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Effect of Repeated Firings on Microtensile Bond Strength of Bi-layered Lithium Disilicate Ceramics (e.max CAD and e.max Press)

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ABSTRACT

Aim: To achieve acceptable contour, color, esthetics, and occlusal relations, the porcelain may be subjected to several firing cycles. This study sought to assess the effect of multiple firing cycles on the microtensile bond strength (MTBS) of lithium disilicate-based ceramics (e.max Press, e.max CAD).

Materials and methods: IPS e.max computer aided design (CAD) cores were fabricated using CAD/(Computer Aided Manufacturing (CAM)) technology, and IPS e.max Press cores were fabricated using the heat-pressing technique ($12 \times 12 \times 4$ mm^3). Cores in each group were divided into three subgroups based on the number of firing cycles (three, five, and seven cycles). After porcelain application, the samples were sectioned into microbars and a total of 20 sound microbars in each group were subjected to tensile load in a microtensile tester at a crosshead speed of 1 mm/minute. Microtensile bond strength of the core to the veneering porcelain was analyzed using one-way analysis of variance (ANOVA). Pairwise comparisons were made using the Tukey's test ($p < 0.05$).

Results: In the e.max CAD, the mean MTBS values were 22.07 ± 6.63, 34.68 ± 7.07, and 26.05 ± 10.29 MPa following three, five, and seven firing cycles respectively. These values for the e.max Press were 34.46 ± 9.28, 23.09 ± 5.02, and 31.26 ± 12.25 MPa respectively. There was significant difference in bond strength of e.max CAD ($p < 0.003$) and e.max Press ($p < 0.002$) based on the number of firing cycles.

Conclusion: Increasing the number of porcelain firing cycles decreased the bond strength of the core to the veneering porcelain in both ceramics.

Clinical significance: It is better to decrease the number of firing cycle as much as possible.

Keywords: Bi-layered restoration, Glass ceramic, *In vitro* study, IPS e-max, Lithia disilicate, Microtensile bond strength, Repeated firing.

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INTRODUCTION

Due to innate fragility, ceramic restorations are often fabricated with a high-strength nonesthetic core; for this reason, they are often veneered with more esthetic and translucent but mechanically weaker ceramics. Fracture in these restorations usually occurs at the weakest point, that is, within the veneering porcelain or at the core–veneering interface, resulting in chipping or delamination of restoration.[1] The strength of core and veneering ceramic and also the quality of the bond between the core and the veneer are among the key factors to ensure structural integrity of ceramic restorations following application of functional loads.[2] Such interface stability relates to some factors, such as the wettability of the core by veneering porcelain, attendance of micromechanical retentions, the chemical bonding, and the thermal agreement between core and veneer.[3] So, the core–veneering interface in bi-phasic restorations is subjected to numerous stresses due to difference in the coefficient of thermal expansion (CTE) of the core and the veneering porcelain, the firing shrinkage, and the speed of healing/cooling.[4] Similar to metal-ceramic restorations, in all-ceramic systems the CTE of the core should be slightly higher than that of the veneering porcelain, but in an ideal bi-phasic restoration, all ceramic restorations mismatch in the CTE of the core and the veneering porcelain should not be significant.[5] Evidence shows that adequate metal–porcelain bond strength is over 25 MPa; however, data regarding the bond strength of the core to the veneering porcelain for the newly introduced ceramics are scarce and an accurate test to obtain data regarding bi-phasic all-ceramic dental restorations does not exist.[6]

Application of several layers of porcelain and several firing cycles may be required for the fabrication of all-ceramic restorations with acceptable esthetics, color, contour, and occlusal relations. In restorations with feldspathic porcelain veneering, such as metal-ceramic restorations, the content of leucite crystals increases during firing. Leucite is a high-expansion crystalline phase and can increase the modulus of thermal expansion of porcelain. This increase in the modulus of thermal expansion, along with change in the structure of the veneering porcelain, weakens the structure of porcelain and compromises the bond at the interface in spite of chemical bonding between metallic substructure and feldspathic porcelain veneer.[4]

