Dentin bonding: Influence of bonded surface area and crosshead speed on bond strength

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This study investigated the influence of the bonded surface area and the crosshead speed on the dentin bond strengths of self-etch adhesives. Bovine mandibular incisors were mounted in self-curing resin and the facial surfaces were wet ground with #600 silicon carbide (SiC) paper. The dentin surfaces were treated according to the manufacturer’s instructions. Adhesives were applied, and the resin composites were condensed into molds (2.4 or 4.0 mm in internal diameter), placed on to the dentin, and then light activated. Ten samples per test group were shear tested at crosshead speeds of 0.1, 0.5, 1.0, 5.0, and 10.0 mm/min. The results showed that higher crosshead speeds were associated with higher dentin bond strengths. This relationship was more significant for specimens with a smaller dentin bonding surface area.

Keywords: Bond strength, Crosshead speed, Bonded surface area

INTRODUCTION

The rapid introduction of adhesive systems has forced dentists to rely on laboratory tests to evaluate the bonding performance of these products1). Much of the research into dentin bonding has assessed the integrity and strength of the interfacial bond. Experimental approaches for the measurement of adhesive bond strengths in dentistry have consisted primarily of tensile or shear bond-strength determinations performed within a defined area2). Although the testing procedures used have appeared to be similar, the results have differed widely among studies3,4). Large variations in bond strength data and the lack of standardized laboratory test procedures have contributed to ambiguities in interpretation5.

Tensile and shear bond-strength measurements are known to be highly dependent on the geometry of the test apparatus, the nature of the load application, the presence or absence of adhesive flash, and the materials involved4,6). Non-uniform stresses act upon the bonded interface, which calls into question the concept of “average stress” in measurements of bond strength7-9). As all of the forces acting on an adhesive bond can be resolved into components acting at right angles to and parallel to the interface (shear), it is necessary to measure the shear strength in order to evaluate a bond adequately. Bond strength-testing rigs were designed such that the maximum stress in the shear apparatus was transmitted along the interface, whereas for the tensile bond strength, the stresses were transmitted through the adhesive to the interface. The path of the fracture when placed under tension therefore passed through the weakest areas in the bulk of the adhesive or the interface7). However, the forces that are exerted clinically on restorations or teeth are complex in nature, and neither tensile nor shear bond-strength tests simulate the intraoral forces. When a resin composite bonded to a flat dentin surface is tension- or shear-loaded, the distribution of the stresses along the interface is extremely irregular7-9).

It is generally accepted that the fracture strength of a brittle solid usually shows considerable statistical scatter, and depends upon the probability that a flaw capable of initiating a fracture at a specific applied stress is present. The rate of load applications is another influential factor that affects the results of shear bond-strength studies10,11). The adhesive interface comprises dentin, adhesives, and resin composites, all of which are brittle materials. When measuring the mechanical properties of brittle materials, a much lower load rate is applied than with elastic materials. Although the normal rate of load application for determining the dentin bond strength is 1.0 mm/min, the strain rate (or crosshead speed) employed varies across a wide range2,5). In addition, the diameter of the adherent surface varied in a relatively wide range, from 3 mm to 6 mm, with the exception of in the micro-tensile test6). Although an adherent diameter of 4 mm was commonly employed to evaluate dentin bond strengths for both tensile and shear modes, no rationale for selecting this value was given in the articles reviewed.

Knowledge about the factors influencing dentin bond strength could help with understanding and improving adhesive systems. The question has been arisen whether the different type of adhesive materials might reveal different behavior by changes in crosshead speed and/or adherent surface area. The purpose of this study was to study the influence of the crosshead speed and the dentin surface area on the bond strength, using shear bond-strength testing apparatus. The null hypothesis tested was that the crosshead speed and the

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bonded surface area did not affect the bond strength.

