasted at 0.2 MPa. The conclusion was that sandblasting of the zirconia at 0.4 MPa pressure helps to develop a strong bond between zirconia and veneering
ceramic regardless the type of the veneering ceramic **(Nakamura et al., 2009).**

In addition, Kosyfaki et al. 2013, used Schwickerath test to evaluate the effect of thermocycling on the bond strength between Vita InCeram zirconia and 4 different veneering ceramic (IPS e.max Ceram, CerabienZR, Vintage ZR and VITA VM9). 10 specimens from each group were subjected to thermal cycling process. Schwickerath test was subjected to all specimens. The result showed that the thermal cycling did not affect the bond strength between the zirconia and the veneering ceramic **(Kosyfaki et al., 2013)**.

Several authors used shear bond strength test to evaluate the bond strength between different veneering ceramics and zirconia. Table 1 shows a summary of some of these studies.

Table 1: Summary of some studies used SBS test to evaluate the bond strength between veneering ceramic and zirconia.

Study	Materials		Shear Bond Strength (MPa)	Fracture mode
	Zirconia	Veneering Porcelain		
Almeida-Júnior, Longhini et al. 2013	Y-TZP	VM9	19.5	Adhesive 67% Mixed 33%
Mosharraf K. et.al 2011	Cercon base	Cercon ceram kiss	30.83	Adhesive 30% Mixed 70%
Saito et al. 2010	Katana ZrO_2	-Cerabian ZR -Cercon ceram kiss -IPS e.max -Vintag ZR -VM9	22.0 - 30.9	100% Cohesive
Özkurt Z. et.al 2010	-Zirkonzahn -Cercon -Lava -DC Zirkon	-Cercon Ceram -Lava Ceram -TriCeram -IPS e.max Ceram -VM9	18.66 - 40.49	Adhesive 50% Mixed 50%
Choi B. et.al 2009	Cercon base	Cercon ceram kiss	18.01 - 30.45	mixed
Fischer et al. 2008	Vita In-Ceram 2000 YZ Cubes	Cerabien ZR IPS e.max Triceram Vintage ZR VM9	23.5 (3.4) – 33.0 (6.8)	
Ashkanani, Raigrodski et al. 2008	Lava zirconia	Lava Ceram *Dry* *Thermocycling*	52.76 (13.75) 42.45 (12.63)	Adhesive 45% Mixed 55%
Aldohan H. et.al 2004	-DC Zirkon - Procera allZircon	-Vita D - Cerabien CZR	27.9 - 28.03	Cohesive 41-58%

The shear bond strength test is defined as a test in which two materials are connected via an adhesive agent and a shear load applied until separation occurs. The shear bond strength is calculated by dividing the maximum applied force by the bonded cross-sectional area.

Mosharraf et al. 2011 used shear bond strength test to evaluate the effect of different surface treatment and zirconia types on the bond strength between zirconia and veneering ceramic. Two types of zirconia have been used, the white and the colored zirconia. Three different surface treatment were applied to each zirconia type, 1. Sandblasting 2. Grinding 3. Sandblasting and liner application. All specimens then were veneered with Cercon Ceram Kiss. The zirconia type was found to have no effect on shear bond strength. Grinding, on the other hand, decreased dramatically the bond strength between the zirconia and veneering ceramic. Fracture analysis of the samples showed 30 % adhesive fracture and 5 % cohesive fracture and 65 % mixed fracture mode **(Mosharraf, Rismanchian, Savabi, & Ashtiani, 2011)**.

Fischer et al. 2009 used shear bond strength test to assess the effect of thermal misfit on shear strength between zirconia and veneering ceramic. 12 veneering ceramics were used, and to create a strong thermal mismatch, one of the veneering ceramic was intended to be used for metal core, and one for alumina core material. The coefficient of thermal expansion and the glass transition temperature were measured. There was no clear correlation between coefficient of thermal expansion and the shear bond strength. However, the results showed that the highest shear bond strength was observed when $\Delta\alpha\Delta T \approx 1000 \times 10^{-6}$ **(Fischer et al., 2009)**.

Fischer et al.2008 investigated the effect of different surface treatment on the bond strength between zirconia and veneering ceramic. The influence of polishing, sandblasting, silica coating, liner application, and regeneration firing were assessed using shear bond strength test. The authors concluded that there were no effect of surface roughening or liner application on the bond strength between zirconia and veneering ceramic. Electron microscope analysis revealed that the veneering ceramic remained on the zirconia surface for all specimens indicating that the bond strength between the zirconia and veneering ceramic is higher than the cohesive strength of the veneering ceramic. This failure result could be caused by the stress that was generated and reached its high level near the interface, due to a difference in the coefficients of thermal expansion between zirconia and veneering ceramics **(Fischer et al., 2008)**.

1.4 Ceramic failure

The failures of dental ceramic structures are often multifactorial and can be associated with atypical crown and bridge designs, thermal incompatibility stress in metal-ceramic and ceramic-ceramic (layered) systems, the presence of critical structural flaws, and non-standardized processing techniques **(Anusavice et al., 1989).**

The major problem of zirconia based restorations is the chipping of veneering porcelain **(AL-AMLEH, Lyons, & Swain, 2010).**

Ceramic production without any surface flaws is not possible, but it is possible to strengthen the ceramic by inducing the residual stress within the surface **(P. DeHoff, Anusavice, & Vontivillu, 1996)**. During manufacturing of ceramic the created tensile and compressive residual stresses may affect the ceramic prostheses as a direct influence on contact induced crack propagation resistance. In another hand, residual stresses can be tailored to increase the strength of the ceramic. The cooling rate of the ceramic, the mismatch in thermal coefficients of expansion in the components of a ceramic composite, and the thickness of the veneering porcelain are the major causes of the residual stresses production **(Belli et al., 2012; Swain, 2009; Taskonak et al., 2008).**

In the previous studies that tested the bond strength between veneering ceramics and zirconia, 3 fracture pattern have been recognized:

1- Adhesive fracture, which known as complete delamination of the veneering ceramic from the zirconia surface. Adhesive fracture mode does not occur in the presence of a good bond strength between veneering ceramic and core material **(Al-Dohan, Yaman, Dennison, Razzoog, & Lang, 2004)**
2- Cohesive fracture, in which the veneering ceramic remains on the zirconia core after the fracture. This fracture indicating that the

bonds strength between veneering ceramic and zirconia core is higher than the cohesive strength of the veneering ceramic **(Fischer et al., 2009)**

3- Combined fracture, in which the tested specimen revealed adhesive fracture in the interface and cohesive fracture in the veneering ceramic. this fracture could be explained by the high resistance of the zirconia that leads to crack deflection toward the interface area **(Petra C Guess et al., 2008).**

1.5 Residual Stress in Dental Ceramic Restorations

Residual stresses in brittle materials can be a major factor in the improvement of the strength and apparent fracture toughness of bilayer ceramic composites as well as the material selection and geometric design. Therefore, it is important to determine the magnitude and distribution of residual stresses **(Taskonak et al., 2008).**
When a glass is heated, it does not show a discrete solid-liquid transition as the non-crystalline material. Instead, what happens is that at some point there is an increase in the rate of change of the specific volume. The temperature at which this change in the slope of the specific volume occurs is known as the *Glass Transition Temperature, Tg* **(van Noort, 2007).**

Below Tg, the ceramic has the properties of a solid and it may develop the stress. Above Tg, it flows more readily, and it cannot shows any stress because the thermal misfit between the veneering ceramics and the core material is compensated by plastic flow **(Fischer, Stawarczyk, Tomic, Strub, & Haemmerle, 2007; O'Brien, 2008).** Residual stresses, which are created during cooling phase of ceramic, could be tensile or compressive residual stresses. There are three main influencing factors that determine the type of the residual stresses in the ceramics, these factors are the coefficient of thermal expansion (CTE), the cooling rate and the thickness of the veneering porcelain. Swain (2009) has concluded that the thick layer of the veneering porcelain on framework with low thermal diffusivity is more susceptible to generate a high tensile residual stresses **(Swain, 2009)**. Thermal tempering is a process of heating the glass to a critical temperature and then rapidly quenching it. This technique has been used to strengthen the glass by inducing the compressive residual stress **(P. DeHoff et al., 1996).**