Lithium-based all-ceramic restorations are produced by the hot-pressing technique. They consist of two crystalline phase (elongated lithium disilicate crystals and lithium orthophosphate) in a glass matrix.[3] The restorations may be finished by glazing or cut back and are veneered with ceramics with compatible modulus of thermal expansion prior to glazing. For this purpose, fluorapatite veneering ceramic (that consists of fluorapatite crystals in a glassy matrix) was introduced for lithium disilicate cores.[6] It seems that the bonding between core and veneering porcelain due to similarity in the chemical composition of the materials would be good.[3]

For the veneering ceramics, such as fluorapatite, the composition of the primary glass, the volume ratio of the size of crystals, and dispersion and morphology of the crystalline phase may be calculated for changes in mechanical properties of ceramics.[7]

Studies on the bond strength of the core to the veneering porcelain and the effect of multiple baking cycles in this regard are rare. Ereifej et al[1] compared the shear bond strength and mode of failure of IPS e.max ZirCAD and IPS e.max CAD ceramics and found no significant difference in bond strength of the two ceramics but the mode of failure in the zirconia was mainly adhesive while the mode of failure was cohesive (in the veneering or core) or mixed (adhesive plus cohesive) in the IPS e.max CAD ceramic. Al-Dohan et al[8] found that the shear strength of tested bi-layered all-ceramics is similar to that of the metal-ceramic restorations. Aboushelib et al[9] evaluated the bond strength of bi-phasic ceramics by microtensile bond strength (MTBS) test and finite-element analysis (FEA), and showed that the bond strength of IPS empress 2 core to its veneering porcelain was higher than that of zirconia core to its veneering ceramic. Tang et al[7] evaluated the effect of 2 and 10 firing cycles on the mechanical properties and microstructure of different zirconia veneering ceramics including IPS e-max Ceram. In all ceramics, density and hardness increased while the porosity decreased after 10 baking cycles. No significant effect on flexural strength, toughness, or microstructure was noted. It is noted that the size and number of fluorapatite crystals in IPS e.max Ceram were affected by the dissolving and ripening effects of multiple firing cycles and it was reported that X-ray intensity of apatite phase depended on the heat treatment temperature.[7]

Now the question is that, in spite of the presence of chemical bonding between core and veneer similar to metal-ceramic restorations, whether repeated firing could weaken the interface in IPS ceramics?

Conspiring the importance of strength and quality of bond between the core and the veneering porcelain in bi-phasic all-ceramic restorations and also the effect of thermal factors in this respect, this study aimed to assess the effect of multiple firing cycles on the core–veneering MTBS in two lithium disilicate-based ceramics (IPS e.max Press and IPS e.max CAD).

MATERIALS AND METHODS

In this *in vitro*, experimental study, IPS e.max CAD blocks (Ivoclar Vivadent AG, Schaan/Liechtenstein) measuring $4 \times 12 \times 12$ mm^3 were fabricated by CAD/CAM technology. The IPS e-max CAD blocks were milled and crystallized in Programat P310 ceramic furnace (Ivoclar Vivadent, AG, Schaan/Liechtenstein). Shrinkage in this process in not significant. Also, IPS e.max Press blocks were fabricated by pressing IPS ingots (IPS e-max Press, Ivoclar Vivadent, Schaan/Liechtenstein) in the space made of resin patterns with the same dimensions (lost wax technique). The properties of used materials are shown in Table 1. The blocks were cast and crystalized in EP 3000 Programat pressing furnace (Ivoclar Vivadent, AG, Schaan/Liechtenstein). Next, the blocks were cleaned with steam jet. Afterwards, the samples in each group were divided into three subgroups for three, five, and seven firing cycles (n = 5).

To standardize the thickness of the veneering porcelain, a cubic aluminum mold measuring $8 \times 12 \times 12$ mm^3 was used. Porcelain (IPS e.max Ceram, Ivoclar Vivadent, Schaan/Liechtenstein) was applied step by step as recommended by the manufacturer (Table 2). Veneering was done by condensation technique and each layer was baked in a Programat P310 ceramic furnace (Ivoclar Vivadent, AG, Schaan/Liechtenstein) until reaching 4 mm thickness (Table 3). Porcelain has 20% shrinkage following firing.

