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Bonding of Veneering Porcelain to Zirconia Based Ceramics Using a Novel Surface Treatment Method

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Keywords: Zirconia, Veneering, Manual Layering, Press-on, Shear bond strength, Selective infiltration etching, Sandblasting, Surface treatment.

ABSTRACT: ***Purpose:*** The aim of this study was to assess the validity of a new technique (Selective Infiltration Etching, SIE) to improve bonding between zirconia core and the veneering ceramic. ***Methods:*** Forty eight Zirconia partially stabilized Y-TZP discs (19 mm in diameter x 2.5 mm thick) were prepared out of Six Cercon Y-TZP milling blocks. The discs were divided into 3 main groups according to surface treatment. Group 1: Received no surface treatment (Control group).Group 2:Airborne particle abraded (sandblasted). Group 3: Received SIE surface treatment. Discs from each group were scanned using a scanning electron microscope (SEM) and EDAX Discs were further tested for surface roughness (Ra) using a Surface Roughness Reader. After surface roughness analysis each main group was further subdivided into 2 sub-groups according to the veneering technique. The first sub-group was veneered using the manual layering technique whereas the second sub-group was veneered using the press-on technique. After veneering the zirconia discs, each disc was tested for shear bond strength. After fracture, the discs were scanned using a SEM to evaluate the mode of failure. ***Conclusions:*** Specimens showed adhesive failure with the manual layering veneering technique. On the other hand, they showed cohesive failure with the press-on veneering technique. SIE group in interaction with the press-on veneering technique showed the statistically significantly highest mean micro shear bond strength value (26.1±19.0 MPa). This was followed by sandblasting/layering (21.6±3.5 MPa), sandblasting/press-on (19.7±3.0 MPa), as sintered/press-on (18.7±1.6 MPa) and as sintered/layering(18.5±3.5 MPa) with no statistically significant difference between the four groups. The statistically significantly lowest mean microshear bond strength was recorded with SIE/layering (10.3±3.3 MPa). *Clinical implications*:1-Whenever layering technique is feasible, airborne particle abrasion would be a suitable surface treatment.2- Selective Infiltration Etching (SIE) is not an effective surface treatment when used with the layering veneering technique.

1. INTRODUCTION

The porcelain-fused-to-metal restoration has proved to be a reliable treatment option for fixed partial dentures (FPD) owing to its high predictable strength and reasonable esthetics.[1] However, the disadvantage of such restorations is its artificial appearance due to the increased light reflectivity caused by the opaque porcelain needed to mask the metal substrate[2] and the graying effect of the metal at the gingival margin.[3] The increasing demand for superior esthetics in addition to the increasing public awareness of the adverse side effects of some dental alloys have accelerated the development of alternatives to metallic dental restorations.[4] Numerous attempts have been made to develop all-ceramic systems that eliminate metal infrastructures providing optimal distribution of reflected light.[5] There is a growing interest in the use of zirconia oxide ceramics as substitutes for metal core structures. This is due to the fact that zirconia oxide ceramic has superior mechanical properties, including a high flexural strength and toughness. In addition, the development of new technology, such as computer aided design/computer-aided manufacturing (CAD/CAM), enables the fabrication of zirconia-based restorations for all-ceramic crowns in a more practical process.

Current processing technologies however, cannot make zirconia frameworks as translucent as natural teeth. In order to achieve acceptable esthetics, the zirconia based core structure is veneered with layering ceramic that has a suitable coefficient of thermal expansion matching the core ceramic. Before veneering, one or more surface treatments are typically performed. Surface treatments, such as sandblasting, polishing and grinding have been advocated to prepare zirconia veneering surfaces. Available clinical studies support zirconia's performance potential, with indications of expanded functionality compared with other ceramics, as in case of long span bridges. The primary issues noted in such studies were not related to framework integrity, but rather chipping, wear and fracture of the veneering ceramics.

Clinical failures of veneered Y-TZP frameworks due to chipping of the veneered ceramic are reported to be 13.0% after an observation period of three years [6] and 15.2% after five years.[7] Selective infiltration etching (SIE) is a novel technique, suggested as a surface treatment for zirconia core material to enhance its bond strength to veneering porcelain using two veneering techniques.

2. MATERIAL AND METHODS

2.1 *Specimen preparation:* Six Cercon zirconia blocks (*Degudent GmbH, Hanau-Wolfgang, Germany)* were used to obtain the 48 discs using a precision cutting instrument (*Micracut, Metkon, Bursa. Turkey*) and a diamond-coated cutting disc (*Metkon, Bursa. Turkey*). The location of the cuts was controlled using a traveling stage and a horizontally displaced digital micrometer. After completion of the slicing process, the specimens were separated with a diamond cutting instrument from the rest of the block. They were trimmed to remove excess material at the site of connection. The specimens were then cleaned using an Ultrasonic cleaner in distilled water to remove grinding dust before further use. The cut specimens were sintered using the relevant electrical induction furnace recommended by the manufacturer (*Cercon Heat, Degudent, GmbH, Hanau-Wolfgang, Germany)*.

