

ORIGINAL ARTICLE

Bonding of lithium-disilicate ceramic to enamel and dentin using orthotropic fiber-reinforced composite at the interface

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Abstract

Objective. To evaluate the effect of orthotropic fiber-reinforced composite (FRC) at the interface on bonding of lithium-disilicate ceramic to dentin and enamel using different adhesive systems. **Material and Methods.** Dentin and enamel surfaces were ground occlusally on human molar teeth. Ceramic blocks of IPS Empress 2 (Ivoclar-Vivadent) were fabricated. Following acid etching and silane treatment of the xceramics, the teeth were divided into two groups (dentin and enamel). Ceramic blocks were bonded to the tooth substance with or without a layer of FRC and dual-polymerizing composite cement (Duolink). Total-etching (etchant (Etch 37) with adhesive (One Step Plus)) and self-etching (self-priming etchant (Tyrian SPE) with adhesive (One Step Plus)) systems were used, with five test specimens in each group. The cement was polymerized with a LED curing unit (Elipar Freelight LED 2) with standard mode of 40 s. The specimens were thermocycled for 6000 cycles and tested with the microtensile tester at a rate of 5 mm/min. Fracture mode analyses were done by light microscope and with SEM. The data were analyzed using three-way analysis of variance (ANOVA). **Results.** ANOVA showed that enamel had statistically significant ($p < 0.001$) higher bond strength values than dentin. Bond strength values were significantly higher ($p = 0.012$) with the total-etching system than with the self-etching system. The existence of FRC also had a minor effect on bond strength values ($p = 0.013$). **Conclusions.** The enamel and total-etching system provided more reliable bonding than dentin and the self-etching system. Use of an FRC layer at the interface did not improve bond strength values, but instead changed fracture pattern behavior.

Key Words: Adhesive, ceramic, fiber-reinforced composite (FRC), microtensile bond strength

Introduction

Glass ceramics have been introduced as an all-ceramic material for single-unit restorations as well as for three-unit FPDs of the anterior region extending to the region of premolars [1]. One of the materials, IPS Empress 2 (Ivoclar-Vivadent), is made of a lithium disilicate framework ceramic and a fluoroapatite layering ceramic [2] and exhibits material properties such as relatively high strength, good esthetics, marginal integrity, and etchability for easy bonding [3].

The introduction of reliable bonding procedures has increased the general acceptance of these ceramic systems [4]. On the tooth side, adhesion to enamel has proved reliable and dentin bonding is more technique sensitive. A major goal has therefore been to simplify the application procedures of the adhesives [5]. Two kinds of two-step adhesive resins

(total-etching and self-etching) are available and used with resin composite cements composed of bisphenol A-glycidyl dimethacrylate (Bis-GMA) or urethane dimethacrylate (UDMA) resin. Resin composite cements are available in light polymerized, auto-polymerized, or dual-polymerizing formulations [6,7].

There has been increasing interest in fiber-reinforced composites (FRCs) in dentistry. The factors that influence the features of FRC are related to the properties of fibers and matrix polymer, impregnation of fibers, adhesion of fibers to matrix, and the quantity and location of fibers. The strength of FRC is also dependent on the fiber direction [8,9]. Design strategies are occasionally employed to provide multi-directional reinforcement, and to minimize the highly anisotropic behavior of unidirectional fiber reinforcement [10]. The reinforcing capacity of fibers can be divided into two or more directions,

known as orthotropic and isotropic, with regard to mechanical properties [8]. The employment of fibers with different orientations can change the dynamics of the adhesive interface and result in a modification of the interfacial bond failures [11]. Some studies on the use of FRCs at the adhesional interface of resin composite and metals have been reported [11–13]. Fiber weaves with fibers in two directions (bidirectional fibers) have been employed at the adhesive interface.

The aim of this study was to investigate the influence of orthotropic bidirectional FRC at the interface on bond strength of lithium-disilicate ceramic on enamel and dentin with composite resin luting cement by using different adhesive systems.

Material and methods

The materials used in this study are listed in Table I. Forty $7 \times 7 \times 5$ mm pressed ceramic blocks of IPS Empress 2 (Ivoclar-Vivadent AG, Schaan, Liechtenstein) were fabricated by the lost wax technique and ingots were injected to an EP 600 furnace (Ivoclar-Vivadent, Liechtenstein, Schaan). The bonding surfaces of the blocks were ground with 220, 360, and 600 grit (Federation of European Producers of Abrasives (FEPA)) in order to standardize the

bonding surface. Ground ceramic blocks were air abraded with $50 \mu\text{m}$ Al_2O_3 particles (Korox; Bego, Bremen, Germany) for 14 s from a distance of approximately 10 mm at 400 kPa with a sand-blasting device (Ar-Ge Dental, Turkey). The blocks were then cleaned in distilled water for 10 min in an ultrasonic bath (Healthsonics, Livermore, California) to ensure a contaminant-free ceramic surface. The blocks were etched with HF acid (Porcelain Etch; Bisco, Schaumburg, Ill., USA) for 2 min, washed, dried, and then silanated (Porcelain Primer; Bisco).