Tensile residual stresses are generated by slow cooling rate, thick veneering porcelain and when the CTE of veneering porcelain is greater than that of the core material. Compressive residual stresses are generated by fast cooling, reducing the thickness of the veneering porcelain and when the CTE of the veneering porcelain is lower than that of the core material **(Fischer et al., 2007; Swain, 2009; Taskonak et al., 2008).**

1.6 Coefficient of Thermal Expansion (CTE)

Coefficient of thermal expansion of any material can be defined as the frictional increase in length per unit rise in temperature **(James, Spittle, Brown, & Evans, 2001).** This change is so small that it is usually expressed in terms of parts per million per degree Centigrade (ppm/°C) **(Richard Van Noort 2007)**. It is calculated as follows equation:

$$CTE\ (\alpha) = (L - L_o) / L_o (T - T_o)$$

Where L is the final length of the material after heating, L_o is the original length, T is the final temperature, and T_o is the starting temperature.

During the processing of ceramics, residual tensile stress is produced as a result of the contraction thermal mismatch, that could lead to ceramic failure **(P. H. DeHoff, Barrett, Lee, & Anusavice, 2008).**

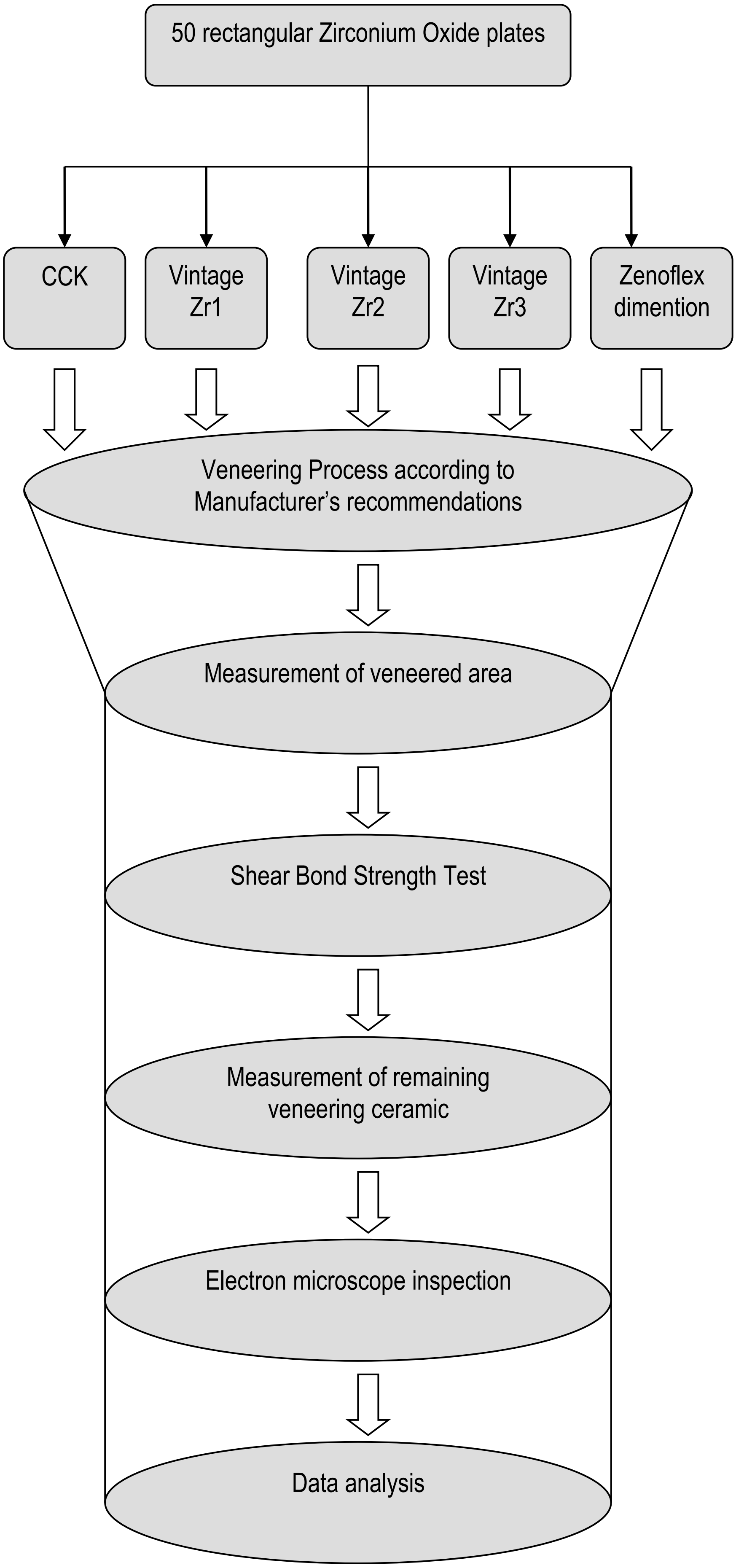
Several studies recommended that the CTE of veneering ceramic should be lower than that of ceramic core. This recommendation will help in establishing a strong bond between the core and the veneering ceramics **(Anusavice et al., 1989; Coffey et al., 1988; Fischer et al., 2009).** Kim in 2005 has illustrated that, if the CTE mismatch between the veneering ceramic and the zirconia-core material is about $5x10^{-6}$/°C the tensile stresses on the zirconia could be more than 150 MPa and that may lead to crack growth and spontaneous failure **(Kim, Bhowmick, Hermann, & Lawn, 2006)**. Furthermore, DeHoff in 2009 concluded that, ceramic systems with CTE mismatch greater than $1.0x10^{-6}$/°C have higher tendency to the failure in clinical use compared with systems having smaller CTE mismatch between the core and the veneering ceramics **(P. H. DeHoff & Anusavice, 2009).** In contrast, Fischer et al. 2009 Investigated the shear bond strength of 12 veneering ceramic to zirconia by using the shear test, and they found that there was no clear correlation between the CTE mismatch and the shear bond strength **(Fischer et al., 2009).**

2 Aim of the Study

Aim of this study was to compare the shear bond strength of five veneering ceramics with different values of coefficient of thermal expansion by using shear bond test according to DIN EN ISO 10477, and to analyze the fracture mode visually and under electron microscope. 3 of those veneering ceramics are experimental ceramics.

The null hypothesis were that in the bi-layered all ceramic systems, the smaller CTE mismatch between the veneering ceramic and the zirconia substrate, the higher will be the bond strength. Figure 2 illustrates the outline of the study.

Figure 2: Study outlines



3 Materials and Methods

3.1 Zirconium oxide

The type of Zirconium oxide used in this study is Zenotec Zr Bridge (Wieland Dental+Technik, Pforzheim, Germany). Table 3.1 shows the properties of Zenotec Zr Bridge zirconia.

Table 3.1: Properties of Zenotec Zr Bridge according to data sheet.

Components	Zirconiumoxide (Zr_2O+HfO_2) 94% Yttriumoxide (Y_2O_3) 5% Aluminiumoxide (Al_2O_3) <1% Other oxides <1%
Vicker's Hardness	1300 HV10
Elasticity Module	210 GPa
Flexural strength	1100 MPa
CTE	$10.5*10^{-6}K^{-1}$

50 rectangular plates of white zirconia have been designed, manufactured, and provided by the company Wieland in pre-sinterd white zirconia blank. The specimens then were trimmed from the blank, and the measurements were prepared with calculating the shrinkage factor to compensate the shrinkage after the final sintering process. Firing protocols have been carried out according to the manufacturers' recommendations (Table 3.2) in Vita Zyrcomat furnace (Vita Zahnfabrik, Bad Säckingen, Germany) (Figure 3.1).

