

Effect of surface treatments and repeated firings on bond strength of zirconia and veneer ceramics

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Abstract

Zirconium oxide based restorations have become one of the most preferred treatment options, but no standard method has been established for optimal adhesion between zirconia and veneering ceramics. The aim of this study was to investigate the effect of surface treatments and repeated firings on shear bond strength (SBS) of veneer ceramics to zirconia. 120 cylindrical specimens (7 mm x 2 mm) prepared from Kavo Everest ZS block and divided into three groups: group SB (sandblasting); group SBL (liner application with sandblasting); group C (control group with no surface treatment). Veneer ceramic was applied by pressing and layering. Repeated firing treatment was performed. SBS test was performed and surfaces were analyzed with SEM. ANOVA indicates that in group SB with pressing technique ($32,23 \pm 7,90$ MPa) had higher value than in group SB with layering technique ($22,87 \pm 8,21$ MPa). Liner application has negative effect on clinical success of restoration. In the limitation of this study, sand-blasting treatment with pressing technique is more efficient in improving core-veneer bond strength compared to liner application

Key words: Zirconium dioxide, bond strength, surface treatment, veneering porcelain.

1. Introduction

Despite the advantages of all-ceramic systems, such as their aesthetic properties, biocompatibility and color stability, the brittle nature of these materials has led to the increasing utilization of zirconia-based ceramics in recent years (Drummond, 2000). Zirconia-based ceramics exhibit high

mechanical performance, durability and resistance, increasing the reliability of all-ceramic restorations (Guazzato, 2004a; Tinchert, 2000). The most frequently used type of dental zirconium oxide (ZrO_2) is yttria tetragonal zirconia polycrystal (Y-TZP), which provides durability due to the excellent proportionality of transformation from the tetragonal to the monoclinic phase. Y-TZP is a fully tetragonal, fine-grained ZrO_2 ceramic composed entirely of small, metastable, tetragonal grains after the addition of ~3 mol% yttrium oxide (Y_2O_3) as a stabilizer (Denry, 2008).

To enhance the aesthetic properties of restorations, opaque ZrO_2 cores are veneered with specially developed ceramic materials (Aboushelib, 2008a). Surface and firing treatments applied during production and veneering treatments, affect the mechanical properties of the core material. These properties are not the sole factors affecting restoration success; weak veneer ceramics may negatively affect the clinical success of a durable core material, potentially leading to failures such as fracture in clinical practice (Dundar, 2007).

The most common failure in two-layer all-ceramic systems is veneer ceramic delamination (layered separation from the core). Delamination may occur due to patient-dependent factors, dynamic loads induced by premature contact, occlusal instability, inadequate bond strength, restoration geometry, material properties, material fatigue, inadequate core support and incompatibility of heat expansion coefficients of the core and veneer (White, 2005).

Although ZrO_2 cores are highly resistant to fracture, veneer fracture poses a problem (Donovan, 2005). Long-term clinical studies have demonstrated that ZrO_2 core-veneer ceramic interface is the weakest structural component and that this bond is affected by the sensitivity of core surface finishing treatments, veneer ceramic application method and repeated firing treatments (Aboushelib, 2005). The purposes of surface treatment are to clean the ZrO_2 surface, increase surface roughness, and promote high surface energy and better wettability, thereby improving interfacial adhesion (ISO/TS 11405:2003).

Some dental zirconia manufacturers recommend airborne-particle abrasion and liner application as routine pretreatment methods. These procedures significantly affect interfacial adhesion and failure modes were significantly affected by airborne-particle abrasion or application of liner material, but their use to enhance ZrO_2 core-veneer adhesion remains controversial (Aboushelib, 2005). Some previous studies have recommended liner application to improve contact between the materials; however, other studies have shown that liner can weaken adhesion (Aboushelib, 2008b; Tinchert, 2001). No standard method for the achievement of optimal adhesion between zirconia and veneering ceramics has been established.

The purpose of this study was to test the shear bond strength of veneering ceramic to pre-sintered ZrO_2 cores after various surface treatments. The first null hypothesis was that the shear bond strengths of veneering ceramic to untreated and surface-treated ZrO_2 would not differ. The second null hypothesis was that shear bond strength would not be affected by veneering techniques and repeated firing.