The discs were divided into 3 main groups according to surface treatment; 16 discs in each group. Group 1: This group received no surface treatment and was left as-sintered. Group 2: This received sandblasting treatment, discs were airborne-abraded with 50-µm aluminum oxide particles at 0.2 MPa pressure at a distance of 2 cm for 30 seconds. Group 3: This group received SIE surface treatment, where the specimens were coated with a thin layer of a glass-conditioning agent composed of silica (65% wt), alumina (15% wt), sodium oxide (10% wt), potassium oxide (5% wt), and titanium oxide (5% wt). The powder of the glass was mixed with water to give a thin, creamy mixture, which was applied on the surface of the specimens using a special spray and a compressor. The specimens were then heated to 650 °C for 3 minutes and then cooled to 23 °C. The heating and cooling rates (90 °C/min) were controlled by a computer-calibrated electrical induction furnace (*Cercon Heat, Degudent, GmbH, Hanau-Wolfgang, Germany*). After cooling to room temperature, all traces of the conditioning agent were completely washed away in 5% hydrofluoric ultrasonic bath for 15 minutes, followed by rinsing with water for 5 minutes after etching. Following surface treatment and analysis, each main group was divided into two sub-groups according to the veneering method with 8 discs each. The first sub-group was veneered using the manual layering technique.*(Table 1)*

The representative sample was ultrasonically cleaned and gold sputter-coated for SEM examination. The surface was examined under different magnifications to measure the surface morphology. After primary SEM examination, the polished sections were slightly etched using 2% hydrofluoric acid for 15 seconds to remove the smear layer and enhance structural analysis. The chemical structure was analyzed using EDAX.

2.2 Veneering technique: Manual layering: Four circular veneers of 2 mm diameter were added on each single surface of each disc. The prepared zirconia discs were placed in an adjustable teflon mold where 1.5 mm clearance was available for condensing the veneer ceramic. The powder of each veneer was mixed with the manufacturer's liquid and the obtained slurry was plotted with tissue paper to draw excess water. The mold was filled, ultrasonically condensed and the condensed slurry was pressed using pneumatic piston. Each layered zirconia disc was fired according to the firing program of the manufacturer (*EP500, Ivoclar, Vivadent, USA*). The previous step was repeated giving a final veneer thickness of 3 mm. The same steps were used to prepare 192 homogeneous veneer discs, which were used to evaluate the micro-shear bond strength. Each layered zirconia disc was fired according to the firing program of the manufacturer was carried out as follows: Pre-drying temperature was 575 °C with a 4-min time. Heating-up time was 7 minutes to reach a firing temperature of 980 °C at a rate of 45 °C/min with vacuum. The holding time was 8 minutes.*(Figure 1,a)*

The second sub-group was veneered using the press-on technique, where 4 circular veneers made of Ceram Cercon R Express (*DegudentGmbH, Hanau-Wolfgang, Germany*)of 2 mm diameter were added on each single surface on each disc. Four sprues with a diameter of 2 mm were attached to each zirconia disc, invested and the samples were processed using manufacturer's instructions and equipment for pressing technique. The pressing time was adjusted to 10 minutes for each disc. Finally, the pressed rings were divested and the ceramic sprue was cut under water cooling using a diamond disc. A firing cycle according to the manufacturer's recommended parameters was carried out as follows: Initial temperature was 700 °C with a 4-min time. Heating-up time was 8 minutes to reach a firing temperature of 940 °C at a rate of 60 °C/min with vacuum. The holding time was 20 minutes with a pressure of 5 bar.*(Figure 1,b)*

2.3 *Testing procedures:* ***Before Veneering,*** discs from each group were tested for surface roughness (Ra) using a surface roughness reader (type, manufacturer, city, state) and the readings were recorded, tabulated and statistically analyzed. Discs were further scanned using a scanning electron microscope (*SEM, Jeol 5300, USA)* and energy dispersive X-ray spectroscopy (*EDAX, Jeol 5300, USA*). **After veneering,** the zirconia discs each disc was tested for micro-shear bond strength. Specimens were fixed in a special sample holder and placed in a universal testing machine (Z010, Zwick, Ulm, Germany). The ceramic block was loaded up to failure at the interface parallel to the zirconia surface with a crosshead speed of 1 mm/min. *(Figure 2 a,b)* **After fracture,** the discs were scanned using scanning electron microscopy (type) to evaluate the mode of failure.