After the final sintering the specimens had a size of 20x10x1.5 mm (Figure 3.2). They were cleaned by steam water and divided equally and randomly into 5 groups.

Table 3.2: Zirconium oxide firing schedule according to manufacturer's recommendations.

Base temperature	20°C
Drying time	1 hour
Vacuum start	1530°C
Vacuum end	1530°C
Holding time during sinter process	2 hour
Final temperature	400°C
Slow cooling time	3 hour

Figure 3.1: Vita Zyrcomat furnace (Vita Zahnfabrik, Bad Säckingen, Germany).

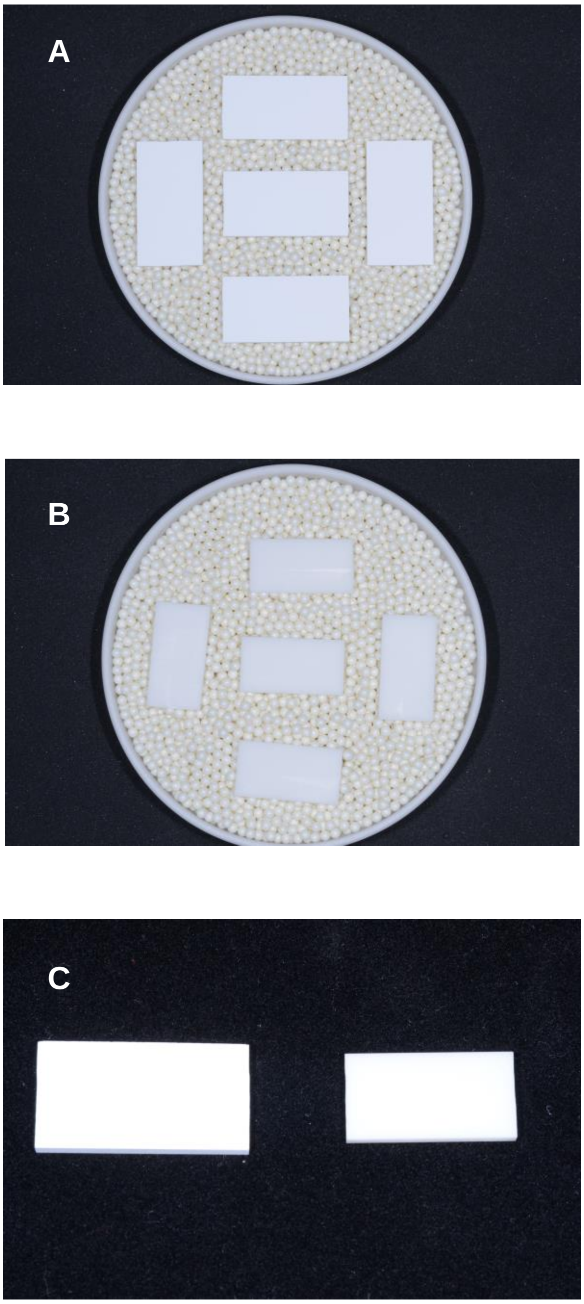


Figure 3.2: A) Ziroconia plates before sintering process. B) Zirconia plates after sintering. C) Size of zirconia plate before and after sintering.

3.2 Veneering Ceramic

5 different veneering ceramics have been used in this study. Each has a different CTE value as shown in table 3.3.

Table 3.3. CTE values of veneering ceramics used in this study at 25-500°C.

Company	Ceramic	CTE (ppm/°C)	Δσ (ppm/°C)
Wieland Dental+Technik, Pforzheim, Germany	Zenoflex dimention	9.4	1.1
DeguDent, Hanau-Wolfgang, Germany	Cercon ceram kiss (CCK)	9.2	1.3
Shofu JNC , Kyoto, Japan	Vintage Zr 1	9.0	1.5
Shofu JNC , Kyoto, Japan	Vintage Zr 2	9.5	1.0
Shofu JNC , Kyoto, Japan	Vintage Zr 3	10.0	0.5

3.3 Fabrication of the specimens

The preparation process of the veneering layer has been done through 5 steps: 1st, 2nd Liner, 1st, 2nd Dentine and Glazing. For all veneering ceramic types, 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 3.4). 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 liner was applied and fired under the same condition to achieve a continuous thin layer of the liner (Figure 3.3 A). For dentin layer, a stainless steel mold was placed on the zirconia plates where clearance of 5 mm diameter and 3 mm height was available above the zirconia plates.

The inner surface of the mold was isolated with isolating fluid (Carat, Hagar und 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 common dental lab. The slurry mixture was then poured to the mold (Figure 3.3 B) and the excess liquid was absorbed with tissue paper. After applying the dentin layer, the mold was carefully removed (Figure 3.3 C) and the non-sintered specimens were then fired according to the manufacturer’s recommendations (Table 3.4). Under the same conditions, a second dentin layer was added and fired to compensate the shrinkage of the sintering process and to establish the correct diameter and thickness of the specimens (Figure 3.3 D). Finally, a glaze firing was carried out according to the manufacturer’s recommendations (Table 3.4). Figure 3.3 illustrated briefly the process of specimens' fabrication and the final sample shape before testing.

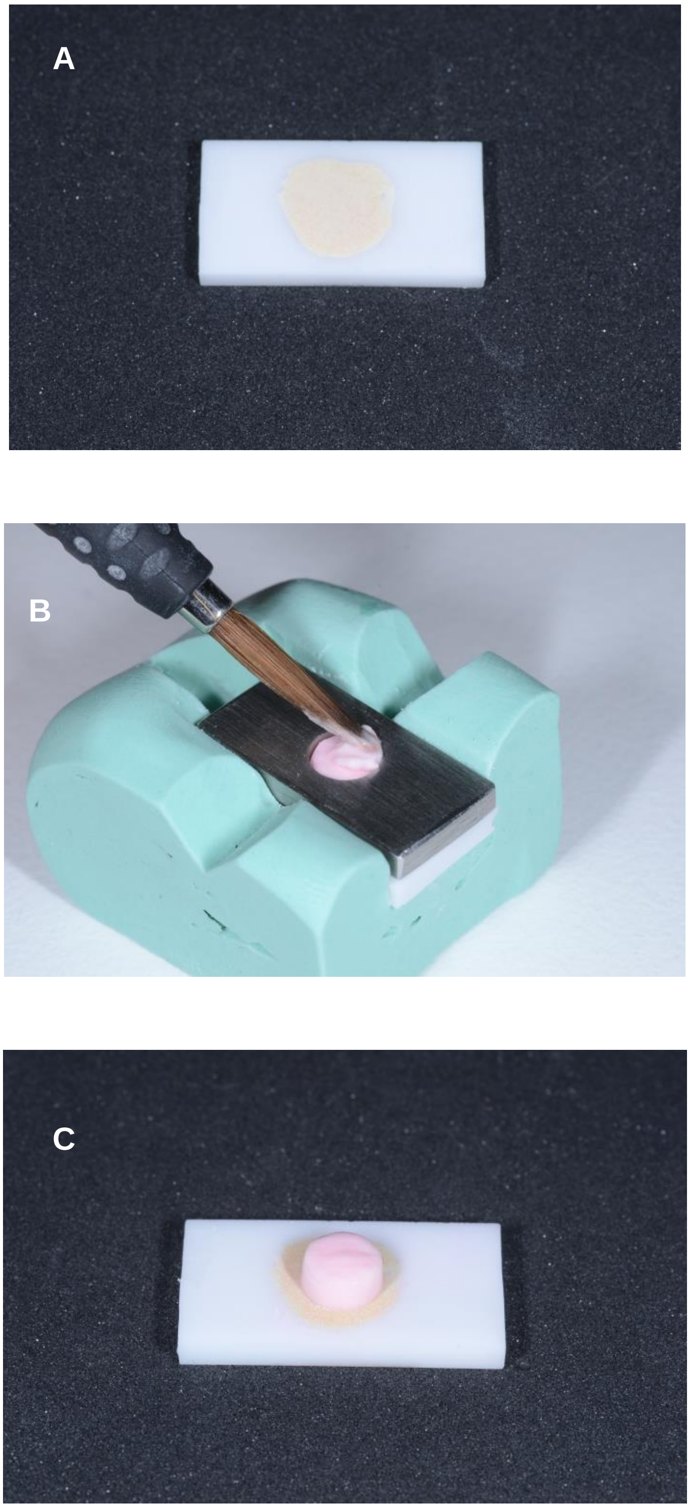


Figure 3.3: A) application of first and second liner. B) veneering ceramic application. C) veneering ceramic before sintering process.