**MATERIALS AND METHODS**

The dentin bonding systems employed in this study were Clearfil SE Bond (Kuraray Medical Inc., Tokyo, Japan) and Clearfil tri-S Bond (Kuraray Medical) (Table 1). A visible light-activating unit (Optilux 501, SDS Kerr, Danbury, CT, USA) was used, and the power density (800 mW/cm²) of the light was checked with a dental radiometer (Model 100, SDS Kerr) before making the specimens.

In total, 200 mandibular incisors extracted from cattle and stored frozen for up to 2 weeks were used as substitutes for human teeth. After removing the roots with a slow-speed saw using a diamond-impregnated disk (Isomet, Buehler Ltd., Lake Bluff, IL, USA), the pulps were removed, and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces were ground on wet 240-grit silicon carbide (SiC) paper to create a flat dentin surface. Each tooth was then mounted in self-curing acrylic resin (Tray Resin II, Shofu Inc., Kyoto, Japan) to expose the flattened area, and placed under tap water to reduce the temperature rise caused by the exothermic polymerization reaction of the acrylic resin. A final finish was accomplished by grinding on wet 600-grit SiC paper followed by ultrasonic cleaning with distilled water to remove the excess debris. The dentin surface was dried with oil-free compressed air to remove visible water.

In all of the preparations, after the adhesive application, the specimens were clamped in the Ultradent Bonding Jig (Ultradent Products Inc., South Jordan, UT, USA), and plastic molds (2.4 or 4 mm in internal diameter, 2 mm in height) were used to form and hold the resin composite on the dentin surface. The adhesive was applied onto the dentin surface according to the manufacturer’s instructions, and the Clearfil AP-X resin composite was condensed into the mold and cured for 40 s. The finished specimens were transferred to distilled water, and stored at 37°C for 24 h.

Ten specimens per group were tested in the shear mode using a knife-edge testing apparatus (Ultradent Products Inc.) in a universal testing machine (Type 5500R, Instron Corp., Canton, MA, USA) at crosshead speeds of 0.1, 0.5, 1.0, 5.0, and 10.0 mm/min. The shear bond strength values (in MPa) were calculated from the peak load at failure divided by the specimen surface area. After testing, the specimens were examined under an optical microscope (SZH-131, Olympus Ltd., Tokyo, Japan) at a magnification of ×10 to define the location of the bond failure. The type of failure was determined based on the percentage of substrate-free material as follows: adhesive failure; cohesive failure in the dentin; and cohesive failure in the composite.

Statistical analysis was used to investigate how the bond strengths were influenced by the crosshead speed and the adherend surface. The data for each group were subjected to two-way analysis of variance (ANOVA) followed by Tukey’s Honestly Significant Difference (HSD) test at a significance level of 0.05. The statistical analysis was carried out with the Sigma Stat software system (Ver. 3.1, SPSS Inc., Chicago, IL, USA).

The fractured surfaces of the specimens after the test were observed by field-emission scanning electron microscopy (SEM). The SEM specimens were dehydrated in ascending concentrations of tert-butanol, and then transferred to a critical-point dryer. The surfaces were coated in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi Inc., Tokyo, Japan), with a thin film of gold (Au). The specimens were observed by SEM (ERA-8800FE, Elionix Ltd., Tokyo, Japan) with an accelerating voltage of 10 kV.

**RESULTS**

The mean shear bond strength to bovine dentin and the fracture mode after the bond strength test are shown in Tables 2 and 3. The two-way ANOVA revealed no statistically significant interaction between the bonded surface area and the crosshead speed (p=0.263 for SE Bond and p=0.122 for tri-S Bond). When comparing the data at the same condition, significant higher bond strength was found for Clearfil SE Bond comparing to those for Clearfil tri-S Bond. For the adhesives used in this study, the dentin bond strength increased with increasing crosshead speed. When the bond strengths were compared at the same crosshead speed, they decreased with increasing bonded surface area.

