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**ORIGINAL ARTICLE**

# Bonding of ceramic insert to a laboratory particle filler composite

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**Abstract**

The push-out bond strength of cylindrical ceramic inserts (CI) to particulate filler resin composite (VC) was evaluated in this study. Various surface treatments to improve the adhesion of CI to resin composite were tested. Additionally, the effect of fiber-reinforced composite (FRC) laminate encapsulation around CI was tested. Feldspathic porcelain CI with a diameter of 3.1 mm was bonded to VC. Adhesive resin was used for bonding. In group 1, no surface treatment of CI was done. In group 2, CI was encapsulated with a thin layer of woven glass FRC. In group 3, the surface of the CI was tribochemically silica coated and silanized. In group 4, the surface of the CI was grit-blasted with 50 μm aluminum oxide and etched with hydrofluoric acid. In group 5, the grit-blasted CI was encapsulated with a layer of FRC. The specimens ($n = 6$/group) were either dry stored or thermocycled in water ($6000 \times 5$–$55^{\circ}$C). The push-out test was carried out with a universal material testing machine. The highest push-out strength was achieved in group 5 (20.4 MPa) and the lowest in group 2 (11.5 MPa). ANOVA revealed that both surface treatment and storage condition had a significant effect on push-out strength ($p < 0.05$). We conclude that the additional glass FRC encapsulation can be used to increase the bond strength of insert to composite.

## Introduction

Resin composites and ceramics have been used for decades in restorative dentistry [1,2]. Both have benefits and limitations when used in inlays, onlays, or crowns [3–8]. The flexural strength of resin composite and conventional ceramics is insufficient for fixed partial dentures. However, ceramics with high strength properties, such as zirconium dioxides and aluminum trioxides, have been introduced to the list of dental solutions, while composites reinforced with fibers (fiber-reinforced composite, FRC) have been introduced for fixed partial dentures [9–11]. It has been suggested that the wear resistance of composites has not fulfilled all clinical requirements, although there has been much improvement in this respect in recent years with the introduction of new filler techniques [12–14]. One additional method for improving wear resistance is based on the use of ceramic inserts at locations of occlusal contact points [15]. A clinically reported problem is that ceramic inserts added mesially or distally are detaching [16]. Prefabricated ceramic inserts like Serana (Nordiska Dental, Ångelholm, Sweden) and Optec (Jeneric/Pentron, Wallingford, USA) are available on the market, but also CAD/CAM-manufactured and dental-laboratory-made ceramic inserts have been designed [17–19]. Some studies have also been published involving the use of ceramics and FRC together [20,21].

Many studies have recently been published regarding the bonding properties of ceramics to resin composite either by modification of the surface properties or by the addition of coupling agents [22–25]. Some information is available suggesting that incorporating a layer of FRC laminate between the materials to be attached can increase bond strength [26,27]. To our knowledge, no studies have been undertaken to evaluate the use of glass FRC encapsulation of ceramic inserts in order to improve their attachment to resin composite.

The purpose of this study was to evaluate surface conditioning methods and the effect of FRC layer encapsulation of ceramic inserts and the push-out bond strength of the insert to particulate filler resin composite.

## Material and methods

The materials for preparation of test specimens are listed in Table I, and the five test groups of this study

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Table I. Materials used in this study

| Brand | Lot no. | Manufacturer | Type of material |
|---|---|---|---|
| Sinfony D4 | 3150783 | 3M ESPE, Seefeld, Germany | Particle filler resin composite |
| Omega Dentin C4 | 3721 | Vita GmbH, Bad Säckingen, Germany | Feldspathic porcelain |
| Modelling Fluid | 7175 | Vita GmbH, Bad Säckingen, Germany | Distilled water |
| StickResin | 304683 | Stick Tech Ltd., Turku, Finland | BisGMA-TEGDMA based light-curing adhesive resin |
| StickNet | 2020218-W-0042 | Stick Tech Ltd., Turku, Finland | Porous PMMA impregnated woven electrical-glass fibre |
| CoJet Sand | 161891 | 3M ESPE, Seefeld, Germany | Silica-coated aluminum oxide, aluminum 95%, silica 5%, particle size 30 μm |
| Espe Sil | 158250 | 3M ESPE, Seefeld, Germany | Silane coupling agent MPTS 1%. Ethanol, water |
| Korox | 401246 | Bego, Bremen, Germany | Aluminum trioxide, particle size 50 μm |
| Porcelain Etch | 56HS | Ultradent, South Jordan, Ut., USA | 10% hydrofluoric acid |