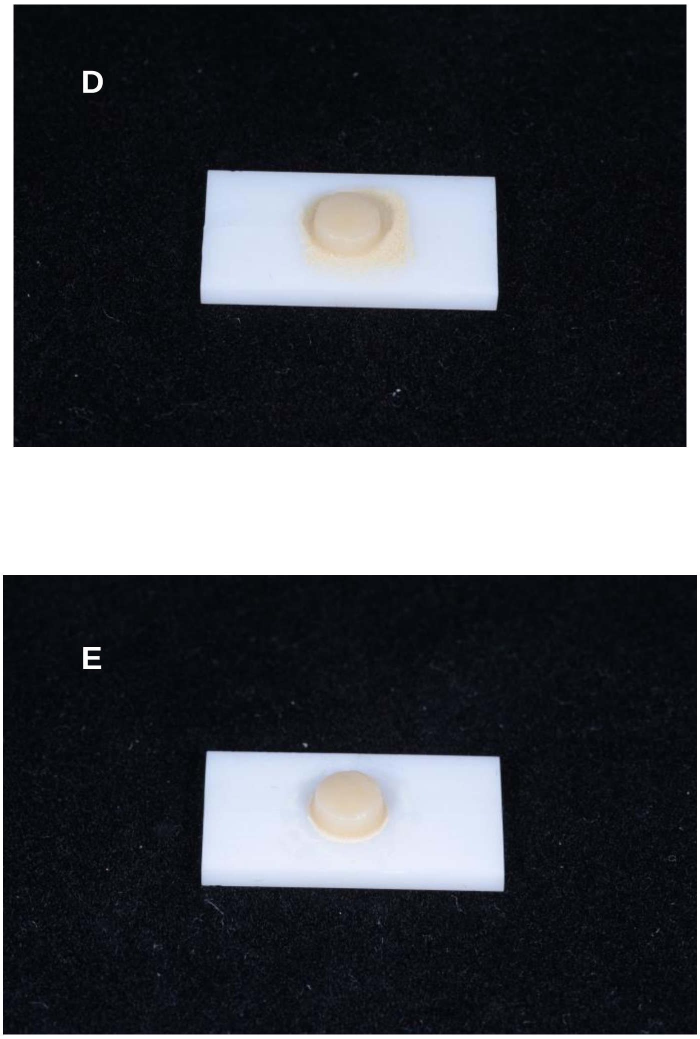


Figure 3.3: D) specimen shape after applying and firing the second dentin layer. E) the final shape of the specimen before testing.

Table 3.4: Veneering ceramics firing schedule according to manufacturer's recommendations.

veneering ceramic	**Temperature (°C)**	**Time (min)**	**Heating Rate (°C/min)**	**Firing Temp. (°C)**	**Holding Time (min)**
1st/ 2nd Liner					
Zenoflex	575	2	45	930	1
Ceram kiss	575	6	55	970/960	1
Vintage ZR1, 2, 3	500	7:30	45	930	1
1st Dentin					
Zenoflex	575	3	45	900	2
Ceram kiss	450	2	55	830	1:30
Vintage ZR1, 2, 3	650	5:30	45	910	1
2nd Dentin					
Zenoflex	575	2	45	890	1
Ceram kiss	450	2	55	820	1:30
Vintage ZR1, 2, 3	650	5:30	45	900	1
Glazing					
Zenoflex	575	1	45	880	1
Ceram kiss	450	0	55	800	1
Vintage ZR1, 2, 3	600	6	55	860	0

3.4 Measuring the veneered area

Prior to the shear bond strength, the veneered area of all specimens were examined with Photomakroskop M400 (Wild Heerbrugg, Gais, Switzerland) (Figure 3.4) and measured in (mm^2) using software Image Pro. Plus v.6 (Media Cybernetics, Washington, USA).

Figure 3.4: Photomakroskop M400 (Wild Heerbrugg, Gais, Switzerland)

3.5 Shear Bond Test (SBS)

The completed spacimens were fixed in a special sample holder and placed in a universal testing machine Z010 (Zwick, Ulm, Germany) (Figure 3.5). The specimens were then loaded with a shear force applied as close as possible to the interface between the veneering ceramic and the zirconia. The shear force was applied with crosshead speed of 1 mm/min untill the fracture occurred. The load at fracture was recorded in Newton (N). For each group, the mean bond strength (MPa) was calculated through dividing the load at fracture (N) by the bonding area (mm^2).

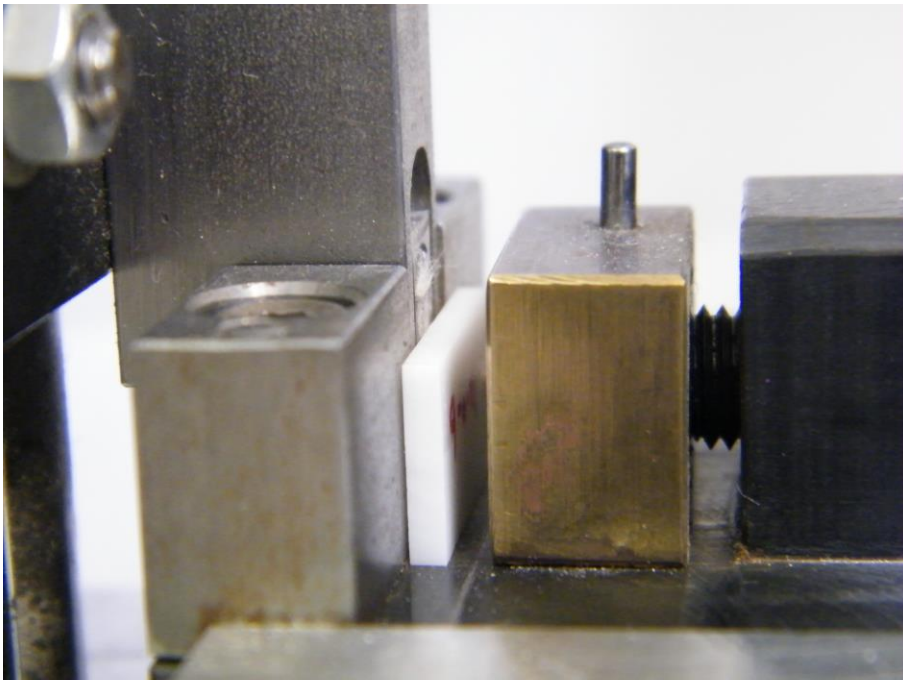


Figure 3.5: Universal testing machine Z010 (Zwick, Ulm, Germany)

3.6 Measuring the remaining veneered area

After the SBS test, the specimens were examined with Photomakroskop M400 (Wild Heerbrugg, Gais, Switzerland) (Figure 3.4) and the remaining veneered area of each specimen was measured in (mm^2) using software Image Pro. Plus v.6 (Media Cybernetics, Washington, USA).