Table 1: Materials used in this study and their properties

Material	*Manufacturer*	*Shade/batch*	*Composition*	*Coefficient of thermal expansion (ppm/°C)*
IPS e-max CAD	Ivoclar-Vivadent, Schaan/Liechtenstein	LT A1, 605318	Glass-ceramic with 70 vol% lithium disilicate crystals ($Li_2Si_2O_5$)	10.45 ± 0.25
IPS e.max Press	Ivoclar-Vivadent, Schaan/Liechtenstein	LT A1, 605273	Glass-ceramic with 70 vol% lithium disilicate crystals ($Li_2Si_2O_5$)	10.55 ± 0.35
IPS e.max Ceram	Ivoclar-Vivadent, Schaan/Liechtenstein	A4/596962	Glass-ceramic veneer with 8.5 vol% fluorapatite crystals $Ca_5(PO_4)3F$	9.5 ± 0.25

Table 2: Veneering procedure in each group

Firing cycles	*Veneering procedure*
3	Wash firing (foundation)/1st dentin & incisal/stain & glaze
5	Wash firing (foundation)/1st dentin & incisal/2nd dentin & incisal (corrective)/stain & glaze/add-on after glaze
7	Wash firing (foundation)/1st dentin & incisal/2nd dentin & incisal (corrective)/3rd dentin & incisal/stain/glaze/add-on after glaze

After completion of porcelain application, the veneered blocks were mounted in polyester for sectioning. The samples were sectioned using a diamond-coated disk under water irrigation (Mecatome T201A, Technimetal, Persi, Grenoble, France). The dimensions of microbars ($8 \times 1 \times 1$ mm^3) were confirmed using a digital caliper with 0.01 mm accuracy. The microbars were evaluated under a stereomicroscope at 40× magnification (SZX9, Olympus, Tokyo, Japan) to verify the integrity. From each group, 20 sound microbars without cracks or internal defects were selected and subjected to MTBS test.

The selected microbars were cleaned in an ultrasonic bath for 5 minutes, rinsed under running water, and dried. The microbars were placed in a microtensile tester (Bisco, Schaumburg, IL, USA) and subjected to tensile load at a crosshead speed of 1 mm/minute until failure. The mean MTBS in each group is shown in Table 4.

To determine the mode of failure, the fracture surface of the microbars was evaluated under a stereomicroscope (SZX9, Olympus, Tokyo, Japan) at 40× magnification. The mode of failure (based on the site of fracture) was divided into four groups of cohesive in the veneering porcelain, cohesive in the lithium disilicate core, adhesive at the core–veneering interface, and mixed in both the core and the veneering porcelain (Table 4). Samples with fractures at the interface were evaluated under scanning electron microscope (SEM) (CamScan MV2300, Oxford, England) at 200× and 600× magnifications to ensure the site of fracture (Figs 1 to 3). Elemental analysis was performed to find fluoride to accurately determine the mode of failure. In these cases, the mode of failure was defined as mixed in core and veneer.

The data were analyzed using Statistical Package for the Social Sciences (SPSS) version 20 (SPSS, Inc., Chicago, IL, USA). Two-way analysis of variance (ANOVA) was used to assess the effects of type of ceramic and number of

Table 3: Layering technique of IPS e.max Ceram on IPS e.max Press or IPS e.max CAD

IPS e.max Ceram on IPS e.max Press or IPS e.max CAD	*Stand-by temperature °C/°F*	*Closing time minute*	*Heating rate °C/°F/minute*	*Firing temperature °C/°F*	*Holding time minute*	*Vacuum 1 °C/°F*	*Vacuum 2 °C/°F*
Wash firing (foundation)	403/757	IRT/04:00	50/90	750/1382	1:00	450/842	749/1380
1st Dentin/incisal firing	403/757	IRT/04:00	50/90	750/1382	1:00	450/842	749/1380
2nd Dentin/incisal firing	403/757	IRT/04:00	50/90	750/1382	1:00	450/842	749/1380
Stain firing	403/757	IRT/06:00	60/108	710/1310	1:00	450/842	709/1308
Glaze firing	403/757	IRT/06:00	60/108	710/1310	1:00	450/842	709/1308
Add-on with glaze firing	403/757	IRT/06:00	60/108	710/1310	1:00	450/842	709/1308
Add-on after glaze firing	403/757	IRT/06:00	50/90	700/1292	1:00	450/842	699/1290