The representative SEM observations of the fractured surfaces after bond strength test are shown in

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Materials tested in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Adhesive system (Lot No.)</strong></td>
<td><strong>Resin composite (Lot No.)</strong></td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>Primer (00780A)</td>
</tr>
<tr>
<td></td>
<td>MDP, HEMA, water, PI, ethanol</td>
</tr>
<tr>
<td>Clearfil tri-S Bond</td>
<td>tri-S Bond (00080A)</td>
</tr>
<tr>
<td></td>
<td>MDP, Bis-GMA, HEMA, ethanol, initiators, accelerators</td>
</tr>
</tbody>
</table>

MDP: 10-methacryloyloxydecyl di-hydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, CQ: dl-camphorquinone, Bis-GMA: 2,2bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]phenyl
Table 2  Influence of cross sectional area and crosshead speed on dentin bond strength (in MPa) and fracture mode of Clearfil SE Bond

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Diameter</th>
<th>Crosshead speed (mm/min)</th>
<th>Bond strength</th>
<th>Fracture mode</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0.1</td>
<td>0.5</td>
<td>1.0</td>
</tr>
<tr>
<td>Bond strength</td>
<td>2.4 mm</td>
<td>39.8 (2.3)a^b</td>
<td>41.9 (3.3)a</td>
<td>43.0 (4.0)a^b</td>
</tr>
<tr>
<td></td>
<td>4.0 mm</td>
<td>15.4 (2.3)c</td>
<td>20.2 (1.7)d</td>
<td>22.0 (2.9)d,e</td>
</tr>
<tr>
<td>Fracture mode</td>
<td>2.4 mm</td>
<td>4/ 1/ 5</td>
<td>5/ 1/ 4</td>
<td>1/ 2/ 7</td>
</tr>
<tr>
<td></td>
<td>4.0 mm</td>
<td>2/ 6/ 2</td>
<td>3/ 6/ 1</td>
<td>2/ 6/ 2</td>
</tr>
</tbody>
</table>

\( \text{n}=10, \text{values in parenthesis indicate standard deviations.} \)

Values with the same superscript letters indicate no significant difference (\( p>0.05 \)).

Fracture mode: cohesive failure in resin/ cohesive failure in dentin/ adhesive failure

Table 3  Influence of cross sectional area and crosshead speed on dentin bond strength (in MPa) and fracture mode of Clearfil tri-S Bond

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Diameter</th>
<th>Crosshead speed (mm/min)</th>
<th>Bond strength</th>
<th>Fracture mode</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0.1</td>
<td>0.5</td>
<td>1.0</td>
</tr>
<tr>
<td>Bond strength</td>
<td>2.4 mm</td>
<td>27.5 (1.6)a^e</td>
<td>29.1 (1.8)a</td>
<td>32.6 (3.5)b</td>
</tr>
<tr>
<td></td>
<td>4.0 mm</td>
<td>13.5 (1.5)d</td>
<td>15.2 (2.4)d</td>
<td>17.1 (2.3)c</td>
</tr>
<tr>
<td>Fracture mode</td>
<td>2.4 mm</td>
<td>0/ 0/ 10</td>
<td>1/ 0/ 9</td>
<td>1/ 0/ 9</td>
</tr>
<tr>
<td></td>
<td>4.0 mm</td>
<td>0/ 0/ 10</td>
<td>2/ 1/ 7</td>
<td>1/ 3/ 6</td>
</tr>
</tbody>
</table>

\( \text{n}=10, \text{values in parenthesis indicate standard deviations.} \)

Values with the same superscript letters indicate no significant difference (\( p>0.05 \)).

