CHAPTER 3

Microtensile Bond Strength Testing of Luting Cements to Prefabricated CAD/CAM Ceramic and Composite Blocks.

3.1 Abstract

Objective. To investigate the microtensile bond strength (μTBS) and failure mode of resin cements bonded to composite and ceramic CAD/CAM blocks following various surface treatments.

Materials and methods. Paradigm composite blocks and CEREC Vitablocs received three surface treatments following the control treatment that was a grit 600 SiC ground surface. (1) Application of adhesive resin (Adh), (2) etching with hydrofluoric acid and silanization (HF+S), or (3) combination of the previous two treatments (HF+S+Adh). Three resin cements (Tetric Flow, Nexus 2, RelyX ARC) were applied to these surfaces and built-up in layers. After 24-hour water storage at 37°C, the non-trimming version of μTBS test was used to produce 1mm² microbars. The Microbars were subjected to a tensile load using a modified testing device. The broken specimens were examined with a stereomicroscope and SEM to determine the failure mode.

Results. All control and adhesive treated groups of the ceramic substrate showed premature debonding during cutting. The overall mean μTBS for the three resin cements bonded to ceramic following HF+S and HF+S+Adh surface treatment, was 27 MPa and 29.2 MPa and for the resin cements bonded to composite substrate was 42.3 MPa and 54.2 MPa respectively. The mode of failure was 98% adhesive with composite as a substrate and 68% mixed failures with ceramic as a substrate.

Conclusion. HF followed by silanization is necessary to provide adequate resin-ceramic bond. CAD/CAM restorations fabricated from processed composite blocks may have advantage over the ceramic blocks with regard to the higher bond strength with resin cements.
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3.2 Introduction

The use of indirect esthetic restorations has increased substantially, probably to avoid some of the drawbacks associated with direct composite restorations. Shrinkage stresses in composite restorations generated during setting are still one of the major problems in adhesive dentistry.[1-3] Excessive shrinkage stresses being placed on the tooth cusps due to wall-to-wall contraction may lead to cuspal distortion, marginal discrepancies, postoperative hypersensitivity and microleakage.[4-6] The indirect esthetic bonded restorations can overcome these problems by limiting the contraction stresses of the polymerization reaction to the thin resin-cement layer.[7,8]

Since the end of the 19th century where the porcelain inlays were first described [9], several new materials and techniques for the construction indirect esthetic restorations have been introduced. The CEREC CAD/CAM system (Siemens, Benzheim, Germany) offers the opportunity to prepare, design, and fabricate a ceramic restoration in a single appointment, without the need of making impressions, provisional restorations, or dental laboratory support.[10] Moreover, the CAD/CAM milling of ceramic blocks, fabricated under controlled and optimum manufacturer conditions, enables the production of a restoration with a higher intrinsic strength without the material variations inevitable in laboratory produced restorations.[11] Although the CAD/CAM ceramic material can be produced with mechanical properties superior to those of the conventional feldspathic porcelain, the material is still susceptible to brittle fracture under stress bearing conditions in the oral cavity.

The first composite inlays were made from a microfilled material, which was heat and pressed cured [12], followed-up by an inlay system based on a light cured hybrid composite (DI system, Coltene AG, Switzerland), which was launched in 1987. Several studies have shown that a secondary heat cure of the composite inlay materials included in the fabrication process enhanced the physical and mechanical properties.[13,14] Recently composite inlays, onlays, veneers and crowns can be constructed by CAD/CAM techniques using prefabricated composite blocks manufactured under controlled conditions. The manufacturer claims as advantages over restorations milled from CAD/CAM ceramic blocks, easier finishing and polishing, kindness to the natural dentition with regard to wear and easier to make add-on adjustment.