Forty molars extracted from individuals between 16 and 40 years of age were used in this study. The teeth, stored in a 0.5% chloramin-T solution at 4°C for up to 1 month after extraction, were free of caries and restorations. Soft tissues were removed with a scaler (Scaler, H6/H7; Hu-Friedy, Chicago, Ill., USA). After cleaning the teeth with pumice, each tooth was embedded in autopolymerizing acrylic resin (Palapress, Vario; Heraeus Kulzer, Wehrheim, Germany) using a cylindrical plastic mold, 20 mm in height and 20 mm in diameter. Each specimen was ground occlusally with silicone carbide abrasive up to paper no. 1000 (FEPA) under water cooling with a grinding machine (Struers RotoPol 11; Struers A/S, Rodovre, Denmark) to obtain a flat enamel or dentin surface. The teeth

Table I. Materials used in this study

Trade name	Type	Manufacturer	Batch no.	Composition
IPS Empress 2	Lithium-disilicate ceramic	Ivoclar-Vivadent AG, Schaan, Liechtenstein	H14142 Shade 100	SiO 57–80%, Al_2O_3 0–5% La_2O_3 0.1–6.0% MgO 0.0–5.0%, ZnO 0.0–8.0%, K_2O 0.0–13.0%, Li_2O 11–19%, P_2O_5 0.0–11.0% Pigments 8.0%
Duolink	Dual polymerizing resin cement	Bisco Inc., Schaumburg, Ill., USA	0400003526	Bis-GMA ^a 5–30% TEGDMA ^b 5–20% Glass filler 50–80% UDMA ^c 5–15%
One Step Plus	Filled universal dental adhesive	Bisco Inc., Schaumburg, Ill., USA	0400001415	Bis-GMA ^a , BPDMD ^d , HEMA ^e , acetone, glass filler 8.5%
Tyrian SPE	Self-priming etchant	Bisco Inc., Schaumburg, Ill., USA	010802	Primer A: thymol blue, ethanol, water Primer B: AMPS ^f , BISMEP ^g , phosphate, ethanol H_3PO_4 37%
Etch 37	Semi-gel etchant	Bisco Inc., Schaumburg, Ill., USA	0500005395	
Porcelain primer	Silane coupling agent	Bisco Inc., Schaumburg, Ill., USA	0400003530	Ethanol 30–70% Acetone 30–70% Silane 1–10%
Porcelain Etch	Hydrofluoric Acid Gel	Bisco Inc., Schaumburg, Ill., USA	0400003543	HF 4%
Stick-Net	Glass fibre weave	Stick Tech, Turku, Finland	2050712-W-0054	Porous PMMA ^h pre-impregnated bidirectional E-glass fibers
Stick resin	Light curing resin	Stick Tech, Turku, Finland	5504765	BisGMA ^a -TEGDMA ^b

^aUDMA, urethane dimethacrylate; ^bBis-GMA, bisphenol A-glycidyl dimethacrylate; ^cTEGDMA, triethylenglycoldimethacrylate; ^dBPDMD, biphenyl dimethacrylate; ^eHEMA, hydroxyethyl methacrylate; ^fAMPS, 2-acrylamido-2-methyl propanesulfonic acid; ^gBISMEP, Bis (2-(methacryloyloxy)ethyl) phosphate; ^hPMMA, polymethyl methacrylate.

were divided into two groups – dentin and enamel ($n=20$). Each of the groups was further separated into two groups according to the adhesive systems total-etching and self-etching ($n=10$) and these groups in turn subdivided into a further two groups with or without FRC (control group) ($n=5$). The groups were coded as follows: Group DTC (dentin, total-etching, control), Group ETC (enamel, total-etching, control), Group DSC (dentin, self-etching, control), Group ESC (enamel, self-etching, control), Group DTF (dentin, total-etching, with FRC), Group ETF (enamel, total-etching, with FRC), Group DSF (dentin, self-etching, with

FRC), and Group ESF (enamel, self-etching, with FRC). In the total-etching system, etchant (Etch 37, Bisco) was applied to dentin for 30 s, to enamel for 15 s and then rinsed for 15 s, slightly dried and left moist. Afterwards, adhesive (One Step Plus, Bisco) was applied in two coats and air dried. With the self-etching system, the teeth were air-dried for 5 s and self-priming etchant (Tyrian SPE, Bisco) was applied in two coats to dentin for 30 s and to enamel for 15 s. The primer was then air-dried and adhesive (One Step Plus, Bisco) was applied in two coats. The interface was applied with polymethylmethacrylate-dimethacrylate resin impregnated glass fiber weaves



Figure 1. Experimental set-up with special loading device (CR: ceramic, C: cement, PM: polyethylene mold, T: tooth).