2.4 *Statistical Analysis:* Data were presented as mean and standard deviation (SD) values. Two-way Analysis of Variance (ANOVA) was used in testing significance for the effect of surface treatment, veneering technique and their interactions on shear bond strength. One-way Analysis of Variance (ANOVA) was used to compare surface roughness (Ra) values.Tukey's posthoc test was used for pair-wise comparison between the mean values when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with PASW Statistics 18.0 *(Predictive Analytics SoftWare*) for Windows.

3. RESULTS

The results showed that surface treatment, veneering technique and the interaction between the two variables had a statistically significant effect on mean shear bond strength.

3.1 *Surface analysis:* SEM examination before veneering also showed that the as-sintered group showed a dense, smooth, homogenous surface. The airborne abraded group revealed more surface roughness features, some elevations and depressions. Sharp micro-irregularities and remnants of airborne were also noticed. The SIE group demonstrated an architecture of numerous retentive micropores along with abundant intergrain spaces.*(Figure 3)*

With only respect to surface treatment results, there was no statistically significant difference between sandblasting group and SIE group Both showed the statistically significantly highest mean Ra values. As-sintered group showed the statistically significantly lowest mean Ra. The mean and standard deviation values of the average surface roughness (Ra) of the as sintered group were 0.33±0.06 µm, 0.66±0.15 µm after sandblasting and 0.80±0.11 µm, after SIE treatment. (Table 2, a. Figure 4)

Irrespective of the surface treatment technique, Press - on veneering technique showed statistically significantly higher mean microshear bond strength value (21.5±4 MPa) than layering veneering technique mean values (16.8 ±5.9 MPa).*(Table 2, b. Figure 5)*

As for the EDAX results, the elemental composition analysis of the as sintered disc revealed the component elements in a descending pattern: zirconia (ZrL), oxygen (OK), carbon (CK), yttrium (YL), and a small percentage of silica (SiK). For the airborne abraded group the composition analysis revealed that the major component elements included high concentrations of oxygen (O), zirconia (Zr), yttrium (YL), and a considerable amount of aluminum (AL). Regarding the last group, SIE the elemental composition analysis revealed higher concentrations of Silica (Si) along with different elements with different percentages such as Carbon (C), Oxygen (O), Aluminum (AL), Yttrium (Y) and Zirconia (Zr).

3.2 *Shear bond strength results:* SIE group in interaction with the press-on veneering technique showed the statistically significantly highest mean shear bond strength value (26.1±19.0 MPa). This was followed by sandblasting/layering (21.6±3.5 MPa), sandblasting/press–on (19.7±3.0 MPa), as sintered/press–on (18.7±1.6 MPa) and as sintered/layering (18.5±3.5 MPa) with no statistically significant difference between the four groups.The statistically significantly lowest mean micro-shear bond strength was recorded with SIE/layering (10.3±3.3 ±). All results are compiled in *(Table 2,c.Figure 6).* **3.3** *Failure mode assessment:* All specimens showed adhesive failure with the manual layering veneering technique, the presence of abundant air-bubbles were revealed (Figure 7, a).On the other hand, they showed cohesive failure with the press-on veneering technique, he presence of a bubble-free surface along with a homogeneous dense structure is apparent. *(Figure 7, b).* The as-sintered group with manual layering technique showed adhesive failure of the veneered layer, de-lamination of veneer from intact zirconia disc, insufficient (poor) wetting between the core and the veneer. The as-sintered group with press-on veneering technique showed cohesive failure of the veneered layer with the press-on, apparent good wetting of the veneer with minimum air-bubbles on the surface. Airborne-abraded group with manual layering technique showed adhesive failure of the veneered layer with remnants of sandblasted particles and veneer. Airborne-abraded group with press-on veneering technique showed cohesive failure of the veneered layer, wave like appearance that showed the integrity of the press-on veneer indicating good wetting to the underlying core. The SIE group with manual layering technique showed adhesive failure of the veneered layer, structural defects within the veneered porcelain with presence of air-bubbles. *(Figure 8,a)* On the other hand, with press-on veneering technique, this group showed cohesive failure of the veneered layer, no de-lamination and as shown cohesive failure resulted in cracks in the press-on veneered ceramic. *(Figure 8,b)*

4. DISCUSSION

The strength of a non-homogenous all-ceramic structure is determined by its weakest component.[8] Usually, this will be the core-veneer interface or the veneering material itself, which has to be strong enough to withstand the stresses of mastication to prevent delamination and fracture of the veneering material. Many variables may affect the core-veneer bond strength, such as the surface finish of the core, which can affect mechanical retention, residual stresses generated by mismatch in thermal expansion coefficient (TEC), development of flaws and structure defects at core-veneer interface, and also wetting properties and volumetric shrinkage of the veneer.[9] The individual and the combined effects of such variables can influence the core-veneer bond strength and therefore the clinical success rate of such restorations.[10]