Fracture mode: cohesive failure in resin/ cohesive failure in dentin/ adhesive failure

![Fig. 1](image_url)  SEM pictures of fractured surface of 2.4- and 4.0-mm-diameter specimens of Clearfil SE Bond. Remnants of fractured dentin were observed for 4.0-mm diameter specimens. Cohesive failures in resin composite were observed for 2.4-mm diameter specimens.
Figs. 1, 2. There was a trend towards differences in failure mode among specimens with different surface areas. After the dentin bond-strength tests, the predominant failure mode of Clearfil SE Bond was cohesive failure in the dentin and resin for the 4-mm-diameter groups, and adhesive failure tended to increase for 2.4-mm-diameter groups regardless different crosshead speeds (Fig. 1). For Clearfil tri-S Bond, predominant failure mode was adhesive for 2.4-mm-diameter group, and cohesive failure in resin and dentin was observed for 4.0-mm-diameter group (Fig. 2).

DISCUSSION

In order to measure the bonding effectiveness of adhesive systems to enamel and dentin, the shear and/or tensile bond strength is usually measured. The ideal bond strength test should be low technique sensitivity and relatively first to get the results. For the shear bond strength test, the diameter of the resin composite has an effect on the contact area between the edge of the loaded blade and the composite column, resulting in different stress distributions. Three-dimensional finite-element analysis indicated that using a wire-loop method of loading led to a smaller stress-concentration effect, but dividing the failure load by the cross-sectional area grossly underestimated the true interfacial bond strength. Using tensile test apparatus might negate this problem; however, in a tensile test the force is transmitted through the body of the resin, and partial cohesive failure, rather than interfacial failure, often occurs. And in the tensile test, the area subjected stress levels close to the maximum is much larger than in the shear test. Therefore our current study used different bonded surface areas with different diameters to test the shear bond strength.

The fracture strength of brittle materials is influenced by a number of factors, including specimen size, thickness, initial crack length, flaw location, and stress-strain state. Though there was no significant interaction between the factors, bonded surface area and crosshead speed, the data from the current study indicated that the shear bond strength of the adhesives tested depended on the bonded surface area: the smaller the bonded surface area, the greater the bond strength. In addition, larger specimens showed more cohesive failures in dentin compared with smaller specimens, especially for Clearfil SE Bond. A previous study found that the fracture strength of brittle materials is influenced by factors such as specimen size, thickness, initial crack length, flaw location, and stress-strain state. Though there was no significant interaction between the factors, bonded surface area and crosshead speed, the data from the current study indicated that the shear bond strength of the adhesives tested depended on the bonded surface area: the smaller the bonded surface area, the greater the bond strength.
study evaluating the bond strength of resin cement reported that the bonded surface area was associated with the incidence of cohesive failures, and specimens with larger cross-sectional areas showed only cohesive failure in dentin\(^{16}\). A smaller bonded surface area appeared to improve the stress distribution of the specimen, and to reduce the number of internal defects, such that smaller specimens exhibited adhesive failure after the test. The effect of the elastic modulus of the resin composite on the shear bond strength seemed to be consistent regardless of the specimen design. Smaller specimens contain fewer internal defects and have more homogeneous stress distributions, hence the strength of materials is dependent on the size of the sample\(^{17}\). Bonded surface area has been also reported to be associated with the incidence of failure mode, with larger bonded surface area showed exclusively cohesive failure in dentin\(^{18}\). This tendency was observed especially for Clearfil SE Bond, and this might relate to the differences in the thickness of adhesive layer and mechanical properties.

In this study, two different type of adhesive systems were used, two-step and single-step self-etch systems. Clearfil tri-S Bond yielded an adhesive layer of around 10 µm, and Clearfil SE Bond yielded even thicker layer of around 50 µm\(^{19}\). The dentin bonding systems tested here comprised three materials (composite, adhesive, and dentin), and the mechanical properties of adhesive layer differed. The elastic modulus of the successive layers across the resin-dentin bonding area showed a gradient. Such an elastic layer might have a strain capacity sufficient to conserve the dentin bond\(^{20,21}\). In the case of shear strength, the stress is concentrated at the interface, and the fracture path will not readily deviate unless there is a major flaw in the adhesive or at the dentin surface. The shear bond strength might, at least in part, relate to the modulus of the interface of the bonded area. Comparing to Clearfil tri-S Bond, Clearfil SE Bond consists of a separate hydrophobic solvent-free adhesive, which is known to polymerize better\(^{22}\), leading to higher mechanical properties. Thicker adhesive layer with higher mechanical properties might lead to higher bond strength and cohesive failures after the test.

