Influence of Different Sintering Stages of Zirconia core and Number of Firing Cycles of Veneer on Shear Bond Strength of Veneered zirconia. (In vitro study)

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ABSTRACT

Statement of the problem: Chipping of veneering layer and delamination occurred frequently, so several techniques used to enhance bond strength between zirconia core and veneering material.

Aim of the study: Evaluation of the effect of air abrasion/sintering order and number of veneer firing cycles on shear bond strength of veneering ceramic to zirconia core.

Materials and Methods: Forty zirconia blocks were milled from pre sintered zirconia disc (CeramillZolidht+ white) and randomly divided into two main groups (n=20) according to airborne abrasion time whether before or after sintering of zirconia. Three specimens from each group were analyzed by x-ray diffraction (XRD) to determine any phase transformation on zirconia surface. All zirconia specimens were veneered with IPS e.maxceram and furtherly divided into 4 subgroups (n=10) according to number of veneer firing cycles three or five. Shear bond strength (SBS) was tested using a universal testing machine. All specimens of each group were examined for failure mode analysis using an optical microscope. XRD test was done again to evaluate the effect of firing cycles on phase transformation of zirconia. Data were collected and analyzed with one-way and two-way ANOVA test.

Results: one-way ANOVA test revealed a statistically significant difference in SBS between the 4 study groups (p≤0.05). SBS was significantly higher in post sintered groups than pre sintered groups while there was no significant difference in SBS between 3 and 5 veneer firing cycles. Also, two-way ANOVA test results showed no significant difference regarding the interaction between the two variables in this study (time of air abrasion and number of firing cycles) p=0.247. XRD test revealed phase transformation after airborne abrasion and reverse transformation after sintering and different veneer firing cycles .

Conclusion: Air abrasion performed at post sintering stage had a favorable effect on SBS of all specimens, but it is not recommended at pre sintering stage due to the remarkable decrease in SBS in all pre sintered groups. Increase the number of firing cycles from 3 to 5 had no effect on SBS.

Key words: air borne particle abrasion, pre/post sintering, phase transformation, veneer firing cycles, shear bond strength.

I. INTRODUCTION

New all-ceramic materials had been developed to satisfy the increasing patient demand for highly aesthetic and natural-looking metal-free restorations. Zirconia based ceramics had better mechanical properties than the other ceramic biomaterials, allowing them to be used as a substructure for tooth- or implant- supported fixed partial dentures.[1]

Zirconia presented in three crystallographic phases, monoclinic (m) form at room temperature, tetragonal (t) form between 1170° C and 2370° C, and cubic (c) form above 2370° C.[2] It could undergo phase transformation from tetragonal to monoclinic phase during cooling so that, different stabilizers such as CaO, MgO, CeO_2, Y_2O_3 added to zirconia to stabilize the tetragonal form at room temperature. This phase transformation was known as transformation toughening mechanism allowing zirconia to resist further crack propagation via the increase in volume expansion of about 4% from tetragonal to monoclinic phase.[3, 4]

At low temperature tetragonal to monoclinic phase transformation occurred and known as low temperature degradation (LTD).This aging process leading to micro cracks formation

and degradation of zirconia strength.[5]XRD test used to determine zirconia phase transformation as it could provide information about crystal structures and atomic spacing.

Zirconia had an opaque appearance because it was a solid polycrystalline material that lacked the glassy phase. So that, a suitable ceramic veneer should cover zirconia substructure to reach the proper esthetics.[6] Chipping or cracking of veneering porcelain from zirconia framework was the most common clinical failure.[7] The veneering ceramic chipping could be caused by variable factors like: co-efficient of thermal expansion (CTE) mismatch between core and ceramic, firing shrinkage of ceramic, poor wetting by veneering on core, undesired heating and cooling rates, core thickness and veneer thickness.[8]

It was important to establish a durable bond between ceramic veneer and zirconia core as poor bonding can lead to biological and mechanical failures. This reliable bonding of zirconia ceramics could be achieved by increasing surface roughness through utilizing different surface conditioning including grinding, Al_2O_3 airborne particle abrasion, tribochemical silica coating, liners and lasers.