This study was designed with aim to evaluate the influence of a novel surface treatment technique on the core along with the influence of different veneering techniques and thus the core-veneer bond strength of all ceramic restorations using shear bond strength tests. The surface roughness analysis test results showed low bond strength results observed for the 'as-sintered' specimens compared with the other two groups. Such findings indicate that establishing a strong chemical bond with zirconia is a difficult procedure when not combined with airborne-particle abrasion as a recommended surface pre-treatment. In this study, selective infiltration etching technique was also used. One of the most common tests used for evaluating the bond strength between core and veneer in different all-ceramic systems is the shear bond strength test.[11]

The results of the effect of the surface treatment on the shear bond strength per se revealed that sandblasting showed the highest mean micro-shear bond strength among all groups which was statistically significant. There was no statistically significant difference between SIE and as-sintered group, both showed the (statistically significantly) lowest value.

This result may be contradictory to our hypothesis which can be explained as the bond strength values of the SIE surface treated group with press-on zirconia were significantly the highest values. On the other hand, the same group when used with manual layered veneer showed significantly lower values which resulted in the reduction of average mean values.

The SIE procedure aimed to create a retentive surface architecture which differs from the common surface-roughening methods such as airborne-particle abrasion in that it is self introduced by the material without any applied external mechanical stresses, second, it occurs on the ultrastructural grain level without the creation of structural defects or material loss.[12] With proper heat-induced maturation treatment, the energy rich grain boundaries become destabilized as the surface grains tend to expand and shrink during the alternating temperature changes. Additionally, the zirconia crystals at the grain surfaces become strained and prestressed.[13] This process could be considered a controlled dynamic thermal etching procedure but at a relatively lower temperature (750 °C).

In a study by Aboushelib et al., structural and chemical analysis of the zirconia-ceramic interface revealed that ions such as silica, sodium, aluminum, and potassium can infiltrate fully sintered zirconia at grain boundary regions, and their concentration decreases from the surface to a depth of 8-10 μm.[14] Additionally, it was also reported that increasing the silica content at grain boundaries increased both grain sliding movements and the creep behavior of fully sintered zirconia, which could also explain how SIE surface treatment can alter the surface of zirconia.[15,16] This improved nanomechanical retention was confirmed by the SEM images and the significantly higher bond strength values of the SIE group, especially with press-on veneering.

According to the results of the present study, the addition of an SIE treatment to the zirconia core material improved the core veneer bond strength with the press-on veneering technique. In coherence with the significant difference in the bond strength values between the manually layered and the press-on technique the fracture mode for the press on remained primarily cohesive in nature; clearly noticed from the SEM images which showed that the zirconia surface was completely covered by the veneer, indicating that the established bond was significantly more resistant to failure than that with manually layered technique. The SEM images confirmed the results also showed better wetting at the edges of the veneer with the zirconia core and revealed the less homogenous bubble containing structure of the layered veneering ceramic.

It was previously reported that press-on veneer ceramics had higher zirconia-veneer bond strength than many available layering ceramics. This superior bond could be attributed to many of the attractive properties of the press-on technology, which is performed under controlled conditions, resulting in less incorporation of structural defects, improved wetting of zirconia surface by the molten pressed ceramic, and less incorporation of air bubbles, which are known to dramatically affect the strength of the veneer ceramic and its bond strength to the underlying framework material.[17,18] On the other hand, preparing a workable ceramic slurry for manual layering technique is operator dependent, and variations in the powder/liquid ratio and mixing technique are known to affect the density, the strength, the percentage of structural defects, and the number and size of air bubbles in the fired veneer.[19]

After the shear bond strength test, veneering ceramic remained on the zirconia surface for press-on technique specimen group only. Thus, it could be concluded that the bond strength between zirconia and the veneering ceramic was higher than the cohesive strength of the veneering ceramic. In other words, the weakest link was not the interface but the veneering ceramic itself. This also showed that the applied test design analyzed not the bond strength, but the shear strength of the veneering ceramic adjacent to the interface. These findings were consistent with the observations of Al-Dohan et al.[20] who also used a shear bond strength test. This failure result could be ascribed to the stress that was generated and which peaked near the interface, due to a difference in the coefficients of thermal expansion between both layers. This led to a stress concentration parallel to the interface, preventing crack propagation along the interface.

