A comparison of fatigue crack growth in resin composite, dentin and the interface

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Abstract

Objectives. The objective of this in vitro study was to evaluate the fatigue crack growth properties of the dentin/resin adhesive interface.

Methods. Compact tension (CT) specimens were prepared from coronal dentin, resin composite, and dentin bonded to resin composite using Optibond Solo Plus adhesive. All specimens were then subjected to cyclic Mode I loading while fully hydrated at a stress ratio of R = 0.1 and frequency of 5 Hz. Steady state fatigue crack growth was modeled using the Paris Law in terms of the exponent (m) and coefficient (C).

Results. The average fatigue crack growth rates in the resin composite ranged from 1.6E−06 to 3.8E−05 mm/cycle with growth occurring over a stress intensity range from 0.40 to 0.77 MPa m1/2; the average growth exponent was 6.9 ± 3.1. Average fatigue crack growth rates for the dentin/resin interface specimens ranged from 5.5E−07 to 6.4E−03 mm/cycle with growth occurring over a stress intensity range from 0.37 to 0.64 MPa m1/2. The Paris Law exponent for these specimens ranged from 16 ≤ m ≤ 25. Fatigue crack growth at the interface occurred primarily in the adhesive resin and at the adhesive–dentin interface. In addition, many of the dentin/resin specimens underwent unstable fracture at a comparatively low stress intensity range without undergoing cyclic crack growth.

Significance. The dentin/resin adhesive interface proved to be significantly more sensitive to fatigue crack growth than either dentin or resin composite. Variation in the cyclic crack growth responses of the dentin/resin interface specimens suggests that the interface, and particularly the adhesive resin, exhibits lower resistance to crack initiation and growth in comparison to dentin.

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1. Introduction

Resin composites have become commonplace in restorative dentistry. Despite many advances since their inception, development of a durable and thoroughly sealed bond has remained a critical issue [1–3]. As a result, the bond strength between the dentin and resin composite has become a topic of considerable importance. The most common approaches employed for evaluating the interface strength are the micro-tensile and micro-shear tests [e.g. 4,5]. Due to the large number of studies that have adopted these methods of evaluation, as well as the more conventional tensile and shear tests, a full review of the methods and their findings is far beyond the scope of this study. Nevertheless, there have been some questions pertaining-
ing to the clinical relevance of these types of evaluations [6,7]. In particular, the interface is subjected to cyclic loading in the oral environment rather than quasi-static loading to failure. In addition, the aforementioned methods have limited sensitivity to microscopic defects that may be present at the bonded interface.

Based on the potential for defects at the bonded interface, some studies have adopted a fracture mechanics approach to quantify the apparent interface fracture toughness [8–11]. The importance of cyclic loading of the interface on the bond strength durability and interface leakage has also been recently considered [12–14]. In general, these studies have shown that cyclic loading decreases the bond strength and increases the potential for marginal leakage. Cyclic loading may promote fatigue and enable microscopic defects and other flaws at the interface [15] to coalesce in the form of a macroscopic defect. Larger inherent defects may also exist as a result of bonding procedures, or develop with aging of the restorative materials. Thus, interface failures may occur through a process that is assisted by fatigue crack growth. While the importance of fatigue crack growth in the failure of adhesive joints between dissimilar materials has received considerable attention in other fields [e.g. 16–18], its potential contribution in failure of the bonded interface in restorative dentistry has not been reported. As such, the primary objectives of this study were to develop an approach for evaluating fatigue crack growth along the dentin/resin interface and to compare properties of the interface to those of dentin and resin composite.