Various investigations have shown that using adhesive cements increases the fracture resistance of ceramic restorations.[15-17] With the contemporary adhesive cements and the new generation of bonding systems achieving a strong and durable bond to both the tooth structure and the indirect restoration could be feasible.
Bond strength measurements are among the methods used to evaluate the effectiveness of adhesive systems, hence predicting their performance in the oral environment. The shear and tensile strength tests are the most widely used. However, various investigations have reported that the mode of failure, occurring after shear bond testing is often cohesive within the substrate rather than adhesive at the interface. [18-20] Cohesive failures are rarely seen clinically with bonded restorations. Testing the bond strength by tensile loading produces more adhesive failures which may favor the evaluation of the true bond strength. [21] However, the results from this test are greatly influenced by specimen geometry and the occurrence of non-uniform stress distributions during load application. [22]

The introduction of the micro-tensile bond strength test (μTBS test) by Sano et al. [23] has shifted the failure pattern further to occur at the adhesive interface. The small bonded interface of specimens used in this test with approximately 1 mm², results in a more uniform stress distribution during loading. Accordingly, higher bond strength values with fewer cohesive fractures can be obtained. [24,25]

Although several investigations have been carried out to test the bond strength of resin cements to ceramics and laboratory processed composites the methods, which have been used, were conventional test methods. [26-30] The use of μTBS test opens a new field on bond strength testing of ceramic-resin bonds. In addition, there is limited information available about the bonding performance of resin cements to prefabricated composite blocks specially designed for the CEREC system. The purpose of the current study was to investigate the effect of different surface treatments of ceramic and prefabricated composite blocks on the bond strength to resin cements. In addition, the failure mode was evaluated.

3.3 Materials and methods

Ceramic and composite blocks, which are designed for the CEREC CAD/CAM system, were used as substrate materials to bond to. Twelve CEREC Vitablocs Mark II (fine-particle feldspar ceramic blocks, size 112; 12 x 10 x 15 mm Vita, Bad Säckingen, Germany) and twelve 3M Paradigm MZ100 blocks (Z100 resin composite blocks, size 14; Ø =14, 3M Dental products St. Paul, MN, USA) were selected. The surfaces to bond at were wet ground on a polishing machine (Buehler Ecomet V, Buehler Ltd, Lake Bluff, IL, USA) using 600 grit SiC paper. The specimens were then ultrasonically cleaned for 5 min in distilled water and air-dried.
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Table 3.1 Resin cements and adhesives used in this study

<table>
<thead>
<tr>
<th>Cement</th>
<th>Composition</th>
<th>Adhesive</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tetric</td>
<td>Bis-GMA, UDMA, TEGDMA, Ba-Al-flourosilicate</td>
<td>Syntac Single-Component</td>
<td>Maleic acid, HEMA, methacrylate modified polyacrylate acid, initiators, stabilizers, water</td>
<td>Vivadent Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Flow C20284</td>
<td>glass(64%wt), photoinitiators</td>
<td>OptiBond Solo Plus</td>
<td>HEMA, silica, barium glass, sodium hexafluorosilicate, ethanol</td>
<td>Kerr Corp. Orange, CA, USA</td>
</tr>
<tr>
<td>Nexus2 101293</td>
<td>Monomers of methacrylic acid esters, Ba-Al-borosilicat glass, Chemical and photoinitiators</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RelyX ARC BLBL</td>
<td>Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia/silica glass (67.5%wt), Chemical and photoinitiators</td>
<td>Scotchbond 1 0FA</td>
<td>Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid, initiator, water, ethanol</td>
<td>3M ESPE, St. Paul, MN, USA</td>
</tr>
</tbody>
</table>

Before application of the three cements of Table 3.1 to each of the twelve specimens, Vitalblock or 3M Paradigm, the surfaces to bond to were treated in one of the following ways:

Treatment 1: no surface treatment (control).

Treatment 2: a thin layer of adhesive (Table 3.1) was applied with a brush and then light cured for 20 seconds with a Translux CL (Kulzer & Co. GmbH, Wehrheim, Germany).