3.7 Determining the fracture mode

After measuring the remaining veneered 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 mode:

a) Less than 20% ➔ Adhesive fracture within the veneering ceramic.
b) Between 20% and 80% ➔ Mixed fracture.
c) More than 80% ➔ Cohesive fracture

3.8 Analysis of the fractured area under scanning electron microscope

After calculating the bond strength of all the specimens in each group, 3 specimens were selected from each group. The main criteria of the selection was the bond strength value of the specimens. The specimens with the highest, middle, and lowest bond strength value in every group, were inspected under scanning electron microscope (SEM) (Leo 1430, Zeiss, Oberkochen, Germany) (Figure 3.6) at 30, 100, and 1000 magnification, and photo documented. The aim was to study and analyze the fractured surface.

3.9 Statistical analysis

Statistical analysis was carried out by using Microsoft Excel 2002 software (Microsoft Corporation, WA, USA). The data were analyzed by using a one-way analysis of variance test (ANOVA) to determine whether significant differences existed between the shear strengths of the 5 groups at ($P< 0.05$). Also, a Tukey multiple comparisons test at ($P< 0.05$) was used to assess the differences among the specified materials.

4 Results

4.1 Shear bond strength

The mean shear bond strength of all 5 veneering ceramics are presented in table 4.1 and graphically in figure 4.1.

The highest mean shear bond strength was recorded for Vintage Zr2 (37.64 ± 10.44 MPa) followed by Vintage Zr3 (31.32 ± 5.6 MPa). The mean shear bond strength of Vintage Zr1 and Zenoflex dimension were (30.96 ± 7.82 MPa) and (27.61 ± 5.56 MPa) respectively. The lowest mean shear bond strength was recorded for Cercon Ceram Kiss (26.44 ± 5.5 MPa).

One way ANOVA revealed a significant difference in the shear bond strength among the five tested veneering ceramics at $P<0.05$. The Tukey HSD comparison test was used to make all pair comparison of mean shear bond strength of the 5 groups. The result of this comparison are presented in table 4.1.

The *P* values of the different comparison show that Zenoflex dimension, CCK, Vintage Zr1, and Vintage Zr3 were not significantly different. Vintage Zr2 had significantly higher mean bond strength than Zenoflex dimension and CCK groups.

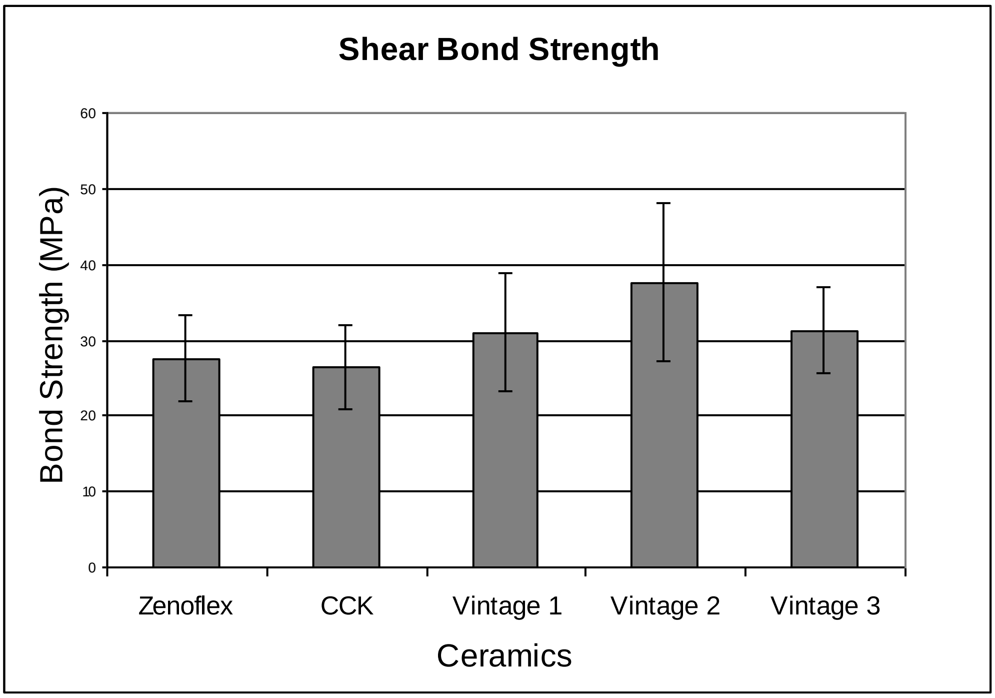


Figure 4.1: Mean shear bond strength of the 5 tested veneering ceramics. The vertical lines shows the standard deviation for each veneering ceramic.

Table 4.1: mean shear bond strength, standard deviation, and significance of all the tested veneering ceramics.

Veneering Ceramic	SBS mean (MPa)	SD	Sign.*
Zenoflex dimen.	27.61	± 5.56	b
CCK	26.44	± 5.5	b
Vintage Zr1	30.96	± 7.82	a b
Vintage Zr2	37.64	± 10.44	a
Vintage Zr3	31.32	± 5.6	a b

* Values with the same letter are not statistically different using Tukey test at $P<0.05$.

4.2 Evaluation of fracture mode

The results of fracture mode analysis of the specimens are summarized in table 4.2. In general, 76% of all specimens demonstrated mixed (cohesive and adhesive) fracture mode, and 34% fractured cohesively. All the specimens fractured within the veneering ceramic.

All of the groups demonstrated either cohesive or mixed fracture mode within the veneering ceramic (Figure 4.2). in Vintage Zr1 and Vintage Zr2 groups, 9 specimens from each group demonstrated a mixed failure mode while one specimen failed cohesively from each group. In Vintage Zr3 and CCK groups, 6 specimens (of each group) demonstrated mixed fracture mode and 4 specimens failed cohesively. In Zenoflex group, 8 specimens exhibited mixed fracture mode and 2 specimens failed cohesively.

Table 4.2: Fracture mode of the all tested veneering ceramics.

	Cohesive	Mixed	adhesive
Zenoflex	2	8	0
CCK	4	6	0
Vintage Zr1	1	9	0
Vintage Zr2	1	9	0
Vintage Zr3	4	6	0