Airborne-particle abrasion was the most widely used surface treatment method for zirconia restorations to enhance micromechanical interlocking of the luting cement by forming micro-sized irregularities which increase the bonding surface area.[9] However, its effect on the mechanical strength of zirconia is still controversial. Some authors concluded that the increase in flexural strength, may be due to the residual compressive stress at the surface as a result of phase transformation,[9, 10] while others reported an unfavorable effect because of substantial surface damage.[11, 12]

In recent investigations, air-abrasion after sintering was found to produce sharp-edged irregularities, which might trigger crack propagation, while rounded form irregularities were observed when air-abrasion was applied before sintering.[13] It has also been stated that performing air-abrasion at the pre-sintered rather than post-sintered stage yields higher surface roughness values with lower monoclinic phase levels.[14] so that, the effect of surface treatment/sintering order on bond strength between zirconia and veneer should be evaluated.

Fabrication of all ceramic restorations requires multiple firing procedures to achieve the proper color, contour and esthetics specially with layering technique. Residual stresses might accumulate during heating and cooling firing procedures due to CTE and cooling rate mismatch between zirconia core and veneering ceramic.[15, 16]

Considering all of the above, the effect of surface treatment/sintering order, as well as number of firing cycles of veneer on shear bond strength of veneered zirconia will be evaluated. The hypotheses of this study were surface treatment/sintering order and increasing number of veneer firing cycles could affect shear bond strength of veneered zirconia significantly.

The null hypotheses tested were that the surface treatment of zirconia at different sintering stages would significantly affect the SBS of veneered zirconia.

The second hypothesis was that different veneer firing cycles could affect the shear bond strength significantly.

II. MATERIALS AND METHODS
1. Specimens grouping:
A total sample size of 40 zirconia blocks was constructed in this study. These blocks were divided into two main groups (N=20) according to surface treatment time whether it was pre-sintering or post-sintering of zirconia blocks.

Group A: Zirconia blocks that received surface treatment before sintering.

Group B: Zirconia blocks that received surface treatment after sintering.

Each group was further subdivided into 2 equal sub groups (N=10) according to the number of firing cycles of veneer either three (A3, B3) or five firing cycles (A5, B5).

2. Specimens preparation:
2.1. Zirconia blocks design and fabrication:
Zirconia blocks were designed by fabrication of a wax pattern with dimensions of (12.54 mm width x12.54 mm length x3.9 mm thickness). Wax was heated by torch and applied into enclosed mold comparable in dimension to the required block, then the wax pattern was scanned by Ceramill map400 (Amman Girrbach AG, Herrschaftswiesen, Austria)to fabricate specimens with standard dimensions.

Zirconia blocks were milled from a blank of partially sintered zirconia (Ceramillzolidht+ white) by Ceramill motion 2 (Amman Girrbach AG, Herrschaftswiesen, Austria)in dry mode using burs that were specially for milling dental zirconia. Finally, 40 zirconia blocks were milled.

2.2.2. Blocks removal from the blank and smoothing:
Zirconia blocks were separated from the blank using a carbide fine point tapered bur(Komet,

Germany) and the projections where the sprues attached were finished by finishing stones. Rubber wheel disc (EVE Ernst Vetter, Germany) size 3.5 was used to smooth the zirconia surface after removal of connectors to produce smooth surface. Zirconia blocks were cleaned using jets of air, then randomly divided among the groups (**Figure 1**). The dimensions of milled zirconia blocks were measured by a digital caliper (150 mm/ 6in/ American spares/ USA). The dimensions were 12.54 mm x12.54 mm x3.9 mm ±0.02 mm.

Figure 1: Zirconia blocks after removal from the blank.

2.3. Air abrasion of zirconia blocks:
Group A was abraded using sandblaster device (Basic eco, Renfert GmbH., Germany) (**Figure 22**). Blocks wereplaced at a distance of 20 mm from the nozzle tip of air-abrasion device and subsequently air-abraded using 50 µm aluminum-oxide particles under 3 bar pressure for 20 seconds. All air-abrasion procedures were performed by a single operator using circular movements during the process.[17] After that, the blocks were steam cleaned to remove debris and dried.

2.4. Sintering of zirconia blocks:
Both group A and group B were placed inside the sintering furnace (CeramillTherm 3, AmmanGirrbach AG, Herrschaftswiesen, Austria) then fully sintered according to manufacture instructions (At 1450° C and a holding time of 2 hours). After sintering, zirconia blocks shrank to dimensions of (10.17 mm x 10.17 mm x 3.03 mm ±0.05 mm) when measured with a digital caliper. Group B was air abraded after sintering following the same procedures used for group A abrasion. X-ray diffraction test was carried out on randomly selected zirconia blocks from each group (three blocks from group A and three blocks from group B) to analyze the phase distribution of zirconia.