Table II. Mean microtensile bond strength values, standard deviations, and failure mode analysis of the groups

Group code	<i>n</i>	Mean	SD	Adhesional ceramic cement	Adhesional dentin cement	Cohesional dentin	Cohesional FRC	Mixed
DTC	12	9.5 (ab)*	3.6	1	9	0	0	2
ETC	21	16.7 (c)*	6.6	5	15	0	0	1
DSC	13	8.5 (a)*	3	1	10	0	0	2
ESC	13	14.3 (bc)*	7.6	0	11	0	0	2
DTF	14	11.1 (abc)*	5.7	2	5	0	6	1
ETF	15	12.7 (abc)*	4.4	2	7	0	6	0
DSF	11	8.3 (a)*	2	0	5	0	6	0
ESF	10	7.1 (a)*	1.7	0	5	0	5	0

*Values designated by the same superscript letter are not statistically significant at $p=0.05$.

(Stick-Net, Stick Tech) to evaluate the previous suggested benefit of the orthotropic FRC layer at the interface.

Five test specimens ($n=5$) were used in each study group. Previous studies have suggested that when the thickness of ceramic inlay is 3 mm or more, light intensity reaching the cement may not be sufficient to complete the polymerization process, therefore autopolymerization is needed [7,14]. Dual-polymerizing resin cement was thus used in the present study (Duolink, Bisco). Polyethylene molds ($0.1 \times 6.8 \times 6.8$ mm) were used and a special loading device (Figure 1) was designed to apply a constant load of 9.2 N to the ceramic block. This load was used to create a uniform resin luting agent layer of approximately 100 μm to simulate the range of film thickness for all-ceramic crowns [15]. Polyethylene molds were also used for standardizing the film thickness. The composite cement was polymerized with an LED curing device with standard mode of 40 s (Elipar Freelight LED 2; 3M Espe, St. Paul, Minn., USA).

Following water storage in distilled water at 37°C for 24 h, the specimens were thermocycled (Comfort Heto Chill Master, Model CB 8-30E; Heto-Holten A/S, Allerød, Denmark) for 6000 cycles between $5 \pm 2^\circ\text{C}$ and $55 \pm 2^\circ\text{C}$ with a dwell time of 20 s and a transfer time of 5 s. The thermocycling process was completed in four and a half days.

Each tooth was serially sectioned into rectangular beams using a slow-speed diamond wafering blade (Ernst Leitz GMBH, Wetzlar 1600, Germany) and then hand-free trimmed with a fine diamond bur, under water cooling, to an hour-glass shape with approximately 1.55 ± 0.18 mm² cross-sectional areas using a digital caliper. The specimens were attached to a micro-tensile tester (Microtensile Tester, Bisco) with a cyanoacrylate glue (Zapit; DVA, AnaHeim, Calif., USA) and subjected to microtensile testing at a crosshead speed of 5 mm/min until they fractured.

Failure modes were observed with a stereomicroscope (Stereomicroscope, Wild M3B; Heerbrugg, Switzerland) and classified as follows: adhesional

failure between cement and dentin, adhesional failure between cement and ceramic, cohesive failure in ceramic, cohesive failure in cement, cohesive failure in dentin, cohesive failure in FRC, and mixed type of failure. One specimen was randomly selected from each group and prepared for SEM analysis. The debonded specimens from each group were gold sputter-coated (Bal-Tec SCD 050 Sputter Coater; Bal-Tec AG, Liechtenstein) and observed with a scanning electron microscope (JSM-5500; Jeol Ltd., Tokyo, Japan).

Following three-factor analysis of variance (ANOVA) (SPSS Inc., Chicago, Ill., USA), Tukey's post hoc test (SPSS Inc.) was used at a significance level of $p < 0.05$.