BisGMA = Bisphenol-A-glycidyldimethacrylate; TEGDMA = triethyleneglygoldimethacrylate, PMMA = poly(methylmethacrylate), MPTS = 3-methacryloxypropyltrimethoxysilane.

are summarized in Table II. Laboratory particulate filler composite (Sinfony Dentin, shade 3A; 3M ESPE, Seefeld, Germany) was placed in a stainless steel ring mold (inner diameter 5 mm, thickness 4.2 mm) and photo-polymerized for 20 s for each 1 mm increment with a halogen lamp hand-light-curing unit (Elipar; 3M ESPE) between the two glass plates. Final polymerization of the composite disks was carried out in a light curing oven (Espe Visio Beta, 3M ESPE) in vacuum for 15 min.

After polymerization, the disks were wet ground to a height of 4.0 mm ($\pm$0.1 mm) with silicon carbide grinding paper no. 800 (FEPA; Struers A/S, Rodovre, Denmark). Ground disks were cleaned in distilled water in an ultrasonic cleaning machine (Quatrex 90; L&R Ultrasonic, Kearny, N.J., USA) for 15 min. A hole of 3.2 mm diameter was drilled for the ceramic insert. A 3.5 mm diameter hole was used for inserts, which were bonded by the addition of FRC encapsulation. This allowed having the same thickness of adhesive resin surrounding the insert. The thickness of the resin layer was ($0.08 \pm 0.02$) mm.

Ceramic inserts were made from feldsphathic Omega porcelain powder (Vita GmbH, Bad Säckingen, Germany) and Modelling Fluid (Vita GmbH). A syringe mold with a diameter of 3.1 mm was filled with a mixture of porcelain powder and modeling fluid. A cylinder-shaped ceramic rod was pushed out from the syringe and fired at 930°C in the ceramic oven (Vitamat 200, Vita GmbH). After firing, the ceramic rods were cut into 5-mm pieces and measured, the final diameter of ceramic cylinders being $3.05 \pm 0.1$ mm. The surface of the CI was examined with a scanning electronic microscope (SEM) (JSM 5500; Jeol Ltd., Tokyo, Japan) prior to the adhesive procedure to find the differences in the surface topography after the treatments.

The bonding procedure of CI to the particle composite disk was as follows: group 1 (control) was manufactured without any surface treatments of the ceramic insert by adjusting the measured insert into the hole with adhesive resin. Light-curing adhesive resin (StickResin; Stick Tech Ltd., Turku, Finland) was light polymerized from both sides of the disk with a hand-light-curing unit for 20 s. Final polymerization of the disk with CI was made in a light-curing oven (Espe Visio Beta) for 15 min. The test specimens were then wet ground to a height of 4.0 mm ($\pm$0.1 mm) making both sides parallel. Specimens of all groups were polymerized and ground similarly.

In group 2 a layer of woven fiber-reinforced composite (FRC) (StickNet), thickness 0.06 mm, was resin impregnated (further impregnation) by light-curing adhesive resin (Stick Resin). The FRC weave was then placed on the insert and the FRC capsulated CI was applied to the hole of the composite disk.

In group 3, the surface of the CI was tribochemically silica-coated with an air-abrasion device (CoJet Prep;

Table II. Classification of the test groups according to the surface treatment of the ceramic insert and FRC capsulation (FRC = additional FRC encapsulation)

| Groups | Surface treatment | FRC |
|---|---|---|
| 1 | No treatment | No |
| 2 | No treatment | Yes |
| 3 | Silica particle air-abrasion, Espe Sil | No |
| 4 | $Al_2O_3$ air-abrasion, Porcelain Etch, Espe Sil | No |
| 5 | $Al_2O_3$ air-abrasion, Porcelain Etch, Espe Sil | Yes |

Table III. Results of the analysis of variance for the effect of surface treatment of CI and storage condition on push-out strength

| | d.f. | MS | F | Sig. |
|---|---|---|---|---|
| Storage | 1 | 64.460 | 5.514 | 0.023 |
| Surface treatment | 4 | 174.780 | 14.915 | $<0.001$ |
| Interaction | 4 | 23.946 | 2.048 | 0.102 |

3M ESPE) using silica-coated aluminum oxide (particle size 30 μm) (CoJet Sand; 3M ESPE) at 300 kPa air pressure. The inserts were then silanized with silane coupling agent (Espe Sil; 3M ESPE) and dehydrated for 5 min before adhering the CI to the disk with light-curing resin.