Another study proposed by Fischer et al.[21], which aimed to assess the effect of different surface treatments on the bond strength of veneering ceramics to zirconia. They stated that phase transition from monoclinic to tetragonal occurs at temperatures above 900ºC. However, micro-cracks do not close during heating at 1000ºC. The reduced shear strength observed in his study might be interpreted as follows: During regeneration firing, the compression layer at the surface relaxed, thus reducing the internal compression. Further, micro-cracks did not close at this temperature, consequently, the overall strength of zirconia decreased, thus affecting the shear strength of the veneering ceramic adjacent to the interface. That is, Y-TZP can be destabilized implying a degradation of its mechanical properties by spontaneous transformation of the metastable tetragonal phase into the monoclinic phase. And since the coefficient of thermal expansion of the monoclinic zirconia differs and is much lower than that of the tetragonal zirconia, this may result in difference in the TEC which may, in turn, create tensile stresses at the interface increasing the interfacial failure.[22,23]

Addition of Selective Infiltration Etching to the layering group negatively affected the shear bond strength which showed multiple porosities within the layered veneer and improper wetting at the edges of the veneer with the zirconia. A previously published research[24] stated that heat treatment and/or veneering negatively affected the mechanical properties of the restoration. The explanation of this is the formation of a compressive layer on the surface as an effect of the machining process and that the subsequent heat treatment/veneering relaxed these residual stresses.[25,26] Another study conducted by Aboushelib et al. [27,28], who evaluated the effect of combining both pressable and conventional layered ceramics in one restoration, on the core-veneer bond strength. The addition of the layered veneering ceramics resulted in an increase in the percentage of interfacial failure, compared to the pressable veneer group which demonstrated 100% cohesive failure. This was explained by the difference in thermal expansion coefficient (TEC) between the press-on technique and the conventional layered ceramics leading to undesirable residual tensile pre-stresses.

5. CONCLUSIONS:

Within the limitations of this study the following conclusions can be drawn:1- Press-on veneering technique is a more reliable veneering technique than the manual layering. 2- SIE surface treatment proved to be effective when used with the press-on technique. *Clinical Recommendations:* 1- Whenever layering technique is feasible, airborne particle abrasion would be a suitable surface treatment. 2- Selective Infiltration Etching (SIE) is not an effective surface treatment when used with the layering veneering technique.

6. Conflict of interest

All authors declare no conflict of interest.

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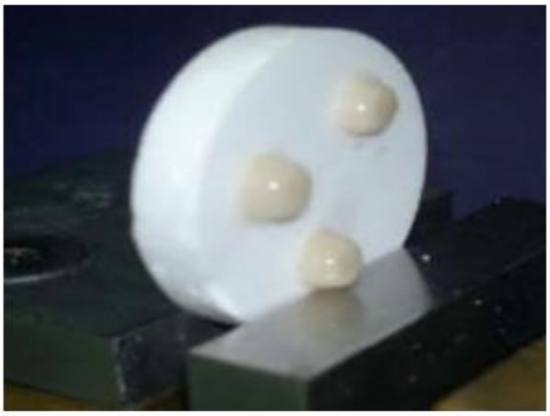
8. Figure

(B) Press-on veneer on zirconia disc

Fig. 2.