Treatment 3: 8% hydrofluoric acid (HF) was freshly prepared and applied for 2 min, washed thoroughly for 1 min under tap water, dried with air and treated with silane primer Monobond-s (Vivadent, Liechtenstein) for 60s and air-dried.

Treatment 4: same as treatment 3, but with a thin layer of adhesive applied to it, which was light cured for 20 seconds with the Translux CL (Kulzer & Co. GmbH, Wehrheim, Germany).

A survey of the treatments is given in Table 3.2.
Table 3.2 The twelve surface treatments for each of the twelve Vitablocs and twelve 3M Paradigm specimens before application of the cement (HF concentration was 8 % and the silane agent was Monobond-s).

<table>
<thead>
<tr>
<th>Treatment code</th>
<th>Surface treatment steps</th>
<th>Cement</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Step 1</td>
<td>Step 2</td>
</tr>
<tr>
<td>No treatment</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Adhesive</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>HF + S</td>
<td>HF</td>
<td>Silane</td>
</tr>
<tr>
<td>HF + S + Adhesive</td>
<td>HF</td>
<td>Silane</td>
</tr>
<tr>
<td>No treatment</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Adhesive</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>HF + S</td>
<td>HF</td>
<td>Silane</td>
</tr>
<tr>
<td>HF + S + Adhesive</td>
<td>HF</td>
<td>Silane</td>
</tr>
<tr>
<td>No treatment</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Adhesive</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>HF + S</td>
<td>HF</td>
<td>Silane</td>
</tr>
<tr>
<td>HF + S + Adhesive</td>
<td>HF</td>
<td>Silane</td>
</tr>
</tbody>
</table>

The selected resin cement was applied and built-up in layers to a total thickness of 5 mm. Each layer was light cured for 40 sec using the Translux CL. The light power density was 400-450 mW/cm² measured with the Radiometer (Demetron Research Corp, Danbury, CT 06810).

The built-up specimens were stored in distilled water at 37 °C for 24 hr. Using a low speed cutting saw (Buehler Isomet 1000 Low Speed Saw, Buehler Ltd, Lake Bluff, IL, USA), each specimen was cut into slabs of 1 mm thickness, starting at the cement side, through the ceramic or composite block perpendicular to the bonded interface. The cutting advanced until 1 mm remained in order to keep the slabs fixed in position. The block of slabs was then rotated 90° and again cut perpendicular to the bonded interface to gain 1 ± 0.1 mm² rectangular microbars. During this second cutting procedure, premature debonding of the cement from the substrate occurred in a few cases, but for the ceramic controls in all cases. The survived microbars per built-up specimen were counted after cutting. Ten microbars were selected randomly and there cross-sectional areas were measured with a digital caliper (Mitutoyo Corp., Japan) before testing. The other remaining microbars were left without testing. The microbars were glued to a testing device (Figure 3.1) by means of a light curing adhesive (Clearfil SE Bond, Kuraray Co., Japan).
Figure 3.1 A) Schematic illustration showing a microbar fixed to the modified Microtensile testing device. The upper stationary part (a) is connected to the lower articulating part (b) through a 0.35 mm thick brass sheet. The brass sheet allows hinge movement between (a) and (b) when the force (F) is applied to (b) via a rod and ball. B) Cross sectional view of the testing device.