Table 4: Microtensile bond strength and mode of failure according to the firing cycle and core type

	MTBS ± SD (MPa)		*Mode of failure*							
			Cohesive in core		*Cohesive in veneer*		*Adhesive in core–veneer interface*		*Mixed*	
Firing cycle	*e.max Press*	*e.max CAD*	*e.max Press*	*e.max CAD*	*e.max Press*	*e.max CAD*	*e.max Press*	*e.max CAD*	*e.max Press*	*e.max CAD*
3	34.46 ± 9.28	27.07 ± 6.63	0	0	16	16	3	3	1	1
5	23.09 ± 5.02	34.68 ± 7.07	0	0	16	17	3	3	1	0
7	31.26 ± 12.25	26.05 ± 10.29	2	0	14	10	4	5	0	5

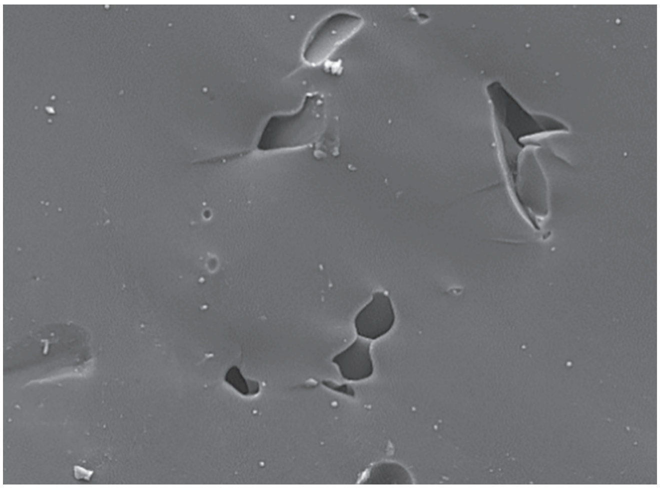

Fig. 1: Scanning electron microscopic image; 600× magnification, adhesive failure in IPS e.max CAD after five firing cycles

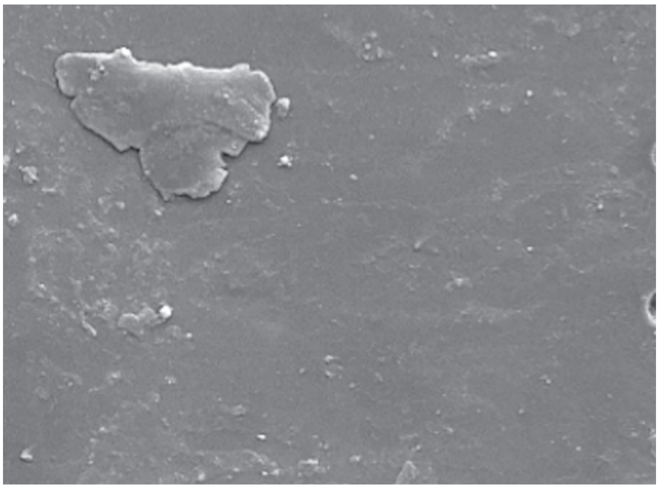

Fig. 2: Scanning electron microscopic image; 600× magnification, mixed failure in IPS e.max CAD after seven firing cycles