(A) Specimen before fracture



(B) Specimen after fracture

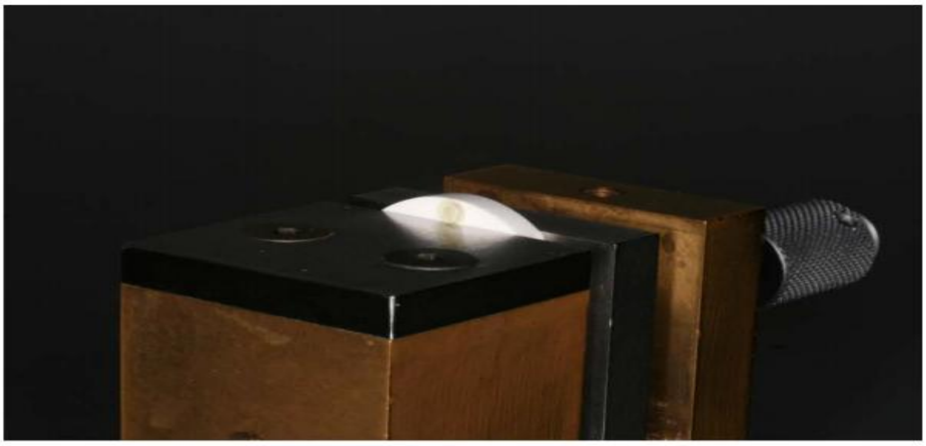


Fig. 3. SEM photomicrography of SIE treated zirconia disc before veneering

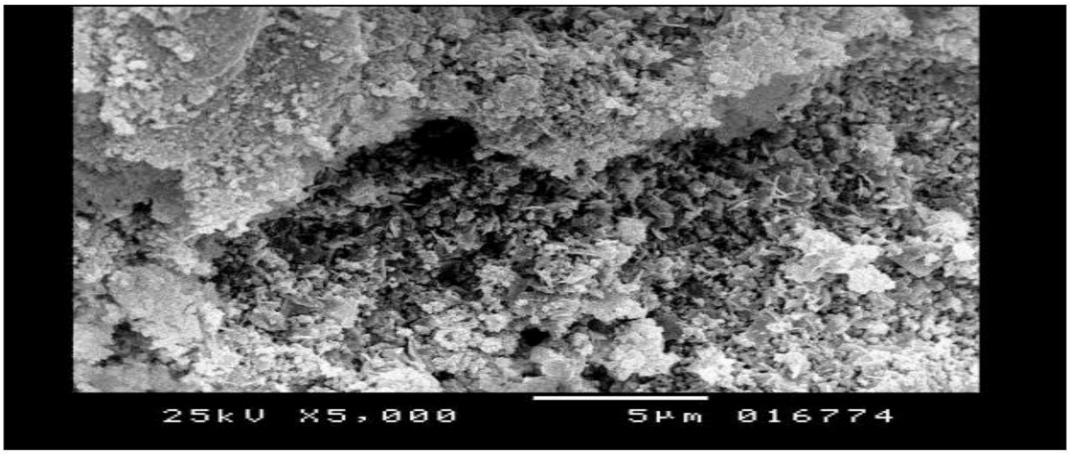


Fig. 4. Mean values of Ra(surface roughness) of the different groups