2.5. Mold construction for application of veneer material:
A special circular metal mold was designed and constructed to build up veneer with standard dimensions. Its outer part was made of copper, while the inner part was made of Teflon. The mold had a central depression of 5 mm

diameter and fixed 2 mm thickness controlled by a regulator. When the regulator rotated clockwise, a space of 5 mm diameter and 2 mm thickness created to insert the veneering material inside it, then the regulator was rotated anti clockwise to raise the veneering material up. The mold was cleaned and dried before veneer application. (**Figure 2**)

Figure 2: mold made of copper and Teflon with a regulator.
A: Closed mold.
B: The regulator rotated clockwise providing 2 mm thickness and 5 mm diameter for veneering material.

2.6. Application of the veneering material:
The mold was adjusted to receive the veneering material and after the placement of veneering material inside the mold the regulator rotated anti-clockwise to raise it up. Knife was used to remove the veneering material from the mold and place it on the abraded zirconia surface. After that, the veneering material had the following firing cycles.

*Foundation firing:**
Foundation firing was made by the Dentin materials (IPS e.max Ceram). IPS e.max layering material dentin was mixed with the build-up liquid on clean dry surface to form a thin slurry consistency. the mix was applied on the zirconia block with a brush (Fusion 4, HPdent GmbH, Germany) as a thin layer then the disc was held with tweezer and vibration was done using brush to achieve regular and smooth foundation layer. blocks were carefully put on a tray and inserted in the furnace (Programat EP 3010, IvoclarVivadent, Schaan, Liechtenstein). The foundation firing was done according to manufacturer instructions (**Table 1**).

*Dentin firing:**
Dentin material (IPS e.max Ceram) was done after the foundation firing is completed by

mixing IPS e.max layering material dentin with the build-up liquid on clean dry surface to form mix thicker than that of foundation layer. The mix was applied in the mold using a brush (Fusion 4, HPdent GmbH, Germany), then the mold was vibrated to get rid of any air bubbles entrapped in the mix. After finishing veneering of all zirconia blocks, they were put on a tray and inserted in the furnace (Programat EP 3010, IvoclarVivadent, Schaan, Liechtenstein). Dentin firing was carried out according to manufacturer instructions. (**Table 1**)

*2nd **dentin firing (corrective firing):**

Specimens were measured using caliper after first dentin firing and another layer of e.maxceram build-up mix was added to the specimens to compensate for the shrinkage, then 2nd dentin firing was done according to manufacturer instructions (**Table 1**).

Table 2: The recommended firing cycles of IPS e.max Ceram veneer using layering technique.

	Stand-by temperature (°C/°F)	Closing time (min)	Heating rate (°C/°F/min)	Firing temperature T_1 (°C/°F)	Holding time (min)	Heating rate (°C/°F/min)	Firing temperature T_2 (°C/°F/min)	Holding time H2 (min)	Vacuum 1 (°C/°F)	Vacuum 2 (°C/°F)
Wash firing(foundation)	403/757	4:00	90/162	650/1202	0.0	20/36	730/1346	20/36	400°C	650°C
1st dentin firing	403/757	4:00	90/162	`650/1202	0.0	20/36	730/1346	20/36	400°C	650°C
2nd dentin firing	403/757	4:00	90/162	650/1202	0.0	20/36	730/1346	20/36	400°C	650°C
Glaze firing	403/757	6:00	60/108	725/1337	01:10	-	-	-	450°C	724°C

*Glaze firing:

After completion of the previous firing cycles, the veneered zirconia blocks left to cool to room temperature, then a thin glazing layer was applied to the veneered zirconia surface and inserted in the furnace (Programat P500, IvoclarVivadent, Schaan, Liechtenstein) the firing was done according to manufacturing recommendation (**Table 1**). The glaze firing cycle was repeated three or five times (**Table 2**). Finally, the finished specimens were ready for shear bond strength test.

Table 3: The repeated glaze firing cycles for IPS e.max Ceram veneer.

Stand-by temperature (°C/°F)	Closing time (min)	Heating rate (°C/°F/min)	Firing temperature T_1 (°C/°F)	Holding time (min	Heating rate (°C/°F/min)	Firing temperature T_2 (°C/°F/min)	Holding time H2 (min)	Vacuum 1 (°C/°F)	Vacuum 2 (°C/°F)
403/757	6:00	60/108	725/1337	01:10	-	-	-	450°C	724°C

2.3. Specimens fixation:
Specimens were fixed in cylindrical epoxy resin blocks.

2.4. Shear Bond Strength Testing:
Shear bond strength of all specimens of each group was evaluated using universal testing machine. Shear force was applied at the interface between zirconia block infrastructure and the veneering disc with cross –head speed of 0.5 mm/min till fracture (**Figure 3**).