2. Material and Methods

Specimens ($n = 120$) consisting of pre-sintered ZrO_2 cores and veneer ceramics were prepared with the Kavo Everest system (Kavo Dental GmbH, Biberach, Germany) according to ISO TS 11405 standards. Cylindrical cores had connection surfaces with 7-mm diameters and 2-mm heights and lower sections for fixation into a metal mold (3.2-mm diameter, 7-mm height; cylindrical form). Pre-sintered (non-hot isostatic pressed) zirconia blocks (ZS Blank; Kavo Dental GmbH, Biberach, Almany) were used; the cores were adjusted automatically by the system and abraded with higher volume to compensate for 20% sintering contraction. The core specimens were then subjected to

sintering treatment at 1500°C for 12 h in a sintering oven (Kavo Everest Therm; Kavo Dental GmbH, Biberach, Germany). After sintering, original specimen sizes were obtained with 20% shrinkage.

2.1. Surface Treatment

Specimens were divided into three groups ($n = 40$ each) subjected to no surface treatment (control group), sandblasting with 110- μm Al_2O_3 particles (SB group), and sandblasting with 110- μm Al_2O_3 particles and liner application (SBL group GC Initial Zr-FS Frame Modifier, GC Europe N.V., Interleuvenlaan, Leuven, Belgium). A mechanism was prepared to keep the specimens and the tip of the sandblasting instrument in fixed positions during sandblasting treatment. Sandblasting was applied to the core connection surfaces at 10 mm distance for 15 s with a pencil-point instrument (Renfert Basic Classic; Renfert GmbH, Hilzingen, Germany) and 110- μm Al_2O_3 particles (Cobra; Renfert GmbH) under 2 bars pressure. In the SBL group, liner material (GC Initial Zr-FS Frame Modifier; GC Europe N.V., Leuven, Belgium) with a high feldspar ratio and low glass-phase content, developed by the manufacturer for use with its veneer ceramic, was mixed and applied to the core surfaces after SB treatment.

2.2. Veneer Application

GC Initial Zr-FS (GC Europe N.V., Interleuvenlaan, Leuven, Belgium) and GC Initial PC (GC Europe N.V., Interleuvenlaan, Leuven, Belgium) veneer materials were each applied to 20 specimens per group, using the layering and pressing techniques, respectively. To standardize veneering, a metal mold was prepared for each technique. Mold size was adjusted for the application of 3-mm thicknesses of veneer ceramics to the ZrO_2 cores. After removal from the molds, specimens were fired in a programmable vacuum ceramic oven (Programat P300; Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions (Table 1).

2.3. Firing

Specimens with GC Initial Zr-FS veneers were fired once at the temperature of the first dentin (810°C); specimens with GC Initial PC veneers were fired once at pressing temperature. The firing procedure was applied once and three times to 10 specimens per veneer subgroup, respectively. In the GC Initial Zr-FS subgroups, repeated firing was performed at 800°C of the second dentin, followed by cooling at room temperature. Details of the repeated firing treatment are provided in Table 2 (Fig. 1).

2.4. Bond Strength Testing

Shear bond strength was tested using a universal testing machine (Autograf AG-IS 5K-N; Shimadzu, Kyoto, Japan). A knife edge-shaped apparatus was used for testing of 1-mm thicknesses, as specified in the ISO TR 11405 standards (ISO/TS 11405:2003). The apparatus was positioned perpendicular to the core-veneer bond surface and immobilized with screws. Force was applied and the test was performed at a rate of 1 mm/min (Fig. 2).

Bond surfaces were then examined under a scanning electron microscope (SEM; JSM-5600; Jeol Ltd., Tokyo, Japan). After surface coating, photographs were taken at $\times 100$, $\times 150$, $\times 250$, $\times 500$, and $\times 1000$ magnifications.

2.5. Statistical Analysis

Intergroup comparison of parameters was performed using one-way and two-way analyses of variance ($p < 0.05$), and Tukey's honestly significant difference test was used to identify the group responsible for any difference.