2. Materials and methods

Compact tension (CT) fatigue specimens were prepared from a selected resin composite and from a combination of resin composite bonded to dentin. The resin composite was Vit-l-escence (Ultradent, Provo, Utah; Lot #L0308), a Bis-GMA based small particle resin (microhybrid) with an average particle size of 0.7 μm. An aluminum mold was prepared to enable preparation of CT specimens of resin composite with approximate dimensions of 5.8 mm × 6 mm × 1.2 mm (Fig. 1). The resin composite was cured for 30 s using an Ultradent Ultra-Lume® LED 5 curing light, at a maximum distance of 4 mm. In general, the light was placed directly over the specimen. The specimens were released from the mold and sanded successively with 100, 220, and 400 grit sand paper. Additional features, including a machined notch for initiation of a fatigue crack and two holes for the applied loads, were introduced using a numerically controlled slicer/grinder and miniature milling machine, respectively. Finally, the specimens were stained on one side using a nontoxic (SharpieTM) permanent marker to allow a backlighting technique to be used for enhanced definition of the crack tip. All specimens (N = 14) were then stored in Hanks Balanced Salt Solution (HBSS) at room temperature (22 °C). Opening mode (Mode I) fatigue was applied under load control actuation, frequency of 5Hz and stress ratio of R = 0.1. In general, a maximum cyclic load of between 7 and 13 N was used, which was chosen to enable crack initiation without unstable growth. Measurement of the crack length was accomplished using a

showed no sign of disease or caries. All teeth were stored in Hanks Balanced Salt Solution (HBSS) immediately following extraction. Specimens were prepared from these teeth and tested within 2 weeks. The average patient age was 22 ± 5 years. Sections were obtained either along the buccal-lingual axis or the mesial-distal axis using the slicer/grinder under continuous water coolant. The primary slices were sectioned to obtain half of the completed CT specimen geometry (Fig. 1) with tubules oriented perpendicular to the bonded interface. The bonding surface of dentin was etched using 35% phosphoric acid for 15 s, rinsed with water, and then lightly dried. Optibond® Solo Plus (Kerr, Orange, CA; Lot #410833), a total-etch one-bottle system, was applied to the dentin bonding surface with an applicator brush to form a very thin layer. The solvent was evaporated in air for a short period (e.g. 10 s) and the adhesive resin was then light cured for 10 s. Following these steps, the dentin slices were placed in the molding fixture and an 80 μm width of polyethylene terephthalate (PETE, Mylar) was placed adjacent to the dentin bonding surface at one end for development of a molded notch. The Mylar film was torn to create a biased wedge and then placed within the mold such that the tip was located at the adhesive interface. Vit-l-escence brand resin composite (Ultradent, Provo, Utah; Lot #A0403) was then packed into the mold and cured for 30 s at maximum distance of 4 mm. The specimens were lightly sanded, two loading holes were drilled as previously described, and the molded notch was completed by removing the thin mylar section. All specimens (N = 14) were stored in HBSS for a minimum of 24 h before testing.

Cyclic loading was achieved using an EnduraTEC Model ELF3200 equipped with specialized fixtures. Loading of the specimens was conducted with specimens submerged in HBSS at room temperature (22 °C). Opening mode (Mode I) fatigue was applied under load control actuation, frequency of 5Hz and stress ratio of R = 0.1. In general, a maximum cyclic load of between 7 and 13 N was used, which was chosen to enable crack initiation without unstable growth. Measurement of the crack length was accomplished using a
The variable that loading points and free boundary in front of the crack. In addition, outliers in the growth responses were identified as a function of the load increment until cyclic extension permitted fracture. In general, the increment of cyclic loading ranged from $5 \leq \Delta N \leq 30$ kcycles and the increment of crack growth extended from $0.02 \leq \Delta a \leq 0.15$ mm. Cyclic loading was discontinued after the specimen fractured.