Figure 3: Instron universal testing machine with zirconia block infrastructure veneered with e.maxceram mounted inside.

2.5. Failure mode analysis:

Optical microscope (Olympus SZ61, Olympus, Tokyo, Japan) was used at x12 and x15 magnification to examine all the specimens of the 4 different groups to detect the failure mode. Failure mode can be classified into:

1) Cohesive within zirconia blocks or within veneering ceramic disk.

2) Adhesive failure when fracture occur at the interface between zirconia blocks and veneering ceramic.

3) Mixed failure when both adhesive and cohesive failure occurred.

2.6. XRD Analysis:

The phase distribution analysis was carried out twice: once after surface treatment and sintering and again after the repeated firing cycles. XRD test was applied at 3 randomly selected zirconia specimens from each group using X-ray diffractometry (Empyrean, PANanalytical, Netherlands) that utilized Cu Kα (λ =1.54060Å) radiation source at 40 mA and 40 kV. The scan angle range (2ϴ) of 4°-100° and step size (the angle where the diffractometer moves) of 0.0130°.

The relative amounts of transformed monoclinic phase (Xm) and volumetric fraction (Fm) were calculated based on the methods of Garvie and Nicholson(18) and Toraya et al.[19], as shown in the following equations respectively:

$$Xm = \frac{Im(-111) + Im(111)}{Im(-111) + Im(111) + It(101)}$$

$$Fm = \frac{1.311 \ Xm}{1 + 0.311 \ Xm}$$

Where Im (-111) and Im (111) are the intensities of the m-peaks seen around 28° and 31°, and It (101) is the intensity of the t-peak detected around 30°.

III. RESULTS

Data were collected then analyzed using SPSS program version 26.0. Test of normality (Shapiro Wilk test) results revealed that all data in any tested group was normally distributed. Outliers were assessed by inspection of a boxplot and there was no extreme outliers in any of the 4 sub groups.

1. Shear bond strength evaluation:

One-way ANOVA statistical analysis was used to determine the SBS differences between all tested subgroups, which revealed that there was a significant difference in SBS between the four subgroups (A3, A5, B3, B5) as ($p \leq 0.05$). (**Table 3**).

The mean shear bond strength and standard deviation values of all tested groups are represented in (**Table 3**). The lowest mean SBS was observed in the A5 group (334.1± 74.2 MPa), followed by A3 group (345.8 ± 73.6 MPa), while the highest mean SBS values were noticed in B5 group (447.6 ± 110.8 MPa) followed by B3 group (389.5 ± 108.9 MPa).

Table (3): results of one-way ANOVA to compare SBS between the 4 subgroups

Group	N	Descriptive statistics			Test of significance		
		Mean	SD	SE	F value	P value	Partial η^2
A3	10	345.8^{a,b}	73.6	23.3	3.016	**0.042**	0.201
A5	10	334.1^a	74.2	23.5			
B3	10	389.5^{a,b}	108.9	34.4			
B5	10	447.6^b	110.8	35.0			

Notes: SD=standard deviation. SE=standard error (=SD/square root of sample size). Partial η^2 is a measure of effect size. Post-hoc Tukey HSD test result is presented in letters; the same superscripted letters represented non significant difference, while different letters represented significant difference.

This table shows a statistically significant difference in SBS between the 4 study groups (p≤0.05). This difference has large effect size (Partial $\eta2$ = 0.201, f = 0.501562). Tukey HSD post-hoc test showed thatthe difference was statistically significant between B5 and A5, but not

between the other pairwise comparisons.

2. Effect of Number Firing Cycles of Veneer on SBS in each sintering group:

Independent-Samples t-test was used to compare between SBS of veneered zirconia that had different veneer firing cycles within each sintering group. This test used to compare between (A3 vs A5) and (B3 vs B5).

It was found that increasing the number of veneer firing cycles in each sintering group had no significant effect on SBS. **(Table 4)**

Table 4: Independent-Samples t-test was used to compare between SBS of veneered zirconia that had different veneer firing cycles within each sintering group.

Timing of air abrasion	Number of firing cycles		Test of significance	
	3-cycles	5-cycles	t value	P value
Pre-sintering	345.8 ± 73.6	334.1 ± 74.2	0.352	0.729
Post-sintering	389.5 ± 108.9	447.6 ± 110.8	-1.183	0.252

Notes: Data is mean ± standard deviation. Test of significance is Independent-Samples t-test.
This table showed no statistically significant difference in SBS between the three and five firing cycles in each sintering group.