Results

The mean microtensile bond strength ($\mu\text{-TBS}$) values, standard deviations, and fracture mode analyses of the groups are given in Table II. Three-way ANOVA revealed that the $\mu\text{-TBS}$ values were significantly influenced by the tooth structure ($p < 0.001$). The highest mean $\mu\text{-TBS}$ values were seen in the ETC (16.7 ± 6.6 MPa) and ESC (14.3 ± 7.6 MPa) groups and the lowest in the ESF group (7.1 ± 1.7 MPa). Adhesive system and existence of FRC had minor effects on bond strength values ($p=0.012$, $p=0.013$). However, the effect of the FRC interface layer revealed interaction between tooth structure and fiber layer ($p=0.002$). This effect can be seen in Table II indicating that bond strength values of dentin were not affected by FRC at the interface, whereas bond strength was decreased in enamel groups with FRC at the interface.

In the control group, most of the failures were adhesional in nature at the dentin and cement interface. In the FRC group, also cohesive failures within FRC were observed. SEM micrographs of adhesional failure at the dentin-cement interface and cohesive failure within the FRC layer are shown in Figures 2 and 3.

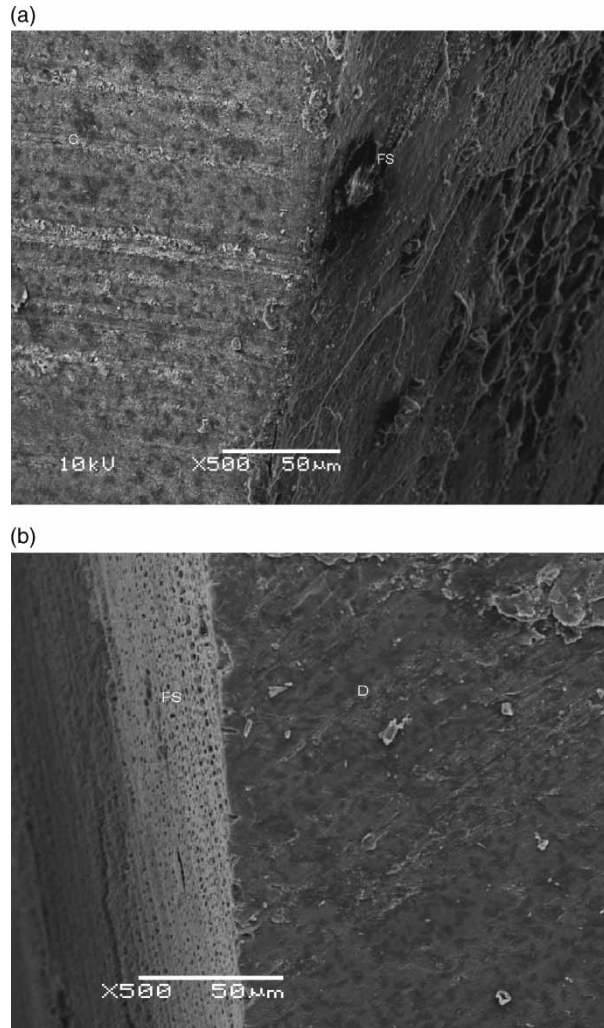


Figure 2. SEM photomicrograph showing the adhesional failure between dentin and cement. (a) Side of cement, (b) side of dentin (C: cement, FS: fracture surface, D: dentin). Original magnification $\times 500$, bar = 50 μm .

Discussion

Microtensile bond strength test was selected in our study because with this test method small dimensions of specimen are used at the interfacial bonding zone and the result is a uniform distribution of the applied stresses [16]. Studies have shown an inverse relationship between microtensile bond strength and bond surface area [17]. Sano et al. [18] explained this behavior as the distribution of defects in the material, because larger specimens contain more defects than do smaller specimens. In order to reduce the bonding area and to produce hourglass-shaped specimens, the method used in the current study readily led to their premature fracture during the cutting procedure. In addition, adhesive systems, which are more susceptible to water sorption, may show lower bond strength values especially after thermocycling. In the present study, a slightly larger cross-sectional area was selected to prevent premature failures during the cutting process and a general