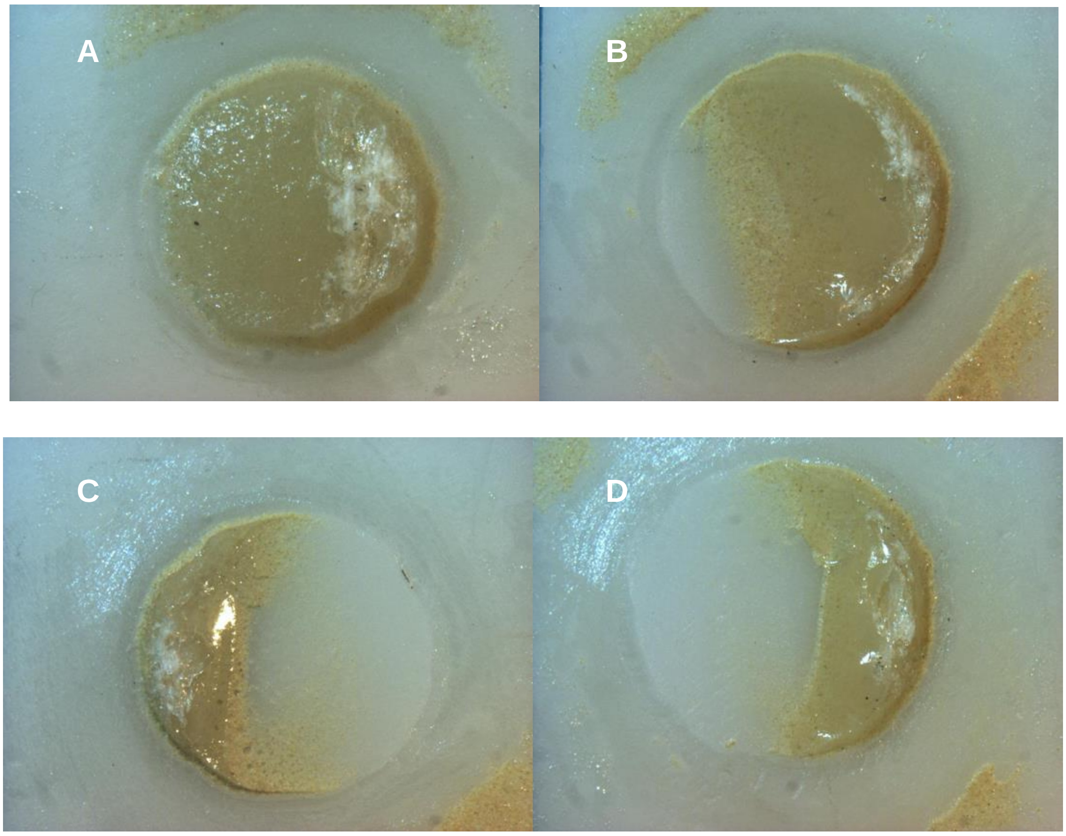


Figure 4.2: A and B cohesive fracture mode. C and D mixed fracture mode.

4.3 Scanning electron microscope (SEM)

Figure 4.3 demonstrates the zirconia plate, veneering ceramic, and the fracture surface.

Generally, SEM images of the examined specimens are characterized by several inherent defect (porosity) (Figure 4.4, 4.5), irregular crack lines (Figure 4.4, 4.7) and detached veneering ceramic crystalline (Figure 4.6).

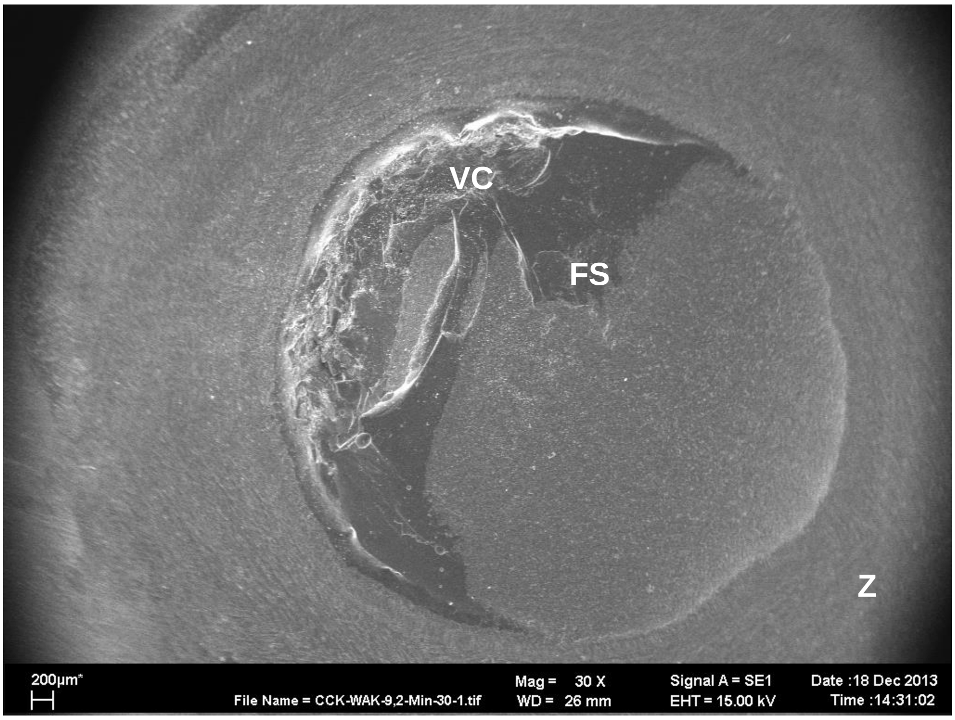


Figure 4.3: (30 magnification) SEM image (VC) is the veneering ceramic, (z) is the zirconia plate and (FS) is the fracture surface.

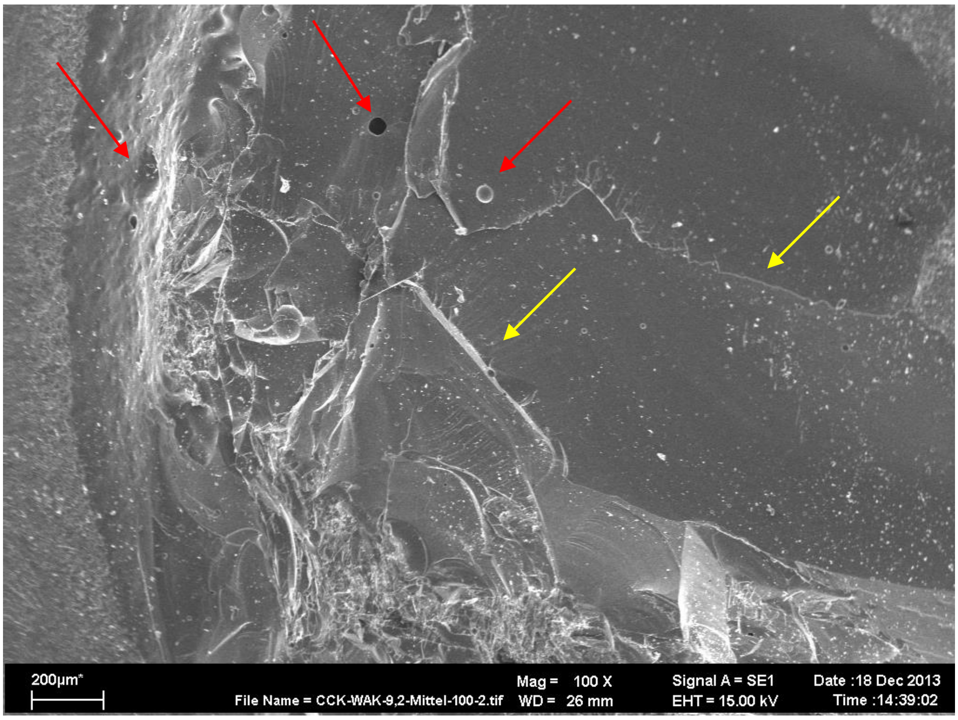


Figure 4.4: (100 magnification) SEM image of a fractured surface, yellow arrows point at the fracture lines, and red arrows point at defects in the veneering ceramic.

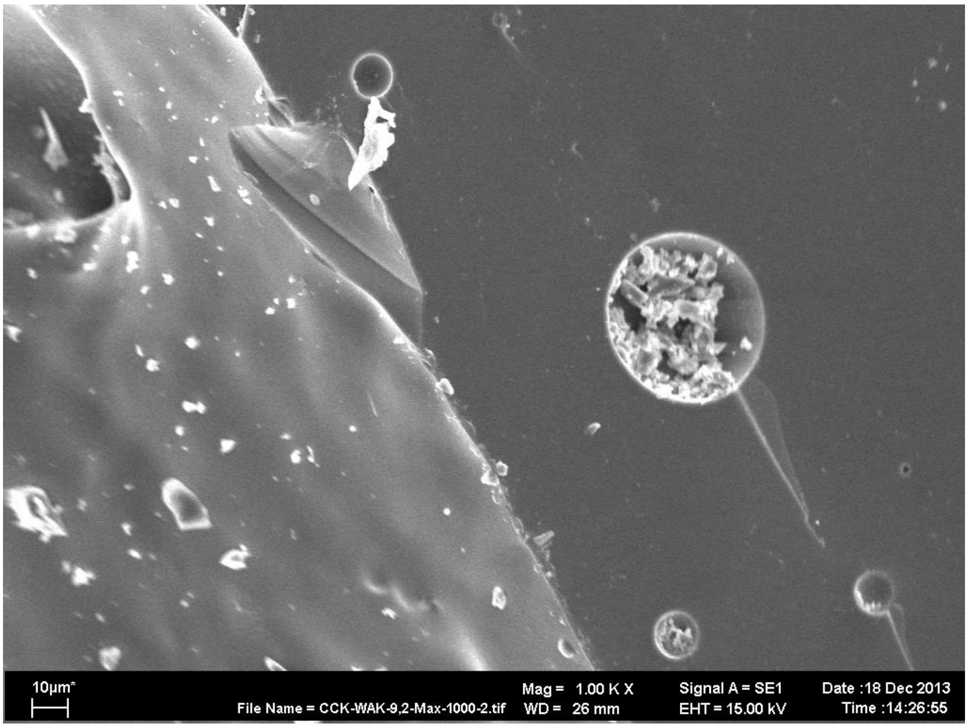


Figure 4.5: (1000 magnification) SEM image shows the pores in the veneering ceramic.