**Testing device**

The device to test the μTBS was specially designed to facilitate accurate alignment of the microbar with the applied force during testing. The device is composed of two stainless steel articulating members, which are attached to each other at one end by a 0.35 mm thick brass sheet (Figure 3.1). This attachment permits hinge movement of the two parts and ensures application of a pure tensile force to the microbar specimen, which is glued to the free ends of the device. By using a universal testing machine (Model no. 6022; Instron, High Wycombe, Bucks, U.K.) at cross head speed of 1 mm/min, a force is applied to the lower member via a steel ball, which loosely fits in an outlet in the upper member. The pitch distance from the ball to the hinge is 80% of the distance from the specimen to the hinge and in order to obtain the forces exerted on the specimen, the measured forces had to be multiplied by a value of 0.80. The design of this device ensures to a great extent elimination of bending that might occur from the misalignment of the applied load to the specimen. Although the circular opening imposed by the hinge movement causes the only bending that might occur, this could be safely neglected due to the fact that the thickness of the specimens (approx. 1 mm) is about 2% of the length of the members (50 mm) so, the stresses exerted at the outer surface of the specimen is only 2% greater than at the inner surface. When mounting a specimen, a small force of 0.3-0.5 N from the weight of the lower member has to be corrected for.
To determine the mode of failure, all specimens were observed immediately after fracturing under a stereomicroscope. The fractured surfaces were classified according to one of the following types: A = adhesive failure at the substrate-cement interface; B = cohesive failure in substrate; C = cohesive failure in cement; D = mixed A and B and E = mixed A and C. For failure modes that could not be accurately established under the stereomicroscope, the surfaces were examined in the scanning electron microscope (Phillips SEM XL 20, Eindhoven, Holland).

Statistical analysis

Statistical analysis was carried out using SPSS statistical software package 9.01 (SPSS Inc., Chicago, IL, USA). Analyses of variance were performed with the bond strength as the dependant variable. Type of substrate (ceramic or composite), substrate surface treatment (No treatment, Adhesive, HF + S or HF + S + Adhesive) and cement type (Tetric Flow, Nexus 2 or RelyXX ARC) were treated as between subject factors. Whenever interaction or main effects were significant, they were further analyzed by simple effects and simple pairwise comparison. Statistical significance was set at $\alpha = 0.05$ for all tests.

3.4 Results

The three-way ANOVA showed that the interaction between the type of substrate, substrate surface treatment and cement type is significant ($P < 0.001$). The means and standard deviations of the microtensile bond strength of all tests are compiled in Table 3.3.

The groups that did not receive a surface treatment (control) and those where only an adhesive was applied to the surface were not included in the analysis, because no intact microbars could be obtained with the Vitablocs as the substrate. In these cases, debonding between the cement and the Vitablocs ceramic substrate occurred for all microbars during the cutting procedure. Further analysis was done by means of simple effects and simple pairwise comparisons. The results show that for similar treatments, significantly higher bond strengths were obtained with the 3M Paradigm composite than with the Vitablocs ceramic ($P < 0.001$) except for the HF + S, Tetric Flow combination. When analyzing the effect of using different surface treatments for the Vitablocs ceramic substrate on the bond strength within the three types of cements, it was found that only the HF + S + Adhesive treatment with Nexus 2 cement gave a significantly higher bond strength ($P < 0.001$). For the 3M Paradigm composite substrate the average bond strengths were significantly higher with the HF + S + Adhesive surface treatment than with the HF + S treatment for the RelyX ARC as well as for the Tetric Flow cement ($P <$
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0.001 in both cases). On the other hand there was no significance difference between the HF + S + Adhesive and the HF + S surface treatments for the Nexus 2 cement \((P > 0.001)\).

**Table 3.3** Means and standard deviations of the µTBS test of three cements bonded to Vitablocs ceramic and Paradigm composite after various surface treatments \((n = \text{number of microbars tested})\).

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Cement</th>
<th>Treatment</th>
<th>Mean (SD) MPa</th>
<th>(n)</th>
<th>Lost microbars (^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vitablocs</td>
<td>Control</td>
<td>(^b)</td>
<td>28.7 (5.9)</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>24.7 (7.1)</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>Nexus</td>
<td>Control</td>
<td>(^b)</td>
<td>24.3 (3.1)</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>36.6 (6.5)</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>Rely X ARC</td>
<td>Control</td>
<td>(^b)</td>
<td>27.6 (8.0)</td>
<td>10</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>26.5 (10.9)</td>
<td>10</td>
<td>34</td>
</tr>
<tr>
<td>3M Paradigm</td>
<td>Control</td>
<td>(^b)</td>
<td>14.1 (4.7)</td>
<td>10</td>
<td>41</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>53.5 (10.5)</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>Nexus</td>
<td>Control</td>
<td>(^b)</td>
<td>21.9 (4.1)</td>
<td>10</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>41.2 (13.1)</td>
<td>10</td>
<td>11</td>
</tr>
<tr>
<td>Rely X ARC</td>
<td>Control</td>
<td>(^b)</td>
<td>41.4 (7.6)</td>
<td>10</td>
<td>56</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>(^b)</td>
<td>39.6 (10.4)</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