3. Effect of air abrasion in each firing cycle:

Independent-Samples t-test was used to compare SBS between pre sintered and post sintered zirconia in each firing cycle. It was used to compare between (A3 vs B3) and (A5 vs B5).
It showed that SBS in B5 was significantly higher than A5, but there was no significant difference in SBS between A3 vs B3. **(Table 5)**

Table (5): SBS between Pre- and Post-sintering in each firing cycles

Number of firing cycles	Timing of air abrasion		Test of significance	
	Pre-sintering	Post-sintering	t value	P value
Three-cycles	345.8 ± 73.6	389.5 ± 108.9	-1.052	0.307
Five-cycles	334.1 ± 74.2	447.6 ± 110.8	-2.690	**0.015***

Notes: Data is mean ± standard deviation. Test of significance is Independent-Samples t-test.*statistically significant p ≤ 0.05.
This table showed a statistically significantly higher SBS in B5 vs. A5, but not between A3 and B3.

4. Two-way ANOVA test:

The two-way ANOVA test was used to detect the effect of each variable and the interaction between them. It showed that the interaction effect between timing of air abrasion and firing cycles of veneer was not statistically significant p=0.247 **(table 11)**. Therefore, an analysis of the main

effects was performed. **(table 12)**

Table 11: Results of two-way ANOVA (Interaction effect).

Timing of air abrasion	Firing cycles of veneer	Shear Bond Strength (SBS)		F	P	Partial η^2
		Mean	**Std. Deviation**	1.386	0.247	0.037
Pre-sintering	Three cycles	345.76	73.62980			
	Five cycles	334.1404	74.18868			
	Total	339.9518	72.18515			
Post-sintering	Three cycles	389.4988	108.89047			
	Five cycles	447.5987	110.82781			
	Total	418.5488	111.00911			
Total	Three cycles	367.6310	93.20897			
	Five cycles	390.8695	108.68709			
	Total	379.2503	100.62821			

The interaction effect between time of air abrasion and number of firing cycles on SBS was not statistically significant p=0.247

Table (12): Two-way ANOVA (Main effects).

Main effect	Mean	SE	F	P	Partial η^2
Timing of air abrasion Pre-sintering Post-sintering	339.95 418.55[*]	20.94 20.94	7.047	0.012[*]	0.164
Firing cycles of veneer Three cycles Five cycles	367.63 390.87	20.94 20.94	0.616	0.438	0.017

Notes: Data is estimated marginal mean and standard error (SE). Test of significance is two-way ANOVA (Main effect). [*]P value is significant at <0.05.

This table showed that the analysis of the main effect for timing of air abrasion indicated that the main effect was statistically significant, F = 7.047, p = 0.012, partial η2 = 0.164. SBS was significantly higher post-sintering vs. pre-sintering, while the analysis of the main effect for firing cycles of veneer indicated that the main effect was not statistically significant, F = 0.616, p = 0.438, partial η2 = 0.017. SBS was insignificantly higher in 5-cycles vs. 3-cycles.

3.   Failure mode test:
Optical microscope analysis showed mixed type failure (adhesive at veneer-zirconia interface and cohesive with a small amount of veneer layer remained connected to zirconia face) in the 4 tested groups with no evidence of pure cohesive or adhesive failure in all tested groups **(Figure 4)**.
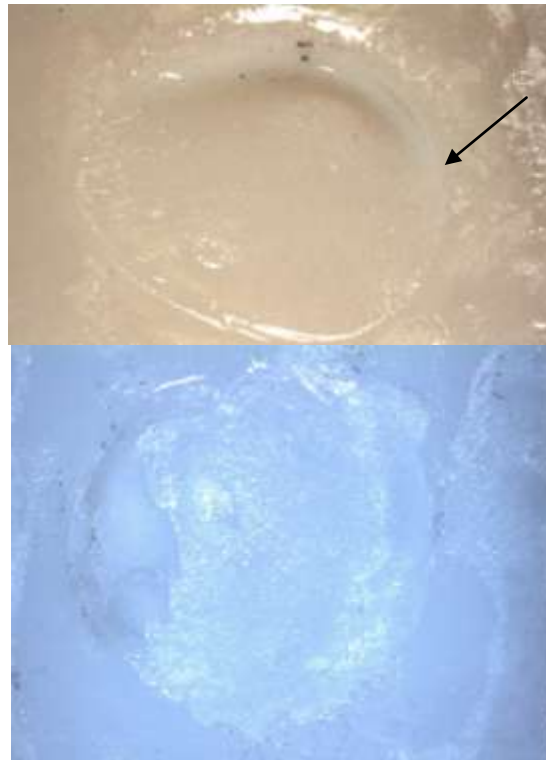
Figure 4: Mixed failure (adhesive & cohesive) under Olympus SZ 30 optical microscope