3. Results

The highest and lowest bond strengths were observed in the SB + GC Initial PC + refiring (38.43 ± 10.12 MPa) and SBL + GC Initial Zr-FS (19.49 ± 1.80 MPa) subgroups, respectively ($p < 0.01$; Table 3, Fig. 3). In the SB group, mean bond strength was significantly higher in the GC Initial PC + refiring subgroup than in the GC Initial Zr-FS + refiring subgroup ($p < 0.01$; Table 4). In the SBL group, mean bond strength was significantly higher in the GC Initial PC + refiring subgroup than in the GC Initial Zr-FS subgroup ($p < 0.05$). In the control group, mean bond strength was significantly higher in the GC Initial PC + refiring subgroup than in the GC Initial Zr-FS and GC Initial Zr-FS + refiring subgroups ($p < 0.01$).

Bond strength did not differ significantly among GC Initial Zr-FS or GC Initial Zr-FS + refiring subgroups. Among specimens receiving GC Initial PC veneers alone, mean bond strength was significantly lower in the SBL subgroup than in the SB and control subgroups ($p < 0.01$). Among specimens receiving GC Initial PC veneers and refiring, mean bond strength was also significantly lower in the SBL subgroup than in the SB and control subgroups ($p < 0.01$)(Table 4).

Microscopic examination of fracture surfaces after shear bond strength testing revealed adhesive ($n = 45$) and combined (adhesive + cohesive; $n = 75$) fracture patterns (Fig. 4). No specimen showed cohesive fracturing alone. The largest number of adhesive failures was observed in the SBL group.

4. DISCUSSION

This study was conducted to examine reported effects of core surface treatment and veneer application on the bond between pre-sintered ZrO₂ cores and veneer ceramics (Guazzato, 2004b; Teng, 2012). It demonstrated that sandblasting treatment resulted in significantly higher shear bond strength than did liner application. The first null hypothesis was rejected, as significant differences were observed among groups receiving different surface treatments. The second null hypothesis was also rejected, as shear bond strength was affected by veneering technique and repeated firing.

Sintering surface treatments have been found to weaken the zirconia structure, causing micro cracks (Guess, 2010), and to increase monoclinic phase content, which also leads to the increased occurrence of fracture (Moon, 2011). This study stated that additional heat should be applied to reduce monoclinic phase content; thus, they applied surface treatments to pre-sintered ZrO₂. Pre-sintering of ZrO₂ has many advantages, such as the elimination of sharp areas that adversely affect ZrO₂ bonding and increase fracture potential of ZrO₂ after surface treatment, as well as enhanced tetragonal phase content, which enhances the mechanical properties of the material. Thus, surface treatments applied to increase bond strength between veneer ceramics and the ZrO₂ substructure was applied before sintering in the present study.

Monaco et al. (2011) obtained the highest bond strength by sandblasting (110- μ m Al₂O₃) pre-sintered zirconia; they claimed that the difference from the control group was significant, and they found that Group SB compared with Group C showed higher bond values on average. In contrast to the properties of polished surfaces, sandblasting may create a large surface area and mechanical undercuts for adhesion, increasing surface energy and wettability (Aboushelib, 2006; Abi-Rached, 2014). Also in the present study, SB groups' mean bond strength was significantly higher in the Group C.

Kim et al. (2011) performed sandblasting and sandblasting + liner treatments on Kavo Everest pre-sintered ZrO₂ cores and abraded control group surfaces with diamond discs alone. They applied Cerabien ZR veneer ceramic using the layering technique. They found that liner application markedly increased the failure rate of core-veneer bonds and reduced bond strength compared with

sandblasting alone. Similarly, among GC Initial Zr-FS specimens, bond strength was greater in the SB subgroup than in the SBL and control subgroups in the present study, with no significant difference between the latter two subgroups.

Aboushelib et al. (2008b) achieved the highest bond strength with sandblasting in specimens to which Nobel Rondo veneer ceramic was applied. They observed significant differences between sandblasting and control subgroups only in the Cercon and Procera groups. Harding et al. (2012) found that sandblasting increased ZrO₂-veneering porcelain bond strength, with no difference between the control and sandblasting + liner groups. Also in the present study; sandblasting groups had the highest shear bond strength and no differences found between the control and sandblasting + liner groups.