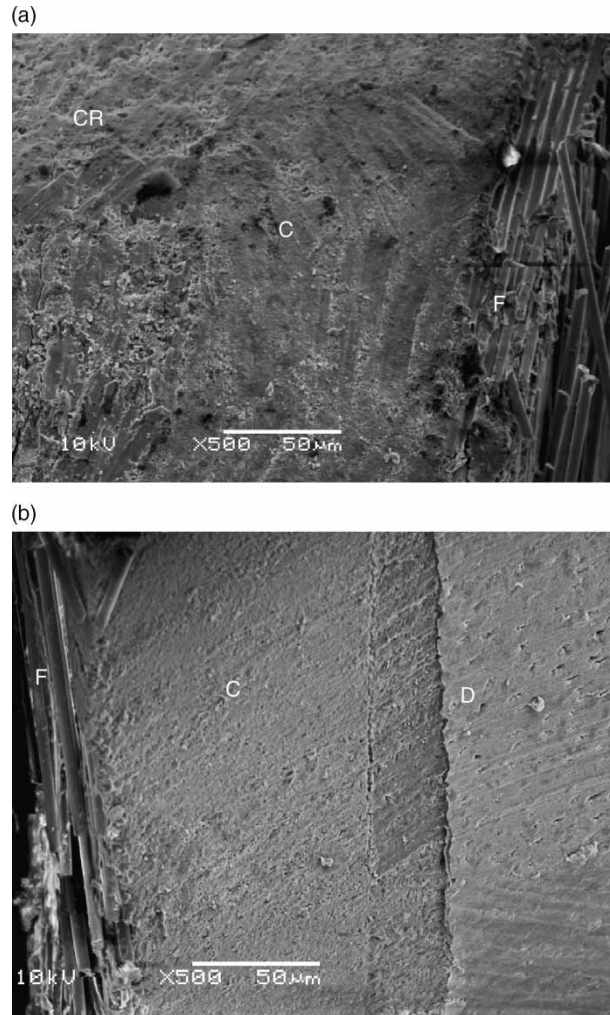


Figure 3. SEM photomicrograph showing the cohesive failure of the FRC layer. (a) Side of ceramic, (b) side of dentin (CR: ceramic, C: cement, F: fiber, D: dentin). Original magnification $\times 500$, bar = 50 μm .

decrease of bond strength values during thermocycling [19].

Several studies have been done on the effectiveness of the self-etching adhesive systems and their adhesion to both dentin and enamel, but controversial results have been reported about the bonding performance of these systems [20,21]. Pilecki et al. [22] concluded that the self-etching adhesive systems produced results similar to total-etch adhesives. Kerby et al. [19], however, reported low bond strength values of the self-etching “no rinse” adhesives such as Prompt L-Pop and Tyrian SPE. Similarly, Can Say et al. [23] observed significantly higher bond strength values with a total-etching adhesive system than with a self-etching system. The findings of Kerby et al. [19] and Can Say et al. [23] are in accordance with this study showing that the specimens with the Tyrian SPE self-etching agent suffered lower bond strength values than specimens with the total-etching adhesive system.

A durable and predictable bond between dental materials and tooth structures is important for clinical success. Carefully done bonding procedures prevent microleakage, caries, and pulpal irritations. Clinical experience has shown that use of the enamel etching technique can provide micromechanical retention and marginal seal of the restorations [24]. Bonding to enamel has become the routine procedure in restorative dentistry [25]. Compared with successful bonding to enamel, dentin is a less favorable bonding structure [24]. Tezvergil et al. [11] suggested that enamel provided higher bond strength values than dentin, which is more technique-sensitive in bonding procedures. In the present study, similarly, the enamel control groups showed higher bond strength values than those of dentin groups.

In the enamel FRC groups, bond strength values were slightly lower compared to those of the enamel control group. This is partially in conflict with a study by Kienanen et al. [26], who showed an increase in bond strength values when adding an FRC layer between the ceramic insert and the resin composite. As opposed to Kienanen et al. [26], Tezvergil et al. [27] concluded that the addition of bidirectional fibers to tooth structure at the interface did not significantly improve the shear bond strength values and that the FRC layer changed the behavior of the fracture pattern. The latter finding is in accordance with the present study.

In dental appliances, FRCs provide handling and esthetic benefits. They are polymeric and can be bonded to tooth structure using current adhesive techniques. Additionally, FRC are metal-free. However, until recently, fiber reinforcement had not met with wide clinical acceptance, perhaps because of the difficulty in handling free fiber bundles. Free fibers tend to unravel and can be difficult to cut [28].

In this study, cohesive failures of the FRC layer were found in increased numbers, suggesting that the cohesive strength of FRC at a 90° angle (antiparallel-in-plane or parallel-in-plane) to the face of FRC layer is lower than the adhesive interface. Parallel-in-plane stress produces an interlaminar type of stress at the interface of fibers and polymer matrix which predisposes to cohesive failure at the FRC laminate. Thus, three-dimensionally oriented fibers at the interface may provide more durable but still flexible bonding allowing stress relaxation in the loading event. This requires further investigation.

Conclusion

It is concluded from this study that enamel and total-etching adhesive systems provide more reliable bonding than dentin and the self-etching system. The use of an FRC layer at the interface did not show any improvement in bond strength values but

instead changed the fracture pattern behavior. An FRC layer can be used to prevent cohesive failures within the tooth structure.

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