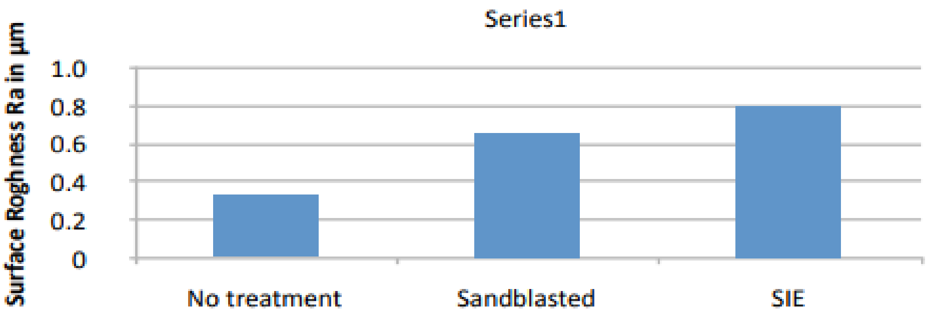


Fig.5 Mean values of microshear bond strength of veneering techniques

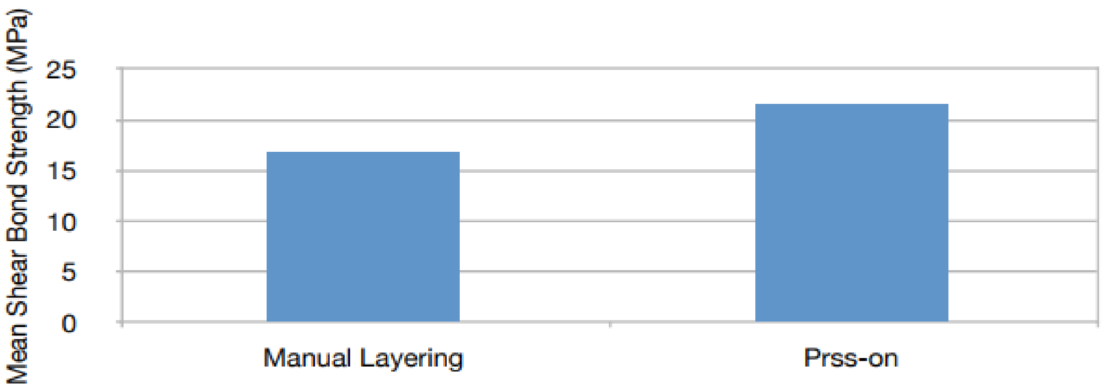


Fig. 6. Mean values of microshear bond strength of different interactions

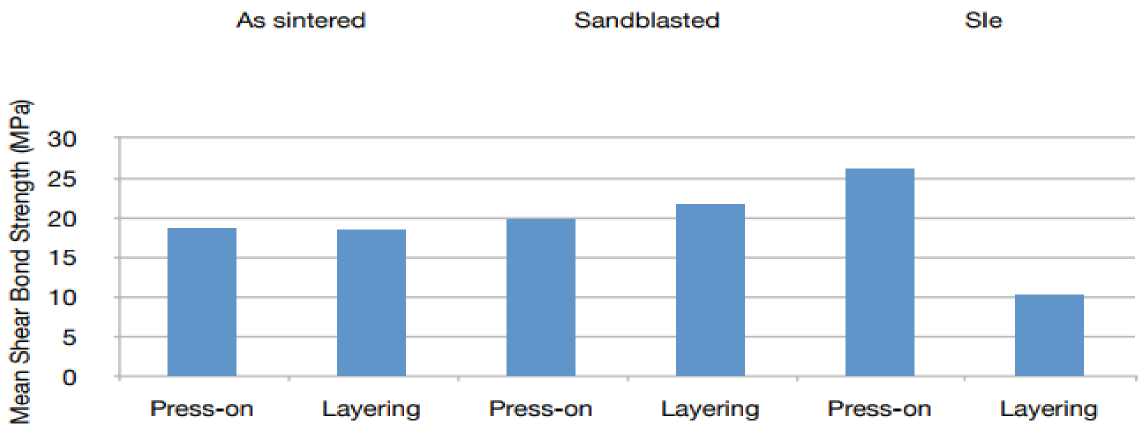
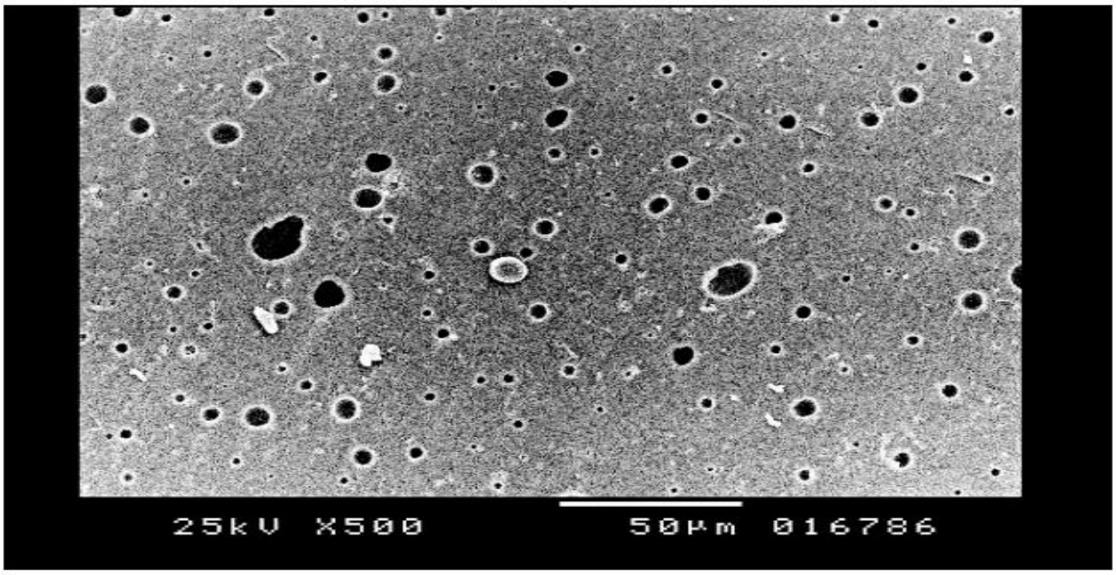