Increasing the modulus of elasticity in bonded area will result in a more even distribution of stress over the bonded area, and less concentration at the point of application of the load. When failure occurs in an adhesive bond, the material at the tip of the crack will deform plastically to some extent, and a plastic zone of radius will form there. The relationship between the size of the plastic zone at the crack tip and the size of the specimen influences the fracture process. Comparing to the thinner adhesive, Clearfil SE Bond might have a wider plastic zone inside the adhesive. Additionally, the thickness of a dental plate relative to the size of the plastic zone at the crack tip has been reported to influence the plain strain, plain stress, and other stress-strain fields\(^{23}\). Thus, the geometry of the specimen, including the bonded surface area, affects the bond strength data\(^{24}\). The differences in plastic zone size, due to the thickness of adhesives, might affect the differences in fracture modes after the bond strength test. It has been reported that a tendency for dentin failure after the bond strength test to increase with thicker adhesive layer by using a failure accumulation computer model\(^{25}\). The differences in failure mode for the two different adhesive systems correspond to the results of this computer model study.

In the current study, higher bond strengths were observed with faster crosshead speeds for both adhesives. The viscoelastic nature of dental adhesives suggests that the shear bond strength and the failure mode might be affected by the rate of stress application. For instance, slower crosshead speeds might allow an extended recovery period during which stress and strain are compensated for by the elasticity of the adhesives; at lower speeds, the resin might behave like a viscous material, showing more deformation as increased pressure is applied, with a resultant increase in bond strength. Conversely, the potential for higher bond strength also exists with faster crosshead speeds, as the resin might perform as a brittle solid, with increased energy directed towards fracture of the specimen rather than molecular deformation and flexure. In either case, significant differences in bond strength between tested materials could result simply from varying the crosshead speed. The strengths of brittle materials generally increase with increasing strain rate\(^{26}\). One significant feature of resin composites is the strain-rate sensitivity of their strength, as these materials are characterized as viscoelastic polymers. Experimental observations have revealed that the yield strength of these polymers is bilinearly dependent on the logarithm of the strain rate, due to the changes of low-order transitions in the materials\(^{27}\). In the lower strain-rate range, the material strength increases slowly with increasing strain rate. When the strain rate exceeds a threshold level, a rapid change of material strength is recorded\(^{28}\). Therefore, the comparison among studies different crosshead speed should consider the strain rate sensitivity of the materials when interpreting the results of bond strength tests.

A report on the strain-rate effect on material behavior showed that the stress-strain curves stiffened as the strain rate increased\(^{29}\). Local events that result in macroscopic fracture can be described as locally stress- or strain-controlled. Brittle fracture in composite materials is invariably modeled as a stress-controlled process, involving the unstable propagation of a crack, which is initiated when the local tensile stresses exceed a critical threshold. Stress is concentrated at the loading position of the specimen in a shear bond-strength test, leading to high stresses at this point. The material in the vicinity of the crack wants to connect in the thickness direction. However, the material at the stress-concentration site is constrained by the adjacent material, which limits the amount of contact that can occur. Shear bond-strength specimens might thus be subjected to forces in the thickness direction, and might experience plain strain when they are loaded. Conflcting findings have been published on the
influence of crosshead speed on dentin bond strengths. In the current study, higher crosshead speeds led to higher dentin bond strengths. This relationship was clearer in specimens that had a smaller contact surface area of dentin. Further studies that focus on fracture mechanics are required to evaluate the bond strength between tooth substrates and dentin bonding systems.

REFERENCES