Phase distribution analysis:

XRD patterns displayed in (**Figure 5**) representing control zirconia group without either surface treatment or firing cycles, pre-sintered zirconia group (A) before application of veneer and post-sintered zirconia group (B) before application of veneer. Monoclinic phase was detected in control group and post sintered zirconia group, while pre-sintered zirconia group was almost free from monoclinic phase.

Highest monoclinic phase amount was detected at control group 0.1% followed by post sintered group without heat treatment 0.02%. This meant that Al_2O_3 airborne particle abrasion caused phase transformation of zirconia from tetragonal to monoclinic and some characteristic peaks representing the monoclinic phase at Im(111) appeared.

Reverse phase transformation from monoclinic to tetragonal was also detected at pre sintered group before veneer application and in all groups after different firing cycles. (**Figure5**)
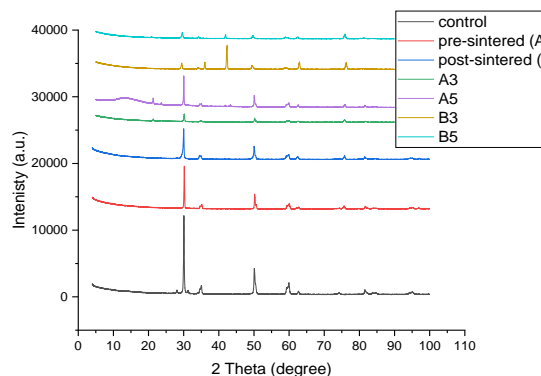
IV. DISCUSSION

This study was designed to evaluate the effect of surface treatment (Al_2O_3 airborne particle abrasion) at different sintering stages (before and after sintering) of zirconia core on shear bond of veneered zirconia. The effect of multiple veneer

firing cycles (3 and 5 firing cycles) on shear bond strength of veneered zirconia was also evaluated.

Based on the results of this study, the first hypothesis stating that surface treatment of zirconia at different sintering stages could affect SBS was accepted because the results exhibited significant difference between subgroups for SBS. The second hypothesis stating that different firing cycles of veneer could affect SBS was rejected as there was no significant difference.

The use of zirconia-based ceramics became very popular in dentistry as they had higher fracture toughness and strength than other ceramic restorations.[20] However, zirconia is opaque so veneering zirconia with porcelain is one of the best options for anterior and posterior single or multiple unit restorations, especially when enhanced esthetics is required as it combined both the zirconia's mechanical properties and porcelain's optical properties.[21]

High fracture rates of the porcelain veneer layer were reported up to 13% in 3 years and 21% in 5 years.[22] Chipping (cohesive failure of the porcelain) and delamination (fracture at interface between Y-TZP and veneer) occurred frequently[23] due to several reasons like: difference in the thermal expansion coefficient between zirconia and veneering ceramic leading to generation of residual stresses, difficulty to enhance bond strength between these ceramic materials, zirconia low thermal diffusivity, as well as the repeated chewing occlusal forces.[24]

Thereby, different procedures have been refined to ensure strong and durable bond strength between zirconia and veneering ceramics. In this in vitro study we utilized Al_2O_3 airborne particle abrasion as it is the most commonly used surface conditioning technique of Y-TZP. This method increased surface roughness, wettability and energy improving bond strength between zirconia and veneering ceramic.[25]

Blasting pressure and particle size significantly affected phase transformation and morphological changes. So that, in this present study, zirconia specimens were air abraded using 50 μm grain size Al_2O_3 particles under 3 bar pressure for 20 seconds and at 20 mm distance as recommended by **Okutan et al.**[17] in agreement with **Grigore et al.**[26] who performed microscopy analysis and concluded that air-abrasion with 105 μm particles under 4 bar pressure created a high concentration of defects and subsurface damage. **Zhang et al.**[27] also suggested air abrasion with 50 μm alumina particles at 0.2 MPa to establish a durable bond for translucent zirconia while increasing the pressure could compromise the

mechanical strength of the ceramic.