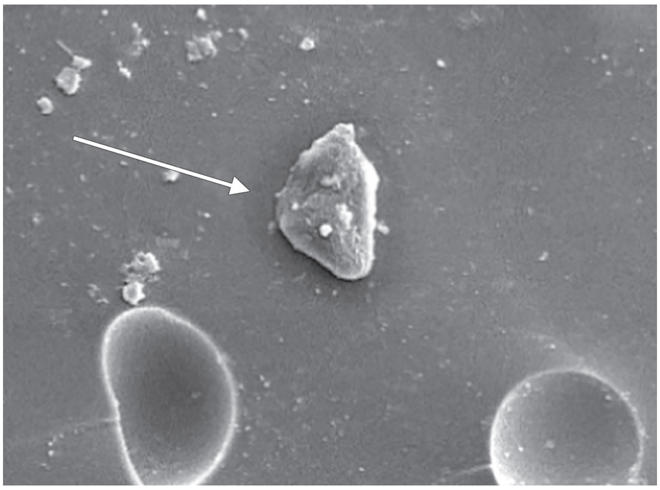

Fig. 3: Scanning electron microscopic image; 600× magnification, mixed failure in IPS e.max Press after seven firing cycle

porcelain firing cycles on the core–veneering bond strength. Also, one-way ANOVA was applied to assess the effect of number of porcelain firing cycles on the core–veneering bond strength of each ceramic. Tukey's test was used for pairwise comparison of core–veneering bond strength of different firing subgroups in each ceramic group. The t-test was applied to compare the core–veneering bond strength values of the same porcelain firing subgroups between the two ceramic groups ($p < 0.05$).

RESULTS

According to the two-way ANOVA, the effects of type of ceramic (CAD and Press) ($p = 0.84$) and multiple porcelain firing cycles ($p = 0.5$) on the core–veneering porcelain bond strength were not statistically significant. However, the interaction effect of the two aforementioned factors on the core–veneering porcelain bond strength was statistically significant ($p < 0.0001$).

In the IPS e.max CAD ceramics, the mean bond strength of the core–veneering porcelain was 27.07 ± 6.63 MPa after three, 34.68 ± 7.07 after five, and 26.05 ± 10.29 MPa after seven firing cycles. One-way ANOVA showed a significant difference in this respect among the three subgroups ($p < 0.003$). The Tukey's test revealed significant differences in the core–veneering bond strength between the subgroups subjected to three and five ($p < 0.01$) and also five and seven ($p < 0.004$) firing cycles but the difference in this respect between three and seven firing cycles was not statistically significant ($p = 0.92$).

In the IPS e.max Press ceramics, the mean bond strength of the core–veneering porcelain was 34.46 ± 9.28 MPa after three, 23.09 ± 5.02 after five, and 31.26 ± 12.25 MPa after seven firing cycles. One-way ANOVA showed a significant difference in this respect among the three subgroups ($p < 0.002$). The Tukey's test revealed significant differences in the core–veneering bond strength between the subgroups subjected to three and five ($p < 0.001$) and also five and seven ($p < 0.02$) firing cycles but the difference in this respect between three and seven firing cycles was not statistically significant ($p = 0.53$).

The t-test revealed no statistically significant difference in equal firing cycles between the two ceramic groups ($p = 0.51$, 0.72, and 0.83 for three, five, and seven firing cycles respectively). The mean bond strength and mode of failure in each group are shown in Table 4.

DISCUSSION

In the current study, MTBS test was preferred to the shear test. Microtensile bond strength test is the most acceptable method of bond strength testing. In general, since the loads are applied vertical to the bonding interface and the possibility of presence of structural defects in the bonding surface is very low (small cross-sectional area of the microbars), results would be more standard. On the other hand, MTBS test may be challenging for the assessment of the core–veneering interface due to the brittleness of the materials and low tensile strength of the veneering porcelain.[10]

Based on the results of the current study, the mean MTBS in IPS e.max CAD group was 29.27 MPa that is comparable with Ereifej's study (29.1 MPa)[1]; also the mean MTBS in IPS e.max Press was 29.6 MPa that is similar to Vidotti's investigation (27.76 MPa).[3] These values are lower than that of Aboushelib's study[9] (IPS Empress 2/Eris: 44.6 MPa, IPS Empress 2/IPS Empress 2:37.2) that could be because of different surface treatment, veneering porcelain, and firing procedure. In Al-Dohan's study[8] the mean shear bond strength in IPS Empress 2/Eris was 30.86 MPa. Regardless of the type of test, in most previous studies and the current study, mean core–veneer bond strength is more than 25 MPa and so is clinically acceptable.