Fatigue crack growth in the CT specimens was modeled according to the Paris Law [20] where the incremental crack growth ($da$) and number of cycles ($dN$) is described in terms of the stress intensity range ($\Delta K$) according to

$$\frac{da}{dN} = C(\Delta K)^m$$

where $C$ and $m$ are the fatigue crack growth coefficient and exponent, respectively. Due to differences between the CT specimen geometry and that defined by ASTM E647 [21], a numerical model was developed to estimate the $\Delta K$ for the new CT specimen geometry. The Griffith approach to fracture [22] was adopted where the stress intensity ($K_i$) is defined in terms of the energy release rate. The stress intensity for both the resin composite and the bonded dentin/resin interface specimens was identified as a function of crack length by

$$K_i = \frac{P}{B\sqrt{W}}\left(0.1133 + 0.0841\alpha + 0.3859\alpha^2\right)$$

where $P$ is the maximum opening load, $B$ is the thickness of the specimen, and $W$ is the distance between the center of the loading points and free boundary in front of the crack. The variable $\alpha$ is the ratio of the average crack length ($\bar{a}$) to $W$ where $\bar{a}$ is the average of two consecutive crack length measurements. Using Eq. (2), the $\Delta K$ was determined from the load range ($\Delta P$).

Results of the experiments were used in constructing plots of the fatigue crack growth rate ($da/dN$) in terms of $\Delta K$ for each specimen. A power law model was fit to the steady state region (Paris Law: Region II) of fatigue crack growth and the Paris Law coefficient ($C$) and exponent ($m$) were determined for each specimen that underwent stable fatigue crack growth. A single factor analysis of variance (ANOVA) and Tukey-Kramer tests were performed ($p<0.001$) to identify significant differences between the resulting Paris exponents. In addition, outliers in the growth responses were identified from the average growth response (estimated using $m$, $C$ or average growth rate) using the Grubbs’ test with a 95% confidence interval. Previous results for fatigue crack growth in human dentin [19] were compared with the resin composite and bonded resin/dentin specimens. For the specimens that failed without undergoing fatigue crack propagation, the maximum applied load ($P_{\text{max}}$) and final crack length ($a_f$) were used (Eq. (2)) to estimate the critical stress intensity at fracture (i.e., the apparent fracture toughness, $K_c$). Fracture surfaces of all the CT specimens were examined with a JEOL JSM-5600 scanning electron microscope (SEM) in secondary electron imaging (SEI) mode to verify the tubule orientation (if applicable) and to evaluate characteristics associated with crack extension.

3. Results

Fatigue crack growth responses corresponding to steady state growth (Paris Law: Region II) are shown in Fig. 2. Cyclic crack growth in the resin composite was achieved in 8 of the 14 specimens and occurred over a stress intensity range ($\Delta K$) from 0.40 to 0.77 MPa·m$^{1/2}$ with growth rates ranging from $1.6E-06$ to $3.8E-05$ mm/cycle. A summary of the Paris Law parameters for each resin composite specimen is listed in Table 1 along with the average, which was estimated from an average of the individual $C$ and $m$ for all specimens. The responses for specimens A and B were identified as outliers according to their unique fatigue crack growth rates. Thus, $m$ and $C$ for these two specimens were excluded from the resin composite average response. In addition, the “pooled” behavior was examined where all growth data was combined to form a single response; the pooled response resulted in $m=5.34$ and $C=7.15E-05$ (mm/cycle) (MPa·m$^{1/2}$)$^{-m}$, respectively. Specimens that underwent unstable fracture were not included in the averaged or pooled results.

Steady state fatigue crack growth was achieved in only 5 of the 14 bonded dentin/resin interface specimens. Cyclic crack growth rates ranged from $5.5E-7$ to $6.4E-3$ mm/cycle and crack extension took place over a $\Delta K$ from 0.37 to 0.64 MPa·m$^{1/2}$. The Paris Law parameters for each specimen that underwent fatigue crack growth are listed in Table 2, along with results describing the average and pooled responses. According to ANOVA and Tukey-Kramer tests, the average Paris exponents for the resin composite and the interface were significantly different ($p<0.001$). Two unique responses were evident in the fatigue crack growth results of the interface specimens (Fig. 3). Three of the specimens (identified as...
Table 1 – Paris Law parameters for the resin composite specimens