In the present study, liner application reduced bond strength compared with sandblasting alone. The main reason for failure on surfaces to which liner has been applied (which are flat) is delamination due to thin veneer layering or low core-veneer bond strength (Aboushelib, 2006; Aboushelib, 2008b) the failure rate on these surfaces exceeds that on untreated surfaces. The long-term performance of all-ceramic restorations depends on the adhesion of veneer ceramic to the core and its mechanical integrity (Al-dohan, 2004).

In the recent researches (Aboushelib, 2006; Aboushelib, 2008b); they investigated effects of liner materials with different components on bond strength and they observed generally delamination of veneer. This situation may occur in cases of thin veneer layer or poor interface bond. There is a poor contact between two materials with flat surface that has a potential of fracture. Aboushelib et al. (2006) found that the Nobel Rondo Dentine and Ceram Express pressable veneer ceramics showed the greatest bond strength to a pre-sintered ZrO₂ core (41.1 ± 11.1 and 38.6 ± 6.4 MPa, respectively). They also investigated the effects of liner material, and recommended its use with veneer ceramics applied with the layering technique; the use of liner with pressable veneer ceramics significantly reduced bond strength and dramatically increased the risk of veneer delamination. Similarly, among GC Initial PC specimens, bond strength was significantly lower in the SBL group than in the SB and control groups in our study. In contrast to Group C, SB treatment may create a large surface area and mechanical undercuts for adhesion, increasing in surface energy and wettability. However, Yoon et al. (2014) indicated that the effects of surface treatment on bond strength at the pre-sintered ZrO₂ core-veneer interface differ according to the type of liner material used.

Under clinical conditions, different types of fracture may occur in restorations under functional occlusal loading. Laboratory technique has important clinical effects, and repetition of firing up to three times may be required due to additions made during veneer preparation (Guess, 2008). To reflect clinical conditions better, firing treatment was applied once to half of specimens and 3 times to the other half during preparation of veneers. Blatz et al. (2006) applied three different veneer ceramics (Lava Ceram, Carebion ZR, and GC Initial) and pre-sintered ZrO₂ cores (Lava Core) and divided experimental groups into two sub-groups. While the first group was only kept in salty water, the second group was exposed to heating cycle 10.000 times. As a result of bond strength of the specimens that exposed to heating cycle were significantly higher than the other groups. It was reported that heating cycle application improved the bond strength. According to Fischer et al. (2008) bonding of the veneering ceramics to polished ZrO₂ surfaces put forward that chemical bonds were established between both materials during firing. Also Nishigori et al. (2014) indicated that when sandblasting is used, heat treatment of the core before veneering should enhance restoration longevity. Consequently, their results showed that firing procedure enhance shear bond strength. Also it is reported that, more than 3 firings, the greater the number of firings

the higher the bond strength (Trindade, 2013). In the present study; also, when the number of veneer firings were increased, the bond strength values increased in a similar way to these studies. Although the mechanism of ZrO₂-veneer ceramic adhesion has not been determined precisely, investigators have suggested that fusion between the core and veneer results in diffusion of the elements of both materials across the bonded interface (Durand, 2012). However, as changes in the crystalline structure of some veneering porcelains have been observed with excessive refiring (Tang, 2012) the avoidance of unwarranted refiring procedures is preferable.

SEM examination has revealed less porosity at ZrO₂ core-pressable veneer interfaces than at ZrO₂ core-layered veneer interfaces. Similar results were obtained in this study, suggesting that the reduced porosity and homogenous ingot structure of pressable veneer ceramics improves bond strength. The advantage of pressable veneer ceramics is due to reduced crystallization in the matrix structures of these materials, and that pressable ceramic exhibit less crystallization (Aboushelib, 2008a). Also in this study; less porosity was observed on the surface of pressable veneer ceramics applied onto ZrO₂. As a result of surface examinations we think that porosity-free structure of veneer ceramic improves the bond strength. Homogenous structure of ceramic ingots we applied with pressing technique enables a stronger bond. In our study, SB + pressing + 3 times firing group (38.43 ± 10.12 MPa) shows the highest bond value and group C + pressing + 3 times firing group (33.83 ± 3.76 MPa) shows the second highest value.