Fig. 7

(A). SEM photomicrograph of Layered veneer ceramic following de-bonding



(B) SEM photomicrograph of press-on veneer ceramic following de-bonding

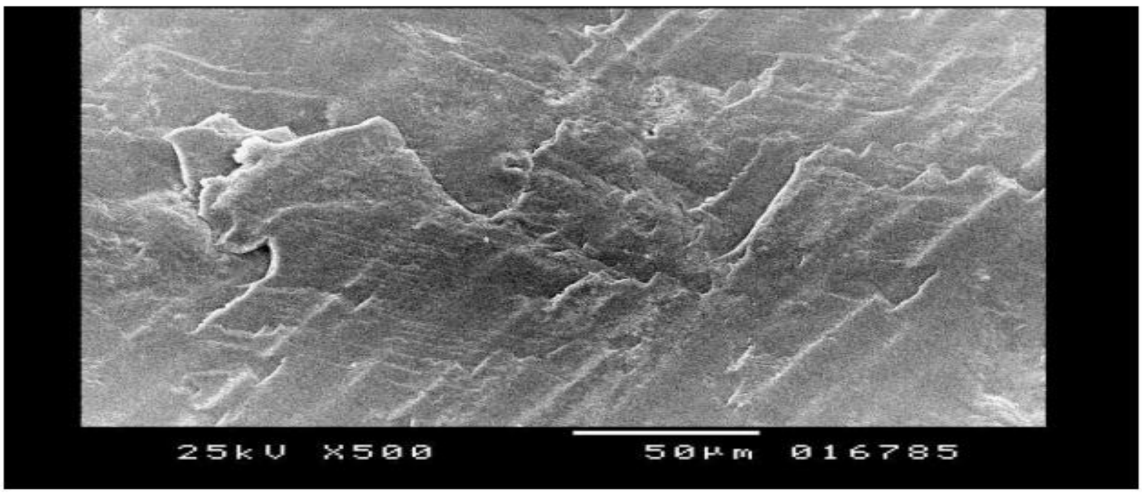
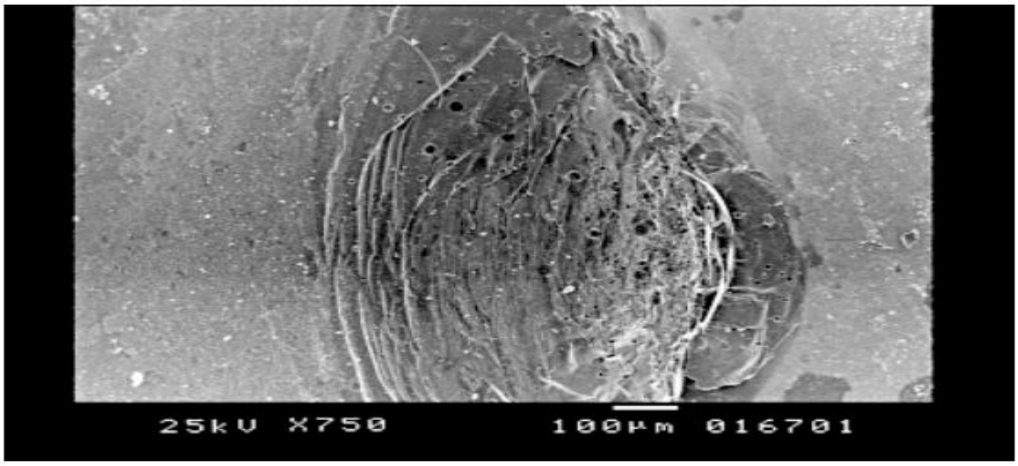
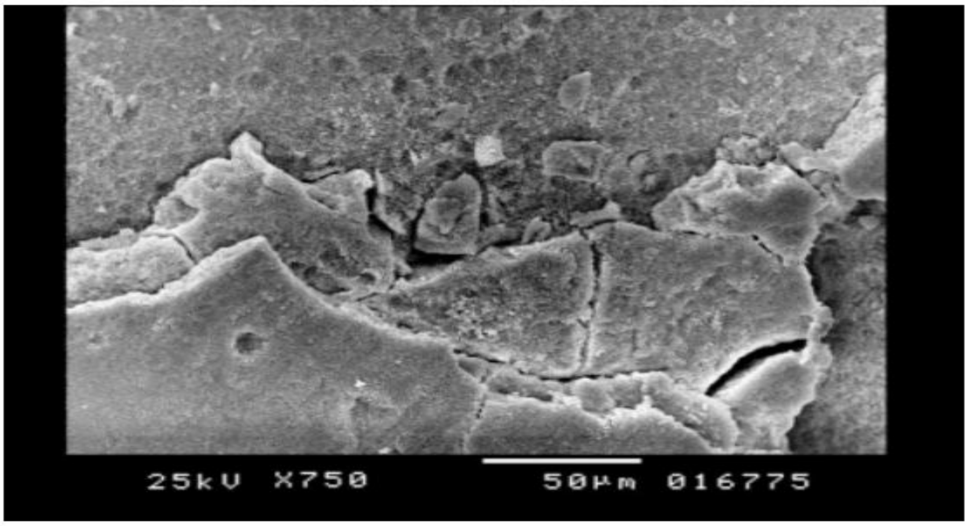


Fig. 8

(A) SEM photomicrograph of de-bonded SIE group with Layering technique



(B) SEM photomicrograph of de-bonded SIE group with Press-on technique



9.Tables

Table 1. Sample grouping

	Layering	Press-on
No treatment	8 samples	8 samples
Sandblasted	8 samples	8 samples
SIE	8 samples	8 samples

Table 2.

(A) Comparison between microtensile bond strength of the three surface treatments regardless of veneering technique

No surface treatment		Sandblasting		SIE		*P*-value
Mean	SD	Mean	SD	Mean	SD	
18.6 [b]	2.7	20.7 [a]	3.4	17.8 [b]	8.5	0.029*

(C) Comparison between microtensile bond strength with different interactions

Surface treatment	Veneering technique	Mean	SD	Rank	P-value
No surface treatment	Press – on	18.7	1.6	B	<0.001*
	Layering	18.5	3.5	B	
Sandblasting	Press – on	19.7	3	B	
	Layering	21.6	3.5	B	
SIE	Press – on	26.1	1.9	A	
	Layering	10.3	3.3	C	

: Significant at $P \leq 0.05$, Means with different letters are statistically significantly different according to Tukey's test