<table>
<thead>
<tr>
<th>Resin specimen</th>
<th>m</th>
<th>C (mm/cycle) (MPa m^{1/2})^{-m}</th>
<th>r^2</th>
<th>Outlier</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>7.89</td>
<td>2.82E–03</td>
<td>0.96</td>
<td>Yes</td>
</tr>
<tr>
<td>B</td>
<td>3.50</td>
<td>1.00E–04</td>
<td>0.45</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>7.43</td>
<td>2.06E–03</td>
<td>0.63</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>4.91</td>
<td>1.59E–04</td>
<td>0.88</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>5.92</td>
<td>9.56E–05</td>
<td>0.87</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>3.72</td>
<td>4.46E–05</td>
<td>0.90</td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>10.4</td>
<td>7.80E–04</td>
<td>0.88</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>5.45</td>
<td>2.03E–04</td>
<td>0.97</td>
<td></td>
</tr>
<tr>
<td>Average values</td>
<td>6.92 (3.07)</td>
<td>2.98E–04 (2.83E–04)</td>
<td>0.55</td>
<td></td>
</tr>
<tr>
<td>Pooled response</td>
<td>5.34</td>
<td>7.15E–05</td>
<td>0.55</td>
<td></td>
</tr>
</tbody>
</table>

Stable fatigue crack growth was achieved in 8 of 14 specimens.

Table 2 – Paris Law parameters for the bonded dentin/resin interface specimens

<table>
<thead>
<tr>
<th>Interface specimen (group)</th>
<th>m</th>
<th>C (mm/cycle) (MPa m^{1/2})^{-m}</th>
<th>r^2</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (I)</td>
<td>25.2</td>
<td>7.11E–02</td>
<td>0.86</td>
</tr>
<tr>
<td>B (I)</td>
<td>24.5</td>
<td>5.28E–02</td>
<td>0.96</td>
</tr>
<tr>
<td>C (I)</td>
<td>23.1</td>
<td>3.15E–02</td>
<td>0.98</td>
</tr>
<tr>
<td>D (II)</td>
<td>16.0</td>
<td>1.53E–01</td>
<td>0.80</td>
</tr>
<tr>
<td>E (II)</td>
<td>13.6</td>
<td>1.31E–00</td>
<td>0.76</td>
</tr>
<tr>
<td>Average value</td>
<td>20.5 (5.3)</td>
<td>1.47E–02 (3.12E–02)</td>
<td>0.44</td>
</tr>
<tr>
<td>Pooled data</td>
<td>12.4</td>
<td>1.30E–01</td>
<td>0.44</td>
</tr>
</tbody>
</table>

Stable fatigue crack growth was achieved in only 5 of 14 specimens.

Group I) fell into a set with average Paris exponent of 24.3 ± 1.1. However, two interface specimens (Group II) exhibited Paris exponents that were considerably lower than those of Group I (Table 2). In Group II, stable growth occurred over a much lower stress intensity range as apparent in Fig. 3.

According to results for the 9 dentin/resin interface specimens that failed without undergoing fatigue crack growth, the apparent $K_c$ was estimated (Eq. (2)) to be 0.52 ± 0.16 MPa m^{1/2}. Similarly, using the final crack length measurements and maximum cyclic load just prior to unstable fracture, the apparent $K_c$ for the 6 resin composite specimens was 0.69 ± 0.13 MPa m^{1/2}. A comparison of results for the resin composite and bonded dentin/resin interface is shown in Fig. 4, along with that reported for dentin [23]. Unlike the fatigue crack growth responses, there were no distinct outliers in either the resin composite or interface responses.

In evaluation of the fracture surfaces there was no distinct features or defects that appeared to contribute to the cyclic crack growth response of the resin composite specimens. In contrast, the bonded dentin/resin interface specimens exhibited fracture surfaces with a variety of unique features. Most specimens exhibited fractured resin tags protruding from the adhesive resin (resin surface) or existing within the dentin tubules (dentin surface). In some regions, failure occurred completely within the resin adhesive without evidence of interfacial failure between the dentin and resin composite. There was no difference in the degree of

Fig. 3 – Fatigue crack growth responses for the bonded dentin/resin interface. Specimens of Group II were found to undergo growth under lower stress intensity range due to presence of flaws (pockets) in the adhesive resin.