In group 4, the surface of the CI was first air-abraded with aluminum trioxide particles (Korox 50 μm; Bego, Bremen, Germany) using an air-abrasion device (CoJet Prep) at 400 kPa air pressure. The inserts were then etched with 10% hydrofluoric acid (HF) (Porcelain Etch; Ultradent, South Jordan, Ut., USA) for 1 min and rinsed carefully under running water for 30 s. After that, silanization and bonding were carried out as group 3.

In group 5, the procedure followed that of group 4, but an additional resin impregnated woven FRC layer was inserted between the CI and the composite disk equally to group 2.

The test specimens in each group were randomly divided into two storing condition subgroups ($n=6$/subgroup). The storing conditions were dry storing for 6 days at room temperature of $(24\pm1)^{\circ}$C or thermocycling for 6000 cycles between 5°C and 55°C in deionized grade 3 water. After the storage period, the test specimens were push-out tested. A load was applied with a universal testing machine (Lloyd LRX; Lloyd Instruments Ltd., Fareham, UK) to the ceramic insert. Push-out force was recorded with Lloyd Nexygen software (Lloyd Instruments Ltd.) and values were used for calculating the ultimate push-out strength with each specimen.

After loading, the fracture pattern from each group was visually analysed with a light microscope and fracture modes were classified to cohesive ceramic failures, resin, or FRC interface failure, and ceramic resin interface failure.

Two-factor analysis of variance (ANOVA) (SPSS Inc., Chicago, Ill., USA), followed by the Tukey post hoc test (SPSS Inc.), was used to determine the effect of surface treatment and storage condition on bond strength.

## Results

Push-out bond strength values are presented in Figure 1. Bond strength values varied from 10.5 MPa to 23.2 MPa. The lowest value was measured with the thermocycled control specimens (group 1) and the highest bond strength with dry-stored specimens that contained CI with an air-abraded and etched surface with the woven FRC layer (group 5). Thermocycling had a tendency to decrease the push-out strength values. ANOVA and the post hoc test (Table III) revealed that the surface treatment ($p<0.001$) and the storage condition ($p=0.023$) had a significant effect on the push-out strength values.

Fracture modes differed between the groups and are summarized in Figure 2 and clarified in Figure 3.

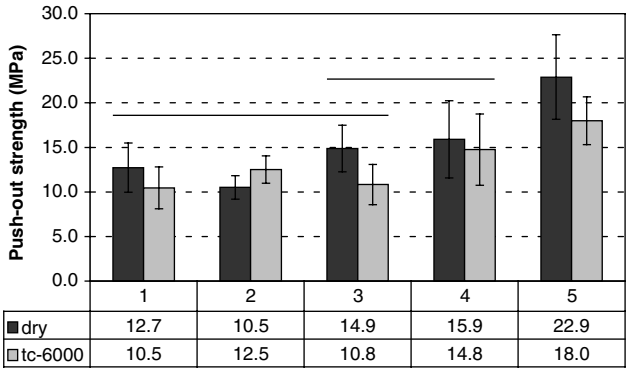


Figure 1. Mean push-out strength of ceramic insert in different groups (1–5, see Table I). Vertical lines represent standard deviation. Bars with horizontal lines did not differ statistically significantly (Tukey post hoc test). Dry refers to dry storage and tc-6000 to the thermocycling.

Groups 1 and 2 (without any surface treatment of CI) revealed adhesion failure between CI and adhesive resin. In group 3, there were mainly adhesive failures between the ceramic and resin interface, whereas group 4 (HF-etched) revealed mainly cohesive failures at CI. In group 5, there were adhesive failures between the FRC layer and particulate filler composite. SEM micrographs of the CI surface topography after different surface treatments are shown in Figure 4.