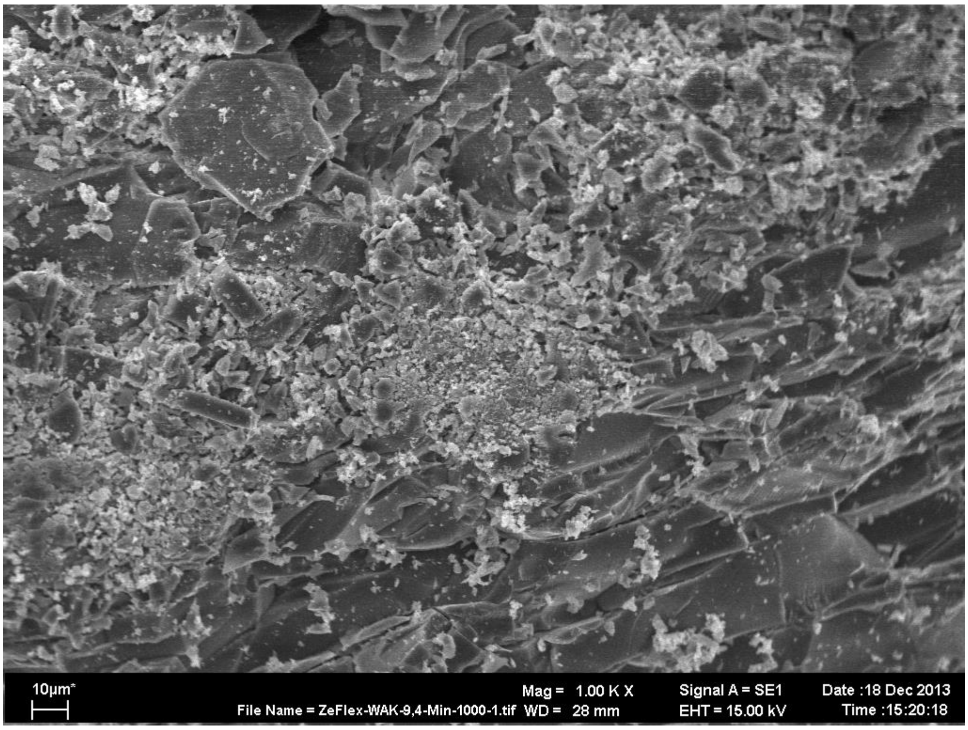


Figure 4.6: (1000 magnification) SEM image of fracture surface shows deattached crystalline of the veneering ceramic.

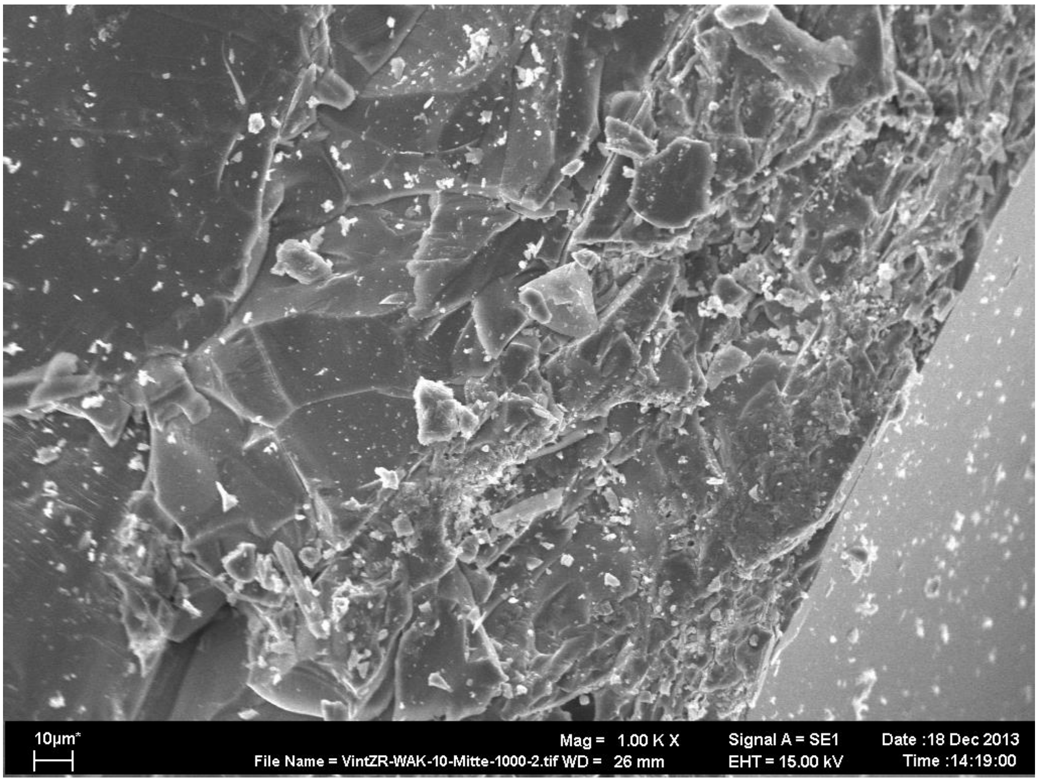


Figure 4.7: (1000 magnification) SEM image shows irregular fracture lines

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5 Discussion

The present study investigated the effect of CTE mismatch between the veneering ceramic and zirconia substrate. Based on the results obtained from this study, the null hypothesis was rejected.

The International Organization of Standardization has standardized the bond strength measurement for metal ceramics systems, through Schwickerath initiation crack test and determined that the mean bond strength of metal ceramic systems should be greater than 25 MPa to meet the ISO 9693 requirements **(ISO-Standard, 1999).** 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. These include, three and four point loading test **(White, Miklus, McLaren, Lang, & Caputo, 2005)**, biaxial flexural strength test **(Yilmaz et al., 2011)**, Schwickerath test **(Kosyfaki et al., 2013; Schille et al., 2012)**, microtensile bond strength test **(Aboushelib et al., 2005; Aboushelib et al., 2009; Aboushelib, Kleverlaan, et al., 2008b)**, and shear bond strength test **(Al-Dohan et al., 2004; Almeida-Júnior, Longhini, Domingues, Santos, & Adabo, 2013; Ashkanani, Raigrodski, Flinn, Heindl, & Mancl, 2008; Petra C Guess et al., 2008; Mosharraf et al., 2011)**. Each test has advantages and disadvantages. Shear bond strength test is defined as a test in which 2 materials are connected by an adhesive agent and loaded in shear until separation occurs. The bond strength is calculated by dividing the maximum applied force in Newton by the loaded area in mm^2 **(Craig & Powers, 2002).** The reason of using shear bond strength test in this study was its simplicity compared with other test methods **(Oilo, 1993)**. Ease of specimens' preparation, clear protocol, and rapid result production 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 from specimen geometry **(Choi, Han, Yang, Lee, & Kim, 2009; Özkurt, Kazazoglu, & Ünal, 2010)**. Therefore, the standardization of shear bond strength test methodology is important for improving the clinical usefulness of SBS test. Standardization of the methodology should include storage conditions, type of substrate, specimen preparation, rate of load application, cross-sectional surface area, and experience of the researcher **(Al-Dohan et al., 2004; Choi et al., 2009; Rismanchian, Shafiei, Askari, & Khodaeian, 2012)**. In the present study, the shear bond strength test was prepared and performed according to the ISO 10477 standards of the shear bond test for metal-resin **(ISO-Standards, 2004)**