Fig. 4 – Apparent fracture toughness of the bonded dentin/resin interface, and composite resin. Results are compared with the toughness of dentin reported in [23].
fractured resin tags, extent of fracture within the adhesive resin, or presence of specific defects between the specimens that underwent fatigue crack growth and unstable fracture. However, there were differences identified between the two groups (I and II) of dentin/resin interface specimens. Specimens of Group I exhibited a combination of cohesive failure in the adhesive resin and failure along the adhesive resin/dentin interface as evident from the fractured resin tags. An example of typical features for the specimens in Group I are shown in Fig. 5 (specimen B), which highlights a relatively large area of cohesive resin failure (~200 μm in diameter). While the same features were also evident on the fracture surfaces of Group II, these specimens also exhibited a noticeably larger number of voids in the adhesive resin. A representative area of the fracture surface is shown in Fig. 6(a).

4. Discussion

In a recent evaluation of the fatigue crack growth properties of human dentin, the mean Paris Law exponent was $m = 13.3 \pm 1.1$ [19]. Based on results of the present study, $m$ for the resin composite (Table 1) was significantly lower than the dentin/resin interface ($p<0.001$) and dentin ($p<0.001$). In addition, the exponent for the dentin/resin interface was significantly greater than that of dentin ($p<0.001$). As $m$ denotes the sensitivity of a material to the stress intensity range (i.e., flaw length), the dentin/resin interface is more sensitive to the presence of flaws than dentin. Indeed, the interface exhibited a very small range of stable cyclic extension (Figs. 2 and 3). In a comparison of the average fatigue crack growth rates for a stress intensity range of 0.65 MPa m$^{1/2}$, dentin exhibits the slowest growth rate (2.0E$^{-07}$ mm/cycle); the average growth rate in the resin composite is nearly 100 times greater (2.0E$^{-05}$ mm/cycle) than that in dentin. More importantly, at this $\Delta K$ the bonded interface would likely undergo fracture due to the low apparent fracture toughness (Fig. 4).

The stress intensity threshold ($\Delta K_{th}$) is generally used to define a critical stress intensity level where fatigue crack growth will progress at an insignificant rate. Though not measured according to ASTM E647, an estimate of the $\Delta K_{th}$ can be obtained from an extension of the Region II response to an appreciably low growth rate (e.g., 1E$^{-07}$ mm/cycle). According to Fig. 2, the $\Delta K_{th}$ of dentin (~0.65 MPa m$^{1/2}$) [19] appears to be much larger than that of the resin composite and of...
the bonded dentin/resin interface. For both materials, $\Delta K_{th}$ appears to be between 0.3 and 0.4 MPa m$^{1/2}$. From a clinical perspective, the combination of small $\Delta K_{th}$ and large average fatigue crack growth rate of the interface (Figs. 2 and 3) is very detrimental. A finite element study by the authors [24] on the stress distribution in restored molars resulting from clinical function indicated that cyclic opening mode stresses at the bonded interface can exceed 20 MPa. Assuming a geometry factor near unity, a stress range of 20 MPa and an apparent $\Delta K_{th}$ of 0.35 MPa m$^{1/2}$, interfacial flaws with effective length as small as 100 $\mu$m could undergo cyclic extension via fatigue crack growth. Flaws (voids) were apparent on the fractured interfaces within this range. Therefore, results of the present study are highly relevant to clinical failures.