## Discussion

In this study, we simulated a fabrication process for indirectly made laboratory composite restoration with the addition of ceramic insert. We demonstrated that attachment of the CI to the laboratory composite varied considerably depending on the bonding procedure. The testing methodology of measuring push-out strength was selected because ceramic inserts are applied by compression, which leads to shear stress formation at the interface of CI and resin composite. *In vitro* conditions allowed fabrication of the test specimen with parallel sides, and therefore the test set-up caused shear stress at the interface. Cylindrical shape also causes friction at the interface during load bearing, and progressive debonding was seen in the push-out test curves; this behavior is desirable in the application rather than the sudden load drop which occurs with

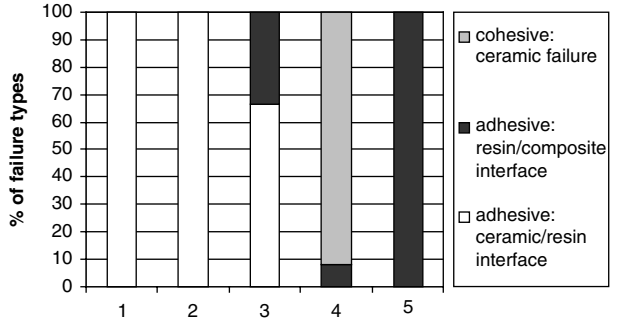


Figure 2. Distribution of the failure modes in groups 1–5.

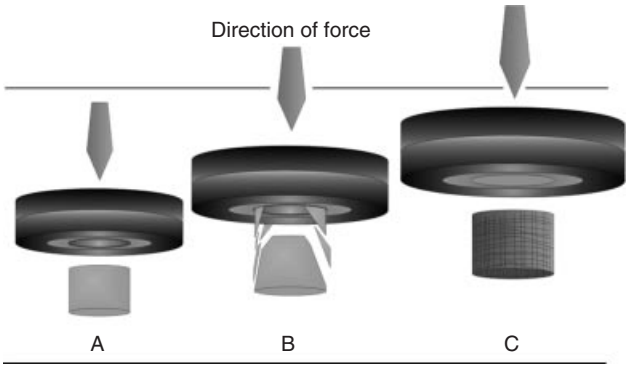


Figure 3. Schematic classification of fracture modes after push-out test. A. Adhesive: resin-composite interface. B. Cohesive: ceramic failure. C. Adhesive: FRC net-composite interface.

conical shape CI. However, it should be noted that although the ceramic insert was equal in diameter in all the groups, the hole diameter varied between the groups with and without the FRC layer. For this reason, the calculated push-out strength values can only be indicative for describing the durability of the attachment of the CI to the particulate filler composite.

It was found that both air abrasion and HF etching, which are known to increase surface area and offer microscopic retention for polymerized resin, improve push-out strength. This is in agreement with studies on ceramic bonding [28]. In groups 1 to 3, the failure type was predominantly adhesive, suggesting that the cohesive shear strength of the materials was higher than ca. 15 MPa. Even when a layer of FRC was added to the untreated ceramic, no change in bond strength nor in failure type was noticed. The woven FRC used in this study was composed of glass fibers and a semi-interpenetrating polymer network (IPN) polymer matrix. The IPN matrix was formed mainly of dimethacrylates, and was plasticized by the presence of polymethylmethacrylate (M.w 220.000) dispersed in the matrix. The modulus of elasticity of the FRC layer was therefore lower than that of the ceramic and surrounding particle filler composite, and could thus have behaved as a stress-breaker interphase.

Failure of the specimens in group 5 was adhesive failure between the FRC and particulate filler composite. It is known that well-polymerized dimethacrylates form a highly cross-linked polymer which does not readily adhere to resin. The factor limiting an increase in bonding of CI to the particulate filler composite thus seemed to be the interfacial bond strength of FRC to particle filler resin composite. However, this problem can be overcome by changing the fabrication process of the specimen or dental restoration.