In previous studies, SEM examination has shown primarily adhesive fractures in specimens to which liner has been applied, and predominantly combined (adhesive + cohesive) fractures in those treated with sandblasting alone (Guess, 2008; Kim, 2011;Matsumoto, 2013). In the present study, adhesive failure was observed more often than combined failure in all specimens, to which liner was applied, perhaps because liner forms a thin layer between the zirconia and veneering porcelain, weakening the bond. Sandblasting is more useful than liner application alone in increasing the bond strength of veneering ceramic to a ZrO₂ core (Kim, 2011). Our findings of a high adhesive fracture rate and low bond strengths in specimens to which liner was applied support the findings of Kim et al. (2011). We thus conclude that surface application, rather than veneer ceramic application technique, plays a role in determining fracture type.

Similar to our results, Aboushelib et al. (2008b) reported an increase in adhesive failure rate in zirconia + veneering porcelain + liner groups compared with sandblasting and control groups. They achieved similar results that supporting of our study. They reported that the most adhesive failures were in the sandblasting + liner group. The application of surface treatments to ZrO₂ cores before sintering may explain the increased surface roughness values.

This study had several limitations. The layered and pressed specimens did not represent the clinical shape of ZrO₂ restorations. In addition, the effects of aging conditions on the bond quality of ZrO₂ to veneering porcelain were not evaluated. Moreover, the design of the specimens could have influenced the heat capacity or thermal conductivity of the core material during firing. The effect of thermal cycling on the bonded zirconia-veneer interface also needs to be evaluated.

5. CONCLUSION

The use of sandblasting on zirconia cores is more effective than liner application in improving core-veneer bond strength. In addition, sandblasting before liner application may improve the bond strength of veneer ceramics applied with the pressing technique. However, the mechanism of core-veneer bonding has not been explained completely. As several parameters in the oral environment cannot be reflected in experimental studies, additional clinical studies with the aims of improving ZrO₂ core-veneer bond strength and reducing complications are needed.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Table 1. Preparation parameters for veneer ceramics used in this study

	T (°C)	D (min)	F (°C)	R (min)	I (°C)	V1 (°C)	V2 (°C)	P (bars)
GC Initial Zr-FS	450	6	810	1	45	450	810	-
GC Initial PC	700	-	950	20	60	500	950	4.5–5

T: preparation temperature, D: pre-drying duration, F: firing temperature, R: duration of retention at firing temperature, I: increase in ceramic oven temperature per minute, V1: temperature at start of vacuuming, V2: temperature at end of vacuuming, P: pressure.

Table 2. Repeated firing parameters

	T (°C)	D (min)	F (°C)	R (min)	I (°C)	V1	V2
Repeated firing	450	6	800	1	45	450	800

T: Preparation temperature, D: Pre-drying term, F: Firing degree, R: Term of retention at firing degree, I: Degree of increase in ceramic oven temperature in a minute, V1: Temperature at start of vacuuming, V2: Temperature at the end of vacuuming

Table 3. Bond strength in experimental groups

Surface application	Veneer application	Number of firings	n	Mean bond strength (MPa)	SD (MPa)	Min (MPa)	Max (MPa)	
Sandblasting	Layering	1	10	22.87	8.21	17.88	36.61	
		3	10	25.24	4.77	17.78	32.23	
	Pressing	1	10	32.23	7.90	23.70	49.66	
		3	10	38.43	10.12	22.28	55.35	
Sandblasting + liner	Layering	1	10	19.49	1.80	16.43	22.50	
		3	10	21.60	2.78	17.96	26.67	
	Pressing	1	10	19.92	5.34	14.72	29.68	
		3	10	23.82	2.48	21.21	29.35	
	None	Layering	1	10	20.87	4.98	13.16	28.01
			3	10	23.36	3.69	16.68	28.21
None	Pressing	1	10	30.59	8.40	22.68	50.95	
		3	10	33.83	3.76	30.17	42.42	

⁺Two-way ANOVA.