The core–veneering bond strength in both lithium disilicate-based ceramics, including IPS e.max CAD and IPS e.max Press, significantly changed the following multiple porcelain firing cycles. The factors related to the core–veneering bond strength include difference in the CTE of the core and the veneering porcelain, difference in the baking shrinkage of the porcelain, defects in the veneer and poor wetting of the core by the veneering porcelain.[11] One of the most important factors in this regard is the difference in the CTE of the two layers. In most cases, the CTE of the veneering porcelain is changed by the manufacturer to match with that of the core in such a way that the veneering porcelain is subjected to compressive loads during cooling. However, in spite of this, the compatibility of the CTE of the core and the veneering porcelain may change after multiple firing cycles.[12]

In the current study, the pattern of change in the MTBS of the core–veneering porcelain was not the same in IPS e.max CAD and IPS e.max Press ceramics. The IPS e.max CAD after five firing cycles showed higher bond strength compared to that after three firing cycles and then showed a significant reduction in bond strength after seven firing cycles. The bond strength value after seven firing cycles was even lower than that after three firing cycles. In IPS e.max Press the highest bond strength values were noted after three firing cycles. By an increase in the number of firing cycles to five, the bond strength significantly decreased and then increased again by an increase in the number of cycles to seven. And generally by an increase in the number of firing cycles from three to seven, MTBS of the core–veneering porcelain decreased in both lithium disilicate-based ceramics.

Increased MTBS following multiple firing cycles in the current study may be attributed to the release of stresses during the first firing cycle of porcelain. When the porcelain is subjected to multiple firing cycles at high temperatures, it is converted from the solid state to the liquid state. At high temperatures, atoms gain adequate energy, break the bond and rearrange until reaching a stable state.[13] If the porcelain reaches a stable state after rearrangement in high temperature, its viscosity will be suitable enough to allow its penetration into core defects and enable efficient wetting of the surface. Also, the stresses created in the first firing cycle are released. Considering the fact that rearrangement of porcelain structure requires time and thermal energy, cooling speed has a significant effect on the stresses created in the ceramic systems as well.[13]

As expected, in the current study fracture in the veneering porcelain was dominant. On the other hand, based on the current results, by an increase in the number of porcelain firing cycles, the frequency of fractures in the veneering porcelain in both groups decreased, which was probably due to increased density of the veneering porcelain following multiple firing cycles.[7] Thus, the fracture often occurs in the veneering porcelain or at the core–veneering interface. Also, the possibility of fracture within the core is low as confirmed by the results of the current study. Although, core fracture in lithium disilicate ceramics is more common than zirconia but in current study it was not seen. In a study by Al-Dohan et al,[8] it was shown that in the group of Empress 2/Eris 42.8% fractures were in veneer, 8% in core-veneer interface, and 49.2% in core. Ereifej et al[1] showed that fracture pattern in IPS e.max CAD/Ceram was 33% cohesive in veneer, 47% from interface, and 20% cohesive in core. In an investigation by Vidotti et al[3] about IPS e.max Press, 60% of failure was cohesive in the infrastructure and 40% was mixed. The main cause of difference between the results of mentioned studies and the present study is that their test was shear bond strength. The results of this study are along with Aboushelib's study[9] that in IPS Empress 2 core veneered with either Eris and IPS Empress 2 veneers, fracture pattern was entirely cohesive in veneer. It seems that failure mode is more dependent on the type of bond strength test.

This was an *in vitro* study with two types of ceramics and it is recommended that the manner of core–veneer bi-phasic all-ceramic restorations be investigated in a widespread clinical study.

CONCLUSION

Within the limitations of this *in vitro* study, the results showed that by an increase in the number of firing cycles, the bond strength of the core to the veneering porcelain decreased in both IPS e.max CAD and IPS e.max Press ceramics. Subsequently, the frequency of failure within the core or at the interface increased.

CLINICAL SIGNIFICANCE

Slow and long cooling process is preferred for materials with a glass matrix to prevent accumulation of residual stresses. Also clinicians must try to achieve their desired contour, size, and color with minimal firing cycles in all-ceramic restorations because by an increase in the number of firing cycles, the risk of chipping of the veneering porcelain under functional loads in the oral cavity increases.

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