HF: Hydrofluoric acid (8%), S: Silane primer Monobond-s (Vivadent, Liechtenstein).

\(^a\) The maximum number of microbars obtainable after cutting a Vitablocs built-up and 3M Paradigm built-up was 56 and 70 respectively.

\(^b\) No microbars were available due to premature debonding during cutting.

For the 3M Paradigm composite substrate, Two-way ANOVA was carried out individually. In this second analysis, all groups were included except the control group, because debonding between the RelyX ARC cement and the composite occurred for all microbars during the cutting procedure. The results show that the treatment by cement interaction effect is significant \((P <\)
Further analysis was done by means of simple effects and pairwise comparisons. The results indicate that regardless of the type of cement used, the bond strength is greater (P values ranging from $P < 0.001$ to $P = 0.014$) or at least not significantly smaller for the HF + S + adhesive treatment compared to the two other treatments.

An additional Two-way ANOVA was carried out for the 3M paradigm composite substrate with Tetric Flow and Nexus 2 as cements. RelyX ARC cement was excluded, because there were no specimens in the control group. The pairwise comparisons tests revealed significantly lower bond strength values for the control untreated groups in comparison with all other treated groups ($P < 0.001$ in all cases).

The numbers of lost microbars during cutting within each of the groups are given in Table 3.3. It shows complete loss of microbars during cutting of the Vitablocs substrate control and adhesive treated groups of the three tested cements. For the 3M Paradigm, only the control group of the Rely X ARC cement showed total loss during cutting.

The distributions of failure modes are listed in Table 3.4. Microbars with the Paradigm composite as substrate showed predominantly an adhesive type of failure (98 %) after testing, while specimens with the Vitablocs ceramic as substrate showed 30 % adhesive failures and 68 % mixed failures (adhesive and cohesive). The total percentage of adhesive failures for both the ceramic and composite substrates was 74%.

<table>
<thead>
<tr>
<th>Type of failure</th>
<th>Substrate</th>
<th>Total (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Vitablocs (%)</td>
<td>3M Paradigm (%)</td>
</tr>
<tr>
<td>A = adhesive: substrate-cement</td>
<td>30.0</td>
<td>98.2</td>
</tr>
<tr>
<td>B = cohesive: substrate</td>
<td>1.7</td>
<td>0</td>
</tr>
<tr>
<td>C = cohesive: cement</td>
<td>0</td>
<td>1.8</td>
</tr>
<tr>
<td>D = mixed A and B</td>
<td>28.3</td>
<td>0</td>
</tr>
<tr>
<td>E = mixed A and C</td>
<td>40.0</td>
<td>0</td>
</tr>
</tbody>
</table>

SEM observations of the fractured surfaces of the bonded ceramic specimens that exhibited mixed type of failure showed remnants of ceramic, or cement that tend to remain attached to the corners of the bonded interfaces (Figure 3.2a). For fractured composite specimens SEM observations showed clear adhesive failure at the bonding interface (Figure 3.2b).
Figure 3.2 a) SEM Micrograph showing type D failure (mixed adhesive and substrate cohesive) at resin cement side, elements of fractured ceramic can be observed at the corners and the periphery of the specimen (arrow). b) SEM Micrograph showing type A (Adhesive) failure frequently took place with the composite substrate.