Table 4. Comparison of bond strength

Surface Application	Veneer Application Technique – Number of Firings	⁺⁺ <i>p</i>
Sandblasting Group (SB)	Layering (22.87) & Layering + 3 times firing (25.24)	0.910
	Layering (22.87) & Pressing (32.23)	0.060
	Layering (22.87) & Pressing + 3 times firing (38.43)	0.001**
	Layering + 3 times firing (25.24) & Pressing (32.23)	0.224
	Layering + 3 times firing (25.24) & Pressing + 3 times firing (38.43)	0.004**
	Pressing (32.23) & Pressing + 3 times firing (38.43)	0.321
Sandblasting + Liner Group (SBL)	Layering (19.49) & Layering + 3 times firing (21.60)	0.512
	Layering (19.49) & Pressing (19.92)	0.992
	Layering (19.49) & Pressing + 3 times firing (23.82)	0.034*
	Layering + 3 times firing (21.60) & Pressing (19.92)	0.687
	Layering + 3 times firing (21.60) & Pressing + 3 times firing (23.82)	0.466
	Pressing (19.92) & Pressing + 3 times firing (23.82)	0.065
No Surface Application Group (C)	Layering (20.87) & Layering + 3 times firing (23.36)	0.750
	Layering (20.87) & Pressing (30.59)	0.002**
	Layering (20.87) & Pressing + 3 times firing (33.83)	0.001**
	Layering + 3 times firing (23.36) & Pressing (30.59)	0.030*
	Layering + 3 times firing (23.36) & Pressing + 3 times firing (33.83)	0.001**

	Pressing (30.59)	&	Pressing + 3 times firing (33.83)	0.567
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Tukey HSD Test

** $p < 0.05$*

*** $p < 0.01$*

Figure 1: Experiment specimen prepared



Figure 2: shear bond strength test on specimens



Figure 3: Graphical evaluation of bond strength values of all groups (MPA).

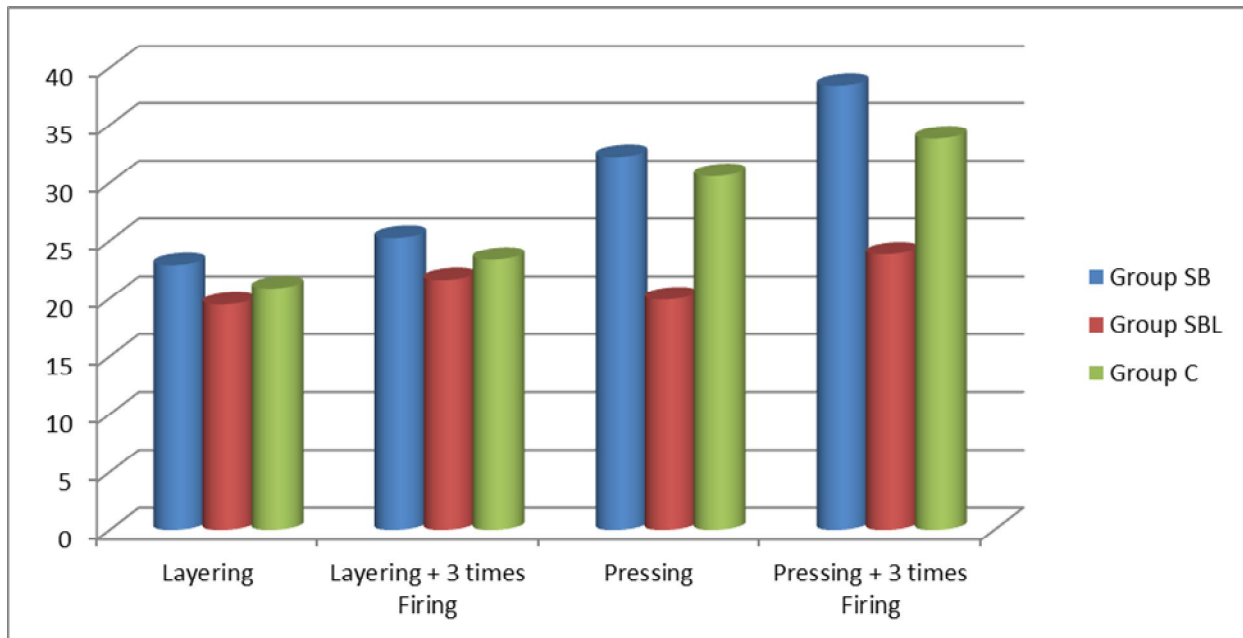


Figure 4: SEM images of the experimental groups.