Fracture modes had significant differences between groups. When CI was given no surface treatment, a fracture line was formed between CI and adhesive, as there was obviously low bonding at the interface. However, two air-particle abrading techniques with CoJet (group 3) or with alumina (group 4) also revealed

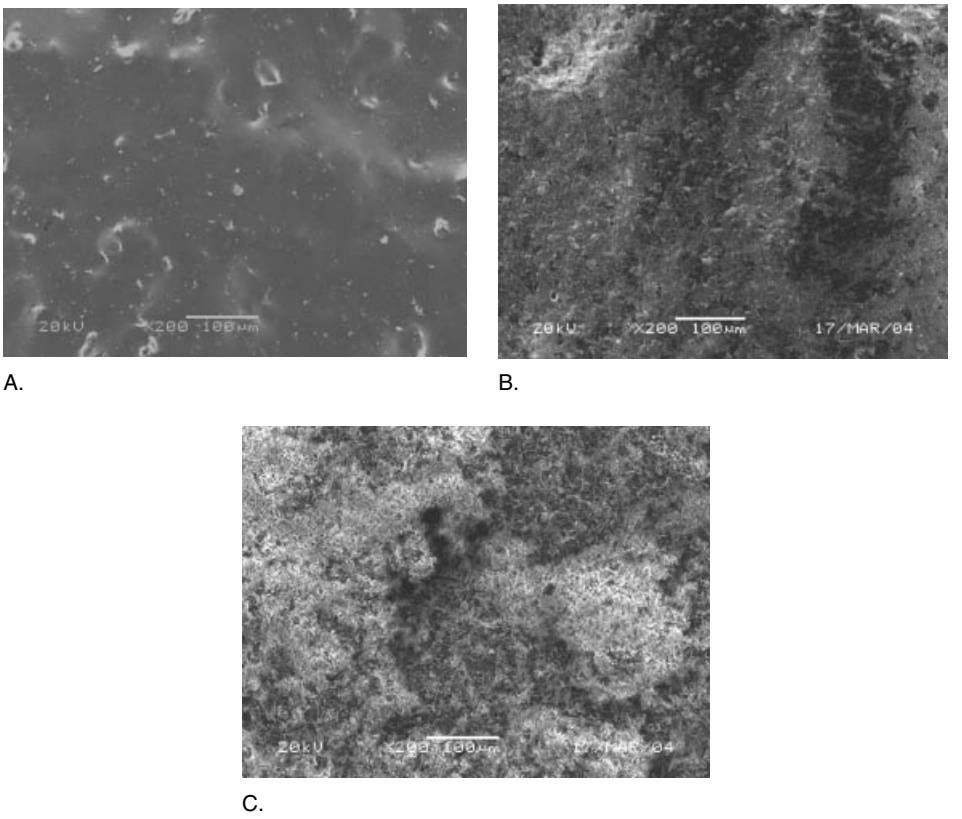


Figure 4. SEM micrographs of the surface of the ceramic insert after surface treatment. A. No treatment. B. Silica particle air-abrasion. C. $Al_2O_3$ air-abrasion with HF etching.

significant differences between fracture modes. The fracture mode in the Cojet group was also adhesive, but the fracture existed partly on the composite resin side. However, group 4, which was alumina grit blasted and etched, revealed a highly cohesive fracture within CI. It could be concluded that the weakest part in that group was the cohesive strength of CI itself. When CI was encapsulated with FRC, the place of the fracture line was changed to between FRC layer and composite, and with this arrangement the fracture load could be increased. A similar finding was made by Fennis, when FRC was used in combination with tooth [28].

It has been shown by Ferracane [29] that marginal breakdown between tooth and filling materials happens due to fatigue. Masticatory forces and filling materials with higher toughness can prevent marginal breakdown. As continuous fibre FRCs are high toughness materials [30], the FRC layer between CI and particulate filler resin composite could analogously be beneficial to marginal integrity when long-term masticatory forces are present.

The FRC layer can also increase stability between the high modulus ceramic and the low modulus composite. When the interface can be formed using materials with gradually lowering modulus of elasticity, high stress concentration peaks can be prevented [31]. The FRC layer can also even out the high stress concentrations between the upper and lower parts of the CI interface during the loading phase [32].

From a clinical perspective, the results of this study provide information about how attachment of a ceramic insert to the particulate filler composite can be increased. The method of adding a layer of woven FRC between the CI and resin composite can be adopted for use.

## Conclusions

Within the limitations of this study, it can be concluded that:

1. By air-abrading and etching the surface of a feldspatic porcelain insert, bonding to the resin composite was increased.
2. By adding a layer of FRC between the air-abraded and etched ceramic insert and the resin composite, the bond strengths were increased even further.

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