3.5 Discussion

The shear test, which is frequently used to determine bond strengths, often produces cohesive bulk fracture of the substrate away from the bonding interface. This way of fracturing gives only limited information about the true bond strength.[20] The frequent unpredictability of the mode of failure is caused by surface flaws, internal material flaws in the substrate material, the adhesive layer, or the bonded composite and flaws in the interface.[21] In addition the results of the shear test, but the tensile bond test as well, are greatly influenced by the non-uniform distribution patterns of the applied stress with stress concentrations at certain sites of the specimens.[31] On the other hand, with the μTBS, the small dimensions and small interfacial bonding zone of the specimens decrease to a great extend the number of these defects and result in a more uniform distribution of the applied stresses.[25]

The modes of failures evaluated in this study after μTBS testing the resin cements bonded to ceramics showed that the majority of the fractures were through the adhesive interface. Only one specimen fractured cohesively within the bulk of the ceramic substrate. This is in agreement with Della Bona et al.[32], who found that most of the failures obtained from μTBS testing of composite bonded to hot-pressed ceramics materials occurred within the adhesion zone. A finite element analysis study for rectangular specimens carried out by Phrukkanon et al. [33] revealed that stresses are concentrated at the corners and the central area between the corners of the adhesive interface. This explains the predominant mixed type of failure in our study where remnants of ceramic, or cement tend to remain attached to the corners of the bonded interfaces.
(Figure 3.2a). On the other hand, the types of failure observed from the composite groups were predominately adhesive, which can be attributed to the more resilient composite substrate (Figure 3.2b). The adhesive failures should be considered as apparent adhesive failures. Establishing true adhesive failures requires more than only fractographic analysis.

Various methods have been investigated in the literature for surface treatment of substrates prior to resin composite bonding. Mechanical roughening with course diamond, airborne particle abrasion with aluminum oxide and etching using different types of acids are all among the methods used to enhance micromechanical retention.[34] However, mechanical surface preparation of the substrate material by air abrasion induces chipping and adversely affects the fit of CEREC, CAD/CAM restorations.[35] HF is commonly used to etch porcelain for indirect restorations.[36,37] As alternatives to avoid the hazards HF, acidulated phosphate fluoride [38], or phosphoric acid were also investigated. However, their effectiveness on the enhancement of the bond strength still doubtful.[39] The benefit of HF is that it creates surface pits for micromechanical attachment by preferential dissolution of the glass phase from the ceramic matrix.[40] Treatment of the etched surface with silane improves the wettability and forms a covalent bond with both the porcelain and resin cement.[41]

The results of the current study support the importance of HF etching and silanization for treating ceramic surfaces, as no surface treatment or applying only an adhesive to the surface resulted in debonding of all the specimens during the cutting procedures (pre-testing failures). The finding that there was no significant difference between the three ceramic groups after HF + S surface treatment, indicates that this surface treatment had the main effect for establishing the bond regardless of the type of the cement used.

When adding a thin layer of adhesive to the HF + S treated ceramic surfaces the bond strengths of Tetric Flow and RelyX ARC did not increase. Also Stangel et al. [42] did not find an increase after application of a dentin adhesive to HF + S treated porcelain. Only the adhesive OptiBond Solo Plus resulted in significant higher bond strength for the cement Nexus 2. This can be attributed to the filler containing OptiBond Solo Plus adhesive. Previous investigations have shown the significance of using filled adhesives over unfilled adhesives in increasing bond strength to dental structures.[43-46]

The CAD/CAM composite blocks for the CEREC system were produced as an alternative to the ceramic blocks. However, bonding of resin cements to preprocessed composite blocks may be challenged by the reduced number of reactive sites due to the high degree of double bond conversion.[47,48] When the composite substrate in this study was treated with a thin layer of adhesive a significant increase in bond strength compared to the non-treated control groups was
observed. Several studies have shown the positive influence of using adhesives as an intermediary resin treatment for composite repair.[49-52]