Chintapalli et al.[28] on the other hand, claimed that air-abrasion with 110 μm particles and a pressure less than 4 bar improved the flexural strength and reliability of zirconia.

The bond strength between core infrastructures and the veneering ceramics was evaluated using shear bond, 3- and 4-point flexure, tensile and micro-tensile bond tests. Shear bond test had been reported as the most prevalent bond strength test. Different load application strategies resulted in different stress distributions. As a result, unequal stress distributions were possible, and the bond strengths stated were nominal values that should be interpreted with caution. The use of bond strength data derived from static load-to-failure tests should be limited to evaluating the relative effects of material properties, microstructure, and treatment conditions on fracture resistance.[29]

The shear bond strength test was simple and straightforward to carry out. The Accurate measuring of the bond strength at the zirconia core/veneer interface was difficult, and there might not be a suitable test design available right now. Shear bond test, on the other hand, was a standard approach for bilayered zirconia-based ceramic systems. As a result, the shear bond test was employed in this study to analyze the bond strength of veneer ceramic to sintered zirconia cores with modified surfaces at different sintering stages.

In the present study, SBS for post sintered zirconia groups was significantly higher than pre-sintered zirconia group. The XRD examination revealed that Al_2O_3 airborne particle abrasion caused phase transformation from tetragonal to monoclinic (t → m). zirconia specimens that were air abraded before sintering were almost free of m-phase. However, m-phase content was increased in specimens that were air abraded after sintering.

It also revealed that sintering process performed after air abrasion and veneer firing cycles led to a reverse phase transformation from monoclinic to tetragonal (m → t). control group had the highest amount of m-phase followed by post-sintered group while pre-sintered group had the lowest amount which agreed with the findings of **Okutan et al.**[17]

The bond strength between zirconia and veneering ceramic related to two opposing mechanisms: the surface compressive layer resulted from tetragonal to monoclinic transformation could resisted further crack propagation and enhanced the bond strength, conversely pretreatments might cause surface defects and decreased the bond strength. However, the mechanical properties of the material not only depended on this transformation toughening

but also the relation between the depth of the compressive layer and length of micro crack.

Papanagiotou et al.[30] and **Kosmac et al.**[31]explained that increase of the strength of the material was due to the surface flaws which were induced by air abrasion did not exceed the thickness of the compressive layer, while other authors reported decrease in the flexural strength due to crack formation instead of increasing the m-phase content and the length of surface cracks caused by air abrasion largely exceeded the depth of the compressive layer.[17]

In the current study, the post sintered zirconia groups before veneering (B) showed a significant relationship between the amount of phase transformation and SBS. The SBS and Fm value (0.02 %) were significantly higher when compared to the pre sintered group without veneering (A) which had Fm (0 %). The favorable influence of the surface compressive layer on the mechanical characteristics can explain this outcome.

Besides, after multiple veneer firing cycles the pre sintered zirconia groups (A3 and A5) were almost free of monoclinic phase which mean that reverse phase transformation from monoclinic to tetragonal monoclinic to tetragonal (m → t) occurred during the sintering process. Nonetheless, these groups had lower SBS values due to the lack of surface compressive stresses.

These results were also supported by **Yilmaz et al.**[2] who explained the decrease of flexural strength in the pre sintered and heat treated specimens may be due to absence of monoclinic phase content and compressive stress layer due to reverse phase transformation and also presence of irregularities and extensive wear on the zirconia surface under FE-SEM image. **Yilmaz et al.**[9] also found that all aged specimens and non-aged specimens abraded after sintering presented monoclinic peaks. He concluded that pre sintering air abrasion not recommended due to the remarkable decrease of flexural strength, meanwhile air abrasion after sintering enhanced the flexural strength in aged as well as non-aged conditions. There was no strength reduction when air abrasion was applied before and after sintering together.

Passos et al.[32] did not recommend air-abrasion of Y-TZP before sintering with 50- or 110-μm Al_2O_3 to avoid damaging this soft material. This may explain the significant decrease in SBS in pre sintered group in this study. On the other hand, some researchers found that using a lower air abrasion pressure compared to that used in this study before sintering did not cause any significant reduction in flexural strength values when compared with control groups.[14, 33]

Abi-Rached et al.[14] stated that using air abrasion before and after sintering increased the flexural strength of zirconia.

In contrast, **Moon et al.**[13] concluded that airborne particle abrasion before sintering improved the bond strength without compromising flexural strength due to the formation of blunt and melted-round surfaces, while coarse and needle-like defect created when zirconia abraded after sintering and this might trigger crack propagation.