Fatigue crack growth responses of the resin composite were pooled and compared to the average of individual responses (Table 1). The average and pooled responses for the resin composite were in far better agreement than results for the dentin/resin interface specimens, which exhibited a large difference between the average and pooled responses (Table 2). This difference suggests that the dentin/resin interface behavior was less consistent than that of the resin composite. Fracture surfaces of the dentin/resin interface showed that variations in the fatigue crack growth response could be attributed to the relative contribution of resin tag fracture, cohesive failure, and the number/size of defects. For Group I (Table 2) crack propagation occurred through a combination of cohesive failure of the adhesive resin and through fracture of resin tags at the adhesive resin/dentin interface (Fig. 5). The fractured resin tags are evidence of an effective bond, whereas the cohesive failure could be an indication of a non-uniform adhesive thickness, or simply, that the adhesive layer served as the weakest link regardless of a potentially non-uniform adhesive thickness. There was no correlation evident between the extent of cohesive failure and fatigue crack growth responses. Similarly, a previous study reporting shear bond strength obtained with Optibond Solo Plus found no correlation between shear bond strength, hybrid layer thickness and details of the fracture surface [25]. On the fracture surface of the interface specimens in Group II, voids were evident in the adhesive resin (Fig. 6). While these specimens exhibited a lower $m$ (suggesting they were less sensitive to the primary crack and stress intensity), they exhibited a larger fatigue crack growth rate for all $\Delta K$ (Fig. 3) and smaller resistance to fatigue crack growth.

Flaws at the interface may develop from polymerization shrinkage, improper bonding procedures, or stress concentrations posed by the cavity geometry [26]. As a result, they can take many forms. Voids in the adhesive resin of Group II interface specimens were the principal flaw type identified. While the specific mechanism responsible for their presence is not known, they most likely developed due to moisture on the etched dentin surface or residual resin solvent. Therefore, despite preparation of the interface in an ideal bonding environment with strict attention to detail, there were flaws present that contributed to the fatigue crack growth responses of the bonded interface. Micro-tensile and shear bond strength tests are not as sensitive to flaws at the interface simply by virtue of size effects and the relatively limited change in effective bonding area. Therefore, the methods developed and utilized in the present study may offer some advantages over the traditional methods used in evaluating bonded interfaces, particularly in identifying both the presence and significance of flaws.

There are several recognized limitations to the study. For example, all specimens were tested at room temperature (22 $^\circ$C), and within 24h of bonding. In addition, while the results were statistically significant, fatigue crack growth was achieved in a limited number of resin composite and bonded interface specimens. Furthermore, the dentin used for the interface specimens was obtained exclusively from third molars of young patients (mean age = 22 ± 5 years). Properties of dentin change with age [19,27] and bonding to old and sclerotic dentin is more difficult [28]. Similarly, the evaluation considered bonding to dentin substrates with a single tubule orientation. Fatigue of dentin is dependent on tubule orientation [29], and fatigue properties of the interface are expected to be a function of tubule orientation as well. Lastly, an isolated evaluation of the fatigue crack growth properties of the adhesive resin was not conducted. Results of the evaluation indicate that the adhesive resin and the dentin/adhesive resin interface are the primary weak links in fatigue.

Despite these recognized limitations, results from this study should contribute in development of a better understanding of mechanical behavior of the bonded dentin/resin interface.

5. Conclusions

Fatigue crack growth in a light cured resin composite, and the bonded dentin/resin interface were evaluated and compared with fatigue crack growth properties of coronal dentin. Based on the results from this experimental study, the following conclusions were drawn:

1. The Paris Law exponent ($m$) for fatigue crack growth in the bonded dentin/resin interface was significantly higher than either the resin composite ($p \leq 0.001$) or dentin ($p \leq 0.001$). The bonded interface is much more sensitive to the presence of flaws and susceptible to fatigue crack growth that initiates from existing defects.

2. The apparent fracture toughness of the bonded dentin/resin interface was found to be $0.52 \pm 0.17$ MPa m$^{1/2}$.

3. Fatigue crack growth and fracture of the bonded dentin/resin interface specimens occurred primarily within the adhesive resin and at the dentin/adhesive resin interface.

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