HF is also used to treat composite surfaces prior to bonding, as it has a roughening effect by preferentially attacking the exposed ceramic filler. An additional silane treatment of the surface would further enhance the bond strength, as the filler particles at the surface are potential sites for silanization. This study showed varying results. Where the bond strength for the Nexus 2 cement was significantly higher after a HF + S treatment, that of RelyX ARC was the same and that of Tetric Flow cement decreased significantly. This latter result corresponds with previous studies, which also demonstrated a significant decrease in bond strength after HF etching of indirect hybrid resin composite.[28,53-55] The decrease was explained by the aggressive etching effect of HF that partially degrades the resin matrix and may cause total dissolution of exposed filler particles.[53]

The significant higher bond strength, which was found after adding an adhesive to the HF + S treated surface of the composite could be related to the effect of the adhesive of infiltrating and repairing the damaged resin matrix as well as increasing the wettability of the treated surface. A comparable positive effect on the bond strength of adhesive application on composite surfaces after aluminum oxide air abrasion was reported by Latta et al.[28] Like HF air abrasion may lead to surface defects, which can be repaired in a similar way.

The present study shows strong evidence that the HF + S + adhesive treatment of composite substrates for CAD/CAM use is the most consistence type of treatment. The small differences in bond strength values between the three brands of resin cements bonded to the preprocessed composite are most likely due to the differences in composition, filler type and filler concentration of the cements.

The current study clearly shows significantly higher bond strength values for bonding resin cements to processed composite than bonding these cements to ceramics. This difference has to be related to the mechanical differences between the composite and the ceramic. The more brittle ceramic material tends to fracture at the adhesive interface at lower values than the more resilient composite. Also, differences in the elastic modules between the two materials could play a role. A study by Van Noort et al. [31] showed that the higher the elastic module of the material the higher the stresses generated at the edge of the bonding interface.

As an approach to evaluate the reliability and consistency of the bond throughout the entire bonded surface of the composite and ceramic blocks, the number of microbars for which the cement debonded from the substrate during the cutting procedure was counted (Table 3.3). The RelyX ARC cement recorded the highest number of premature debonded specimens during
cutting in comparison to the other two tested cements, although the recorded bond strength for that cement was not the lowest. Premature debonding during cutting and regional differences in bond strength has been reported in previous investigations.[56-58] Various factors could bring about these differences in bond strength like the influence of the cutting procedure [25], the heterogeneous structure of the substrate [59], differences in material properties, technique sensitivity of the bonding system and the operator variability.[57] Several conditions in the present study were more favorable, like the more homogenous structure of the substrates as compared to tooth tissue and the use of the non-trimming version for specimen preparation, which places less stress on the bonded interface during the cutting procedure.[25] Yet, a relatively large number of pre-testing failures occurred during cutting. Besides the factors already mentioned a phenomenon of curling up or warping of the resin cement may occur during building up the cement block. Warping of composite material has been observed previously in a study into linear polymerization shrinkage.[60] Curling up of the cement, away from the adhesive surface is caused by shrinkage stresses, which are developing at the adhesive surface.[61] The microscopic detachment of the cement specimen is often not noticed. 

This study showed that the μTBS test is a reliable test for testing resin cements bonded to ceramics or processed composite substrates. Higher bond strength values could be obtained with CAD/CAM composite blocks than with CAD/CAM ceramic blocks. Filled adhesive increased the bond strength to HF etched and silanized ceramic surfaces. Adhesive application to either non-etched or etched processed composite substrate improved the bond strength.

The intent of this investigation was to examine the effect on bond strength of various substrates pretreatments and the reliability to use μTBS for testing resin cements bonded to ceramics or processed composite. This seemed best done by not introducing another interface such as additional substrate interface that might complicate the interpretation of the failure mode. However, for future studies, this test can be used to compare the bond strength and modes of failures by using a thin cement film between two substrates, which may reflect more the clinical situation.

3.6 References

Chapter 3


Chapter 3