Multiple veneer firing cycles were necessary to reach the desired color, contour and esthetics. The coefficient of thermal expansion (CET) of both zirconia core and veneering ceramic should be similar (less than 1×10^{-6} K^{-1} of mismatch) to avoid crack formation and premature failure.[34] The CTE of ceramillzolidht and IPS e.maxceram used in this study were 10.4 and 10.45 $\times 10^{-6}$ K^{-1} respectively. However, even with matching the CTE of core and veneering ceramic stresses could accumulate due to discrepancy in viscosity, volume and density of ceramic layer during cooling. Residual compressive stresses localized in the veneer layer increased the flexural strength significantly, while residual tensile stresses could trigger or maximize crack propagation inside the ceramic, so that the tensile residual stresses at the interface related to veneer fracture (chipping, cracking or delamination).[34]

In this study, a significant increase in SBS values was observed in the post sintered groups (B5 and B3 respectively), while SBS values was slightly and insignificantly higher in 3 veneer firing cycles vs 5 veneer firing cycles. This means that multiple veneer firing cycles in this current study did not negativelyaffect the zirconia material. However, the analysis of the main effect for number of veneer firing cycles was not statistically significant.

Vichi et at.[35]Observed higher flexural strength values with 2 and 5 veneer firing cycles than with only 1 firing cycle when tested with the porcelain veneer in tension and this might be attributed to stress distribution within the specimens and relief of thermal stresses that accumulated during first firing. When the ceramic was heated back to a temperature higher than that of glass transition, a transformation occurred and the ceramic transformed from solid state to the viscous-liquid state. A structural re-arrangement would occur if the ceramic was kept at this temperature for long enough time allowing the ceramic at a viscous state to relieve some of the stresses produced by the first firing through

diffusive or viscous flow. Furthermore, the ceramic could penetrate the zirconia flaws and wet the surface properly. This mechanism might have occurred in the multiple firings specimens. The subsequent two firings partially relieved residual thermal stresses caused by the CTE mismatch between zirconia and the ceramic veneer, but the fourth and fifth cycles did not improve the flexural strength any further.

Also, **Trindade FZ et al.**[36] stated that more than 3 veneer firing cycles might cause high bond strength between zirconia and veneering ceramic.

In contrast, **Lu et al.**[37] tested 2, 4, 6 and 8 firing cycles and there was no significant difference among the flexural strength means.

The mode of failure analysis was very important to explain bond strength results; adhesive failure indicated good quality interfacial bond with a low amount of voids or defects, Mixed failures indicated intermediate bond strength and cohesive failure indicated high bond strength.

This study presented 100% mixed failure mode in all specimens and this could be related to the difference in flexural strength between core and veneering ceramic materials, so the failure mode shifted from adhesive to cohesive within the ceramic with glassy matrix. During heating if the temperature became above the glass transition temperature (T_g), the ceramic melted and filled the voids and flaws on zirconia surface allowing the material to behave as a monolayer ceramic when subjected to tensile stresses.[34]

Limitations of this study were using only one type of zirconia and veneering ceramic and method for conditioning the zirconia surface rather than combining two methods of surface treatment. Additionally, this study was a laboratory study which lacked simulating the oral environment through thermocycling and cyclic loading and their impact should be evaluated. another limitation was the use of XRD test only to evaluate zirconia phase transformation, but Energy dispersive X-ray microanalysis (EDAX) could also be used to analyze the chemical structure of the tested veneering ceramic. Eventually, further studies should be performed to investigate the effect of combined surface treatment methods before and after sintering, and aging on the shear bond strength of veneered zirconia to confirm or negate the results of this study.

V. CONCLUSIONS

Within the limitation of the present study, the following conclusions were derived:
1. Air-abrasion performed at the post-sintered stage increased the shear bond strength indicating a favorable effect of air abrasion after sintering.
2. Surface treatment performed at the pre sintered stage decreased the shear bond strength and should be used with caution to avoid decreasing the strength of the material.
3. Increasing the number of firing cycles from 3 to 5 had slightly and non-significant increase in SBS.
4. Air abrasion caused tetragonal to monoclinic transformation while sintering and veneer firing cycles caused reverse phase transformation.

VI. RECOMMENDATIONS

It was recommended to apply air abrasion after sintering of zirconia to enhance SBS. And for further investigations it was recommended to Use finite element analysis to evaluate the stress distribution in specimens submitted to SBS test.

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