Residual stress considered as one of the factors that could affect the bond strength between the core and veneering ceramic in all-ceramic restoration **(Fischer et al., 2008)**. The major causes of the residual stress production are thickness of the veneering ceramic, cooling rate, and the mismatch of the coefficient thermal expansion between the zirconia and the veneering ceramics **(Taskonak, Borges et al. 2008, Swain 2009, Belli, Monteiro et al. 2012).** In this study the bond strength of 5 veneering ceramic with different CTE values have been tested. Generally, all the 5 veneering ceramics exhibited bond strength in the range of 26.44 - 37.64 MPa which is consistent with most of the previous shear bond strength studies (Table 1). The mean bond strength of Cercon Ceram Kiss obtained from this study is 26.44 MPa. This value is higher than that reported by Ozkurt **(Özkurt et al., 2010),** and Saito **(Saito et al., 2010)**, which were 20.19, and 22.0 MPa respectively. The different between the present study and the other two studies could be explained by the effect of thermocycling. According to most studies on the bond strength, the actual bond strength would decrease further with thermocycling **(Choi et al., 2009)**. In the present study the specimens were tested in a dry environment, while in Ozkurt et al. study and Saito et al. study they stored all the specimens in distilled water at 37ºC for 24 hours prior to testing

(Özkurt et al., 2010; Saito, Komine, Blatz, & Matsumura, 2010). In a study by Aboushelib et al. Cercon ceram kiss exhibited mean bond strength of 36.6 MPa which was higher than the 26.44 MPa obtained in the present study and the mean bond strength reported by Ozkurt et al. Two reasons 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 **(Aboushelib, de Kler, et al., 2008; Özkurt et al., 2010)**. No study could be found to evaluate the bond strength of Zenoflex veneering ceramic or the experimental veneering ceramics Vintage Zr1, Vintage Zr2, or Vintage Zr3.

In general, CTE of the veneering ceramic must be lower than that of the core materials, to generate a favorable compressive stress in the veneering ceramic during cooling process. For metal ceramic system, it is generally accepted that the CTE of the veneering ceramic is about 10 % lower than that of the metal core material **(Fischer et al., 2007).** For all ceramic systems, Dehoff et al. suggested a positive mismatch equal or less than 1 x 10^{-6} between veneering ceramic and core material **(P. H. DeHoff & Anusavice, 2009**). In the present study, however, the three experimental veneering ceramics (Vintage ZR 1, 2, 3) showed higher mean bond strength than Zenoflex and CCK veneering ceramics. The CTE mismatch of Vintage ZR2, Zenoflex, and CCK are 1.0, 1.1, 1.3 ppm/°C respectively. Although the difference in CTE mismatch of those 3 veneering was not significantly high, the mean bond strength of the Vintage ZR 2 showed significantly higher mean bond strength than Zenoflex and CCK veneering ceramics, considering the fact that Zenoflex is the respective veneering ceramic of the zirconia used in the present study. Therefore, we can state that the CTE mismatch in the range of 0.5 - 1.5 ppm/°C does not have a major effect on the bond strength between the veneering ceramic and zirconia substrate. This observation is consistent with the other studies **(Fischer et al., 2009; Petra C Guess et**

al., 2008; Saito et al., 2010). The other properties of the veneering ceramic, such as modulus elasticity and Passion's ratio, may have an effect on the bond strength. It should be taken in consideration that the CTE of ceramic is nonlinear, so, the best way to describe the thermal behavior of ceramic is not by ISO standard (**Isgro et al. 2004**).

Mainly, the specimens showed combined fracture mode as adhesive in the interface and cohesive in the veneering ceramics (Table 4.2). This finding is consistent with previous studies **(Al-Dohan et al., 2004; Petra C Guess et al., 2008; Mosharraf et al., 2011; Özkurt et al., 2010)**. Combined fracture mode could be explained by the deflection of the crack due to superior ability of zirconia to resist crack propagation or as a result of poor bonding between veneering ceramic and zirconia substrate **(Petra C Guess et al., 2008)**. The absence of the adhesive fracture mode in the present study could be explained by the finding of Smith et al. study. They found a chemical alteration of the veneering ceramic adjacent to core substrate, which might lead to changing the physical properties at the interface (e.g strength or CTE). They interpreted the occurrence of the chemical alteration through 2 ways: First, by diffusion of element, unique to the veneering ceramic or core material, across the interface. Second by developing a layer of excess infiltration glass on the core surface during processing **(Smith, Kelly, & Tesk, 1994)**. 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 **(Fischer et al., 2009).**

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 which acted as a stress concentrators. Aboshelib et al. 2008 found porosities free veneer after SEM examination of zirconia press-on veneer interface **(Aboushelib, Kleverlaan, et al., 2008b)**. They found that the mean bond strength of the CCK is 36.6 MPa which is higher than that obtained in the present study. Therefore, veneering technique may play a role in the presence of pores within veneering ceramic and affect the mean bond strength of tested specimens.

Conclusions

Within the limitation of this in vitro study, the following conclusion can be drawn:

1- CTE mismatch in the range of 0.5 -1.5 ppm/°C does not have a major effect on the bond strength between the veneering ceramic and zirconia substrate.

2- Vintage ZR 2 has significantly higher mean bond strength than CCK and Zenoflex veneering ceramics when the zirconia core material is Zenotec Zr Bridge.

3- The experimental veneering ceramics (Vintage ZR 1, 2, 3) revealed higher bond strength than the commercial veneering ceramics tested in the present study.

4- Surface analysis of the fracture surface revealed mainly combined and cohesive fracture mode and no adhesive fracture were observed.

5- Shear bond strength test according to ISO 10477 is a simple test and can be used for all ceramic bi-layer system.

6 Abstract

Statement of problem: In all-ceramic restorations, high occurrence of veneer chip-off has been reported in clinical studies. CTE mismatch is one of the factors that may increase the residual stress in the interface and affect the veneer-core bond strength.

Purpose: The purpose of this study was to evaluate the effect of CTE mismatch on the bond strength of 5 veneering ceramics.

Material and methods: 50 rectangular zirconia plates (Zenotec ZR) were divided equally and veneered with 5 different veneering ceramics (Zenoflex, Cercon ceram kiss, Vintage ZR 1, Vintage ZR 2, and Vintage ZR 3). The bond strength test was performed according to ISO 10477 and the shear force was subjected by using universal testing machine. Fracture surfaces were examined under macroscope and SEM to determine the fracture mode. The collected data were analyzed with a 1-way ANOVA and Tukey HSD test (α=.05).

Results: The mean shear bond strength in MPa (± SD) were 27.61 (± 5.56) for Zenoflex, 26.44 (± 5.5) for Cercon ceram kiss, 30.96 (± 7.82) for Vintage ZR 1, 37.64 (± 10.44) for Vintage ZR 2, and 31.32 (± 5.6) for Vintage ZR 3. Vintage ZR 2 had significantly higher mean bond strength than Zenoflex and Cercon ceram kiss. All groups exhibited either combined or cohesive fracture modes.

Conclusion: CTE mismatch in the range of 0.5 -1.5 ppm/°C does not have a major effect on the bond strength between the veneering ceramic and zirconia substrate.

Key words: Shear bond strength, CTE mismatch, Fracture mode, veneering ceramics.

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