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Degradation in the Fatigue Strength of Dentin by Cutting, Etching and Adhesive Bonding

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Abstract

The processes involved in placing resin composite restorations may degrade the fatigue strength of dentin and increase the likelihood of fractures in restored teeth.

Objective—The objective of this study was to evaluate the relative changes in strength and fatigue behavior of dentin caused by bur preparation, etching and resin bonding procedures using a 3-step system.

Methods—Specimens of dentin were prepared from the crowns of unrestored 3rd molars and subjected to either quasi-static or cyclic flexural loading to failure. Four treated groups were prepared including dentin beams subjected to a bur treatment only with a conventional straight-sided bur, or etching treatment only. An additional treated group received both bur and etching treatments, and the last was treated by bur treatment and etching, followed by application of a commercial resin adhesive. The control group consisted of “as sectioned” dentin specimens.

Results—Under quasi-static loading to failure there was no significant difference between the strength of the control group and treated groups. Dentin beams receiving only etching or bur cutting treatments exhibited fatigue strengths that were significantly lower ($p = 0.0001$) than the control; there was no significant difference in the fatigue resistance of these two groups. Similarly, the dentin receiving bur and etching treatments exhibited significantly lower ($p = 0.0001$) fatigue strength than that of the control, regardless of whether an adhesive was applied.

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Significance—The individual steps involved in the placement of bonded resin composite restorations significantly decrease the fatigue strength of dentin, and application of a bonding agent does not increase the fatigue strength of dentin.

Keywords

adhesive resin; bonding; dentin; etching; fatigue; tooth fracture

Introduction

Resin composites are now the primary material for tooth cavity restorations [1]. But there is growing concern that bonded composite restorations have higher failure rates than their predecessors [e.g. 2,3]. The three most common forms of failure are reportedly secondary caries, marginal degradation and fracture (including either the restorative material, the supporting tooth tissues or both) [4,5]. Although not the most common form of failure, tooth fracture is potentially the most detrimental as it more commonly results in complete tooth loss. Teeth without restorations generally do not fail by fracture, which raises an important question. Does tooth fracture occur due to an increase in stress within restored teeth, or from defects introduced within the hard tissue foundation by the restorative process and subsequent fatigue?

In comparison to materials of the past, the placement of composite restoratives is complex [6]. As such, there are a number of steps that could inadvertently cause the introduction of defects within the tooth structure. For example, the excavation of demineralized tissue involves material removal, and an interaction between the cutting tools and hard tissue under dynamic conditions. Surface defects introduced during machining/grinding of brittle materials are extremely detrimental, and often lead to a reduction in strength [7-9]. The introduction of defects within hard tissues could diminish their structural integrity [10], thereby reducing durability of the restoration and increasing the likelihood of tooth fracture.

Past investigations have evaluated the material removal processes in cutting of hard tissues and the resulting surface integrity [11-14]. For instance, carbide and diamond abrasive bur preparations were found to introduce cracks during cutting of enamel, whereas the same processes were not found to cause damage while cutting dentin [14]. Similarly, though cracks were not found to result from bur treatments in dentin, Banerjee et al [15] reported that sono-abrasion and Carisolv gels introduced flaws. One could perceive that the flaws introduced by cutting are small, and that other aspects of the restorative process serve to enlarge the cracks resulting from cutting. Sehy and Drummond [16] introduced Class I or Class II MOD preparations in molars using either coarse diamond burs or an Er:YAG laser. The preparations were followed by placement of a resin composite, bulk curing to maximize interfacial stresses, and then evaluation of the tooth-composite interface via microscopy. Neither of the two cutting processes and subsequent steps resulted in visible microcracks in dentin.

Using measures of strength to assess the presence of damage, Staninec et al. [17] showed that cracks exceeding 100 μm in length were introduced within the dentin by laser preparations under some treatment conditions. That could suggest that flaws introduced with

dental burs are too small to see in direct evaluations (i.e. microscopy), but they certainly alter the natural flaw population and distribution within the tissue. As dentin is susceptible to degradation by fatigue [18,19] small flaws may propagate and facilitate fracture by fatigue crack growth [20,21]. Indeed, Majd et al [22] reported that while there was no influence of burs or airjet surface treatments on the strength of dentin under quasi-static loading, both preparations caused a degradation of strength when assessed by cyclic loading. That study did not consider other steps used in the placement of composite restorations (e.g. etching or adhesive bonding), or that flaws introduced by cutting operations may be removed by subsequent etching. Despite the importance of this topic to restored tooth integrity, this area of investigation has received limited attention.

The primary objective of this investigation was to evaluate the reduction in quasi-static strength and fatigue resistance of dentin resulting from the steps involved in preparing cavities and placement of resin-composite restorations. The null-hypothesis to be tested was that etching and application of a resin adhesive in the use of 3-step (etch-and-rinse) bonding systems, has no influence on the fatigue strength of dentin, regardless of whether or not the tissue has been prepared by bur cutting.

Materials and Methods

Caries-free third molars were obtained from participating dental practices in Maryland according to a protocol approved by the Institutional Review Board of the University of Maryland Baltimore County (Approval Y04DA23151). All teeth were from donors between 18 age 25 years old. The teeth were maintained in Hanks Balanced Salt Solution (HBSS) with 0.2% sodium azide as an antimicrobial agent at 4 °C, then cast in a polyester resin foundation and sectioned using a highspeed grinder (Chevalier Smart-H818II, Chevalier Machinery, Santa Fe Springs, CA, USA) and diamond abrasive slicing wheels (#320 mesh abrasives) with water-based coolant bath. Primary sections were made in the bucco-lingual plane, and secondary sectioning was performed to obtain beams as shown in Figure 1(a). The beams were prepared with width of 1.5 mm and thickness of either 0.5 or 0.65 mm, depending on whether a bur treatment was performed. Each of the beams was inspected; those with pulp horn intrusions, enamel end-caps or other non-uniformities were discarded.

Five different groups of beams were prepared including a nominally “flaw-free” control group that was evaluated directly as-sectioned, and a total of four treatment groups. Two of the treated groups received a single surface preparation, and two additional treated groups received a combination of preparations. One of the treated groups was subjected to a bur treatment (i.e. BT) in which cutting was performed using a 6-flute tungsten carbide straight fissure bur (Model FG 57, SS White, Lakewood NJ, USA) as shown in Figure 1(b). The cutting was performed using a commercial air turbine (Midwest Quiet Air-L High Speed Handpiece, Dentsply, York, PA, USA) and with water spray irrigation. Material was removed from one surface only (Figure 1(b)) in three equal passes for a total depth of material removal of 0.15 mm; the final beam thickness after material removal was 0.5 mm. The appropriate depth of cut was determined by preliminary tests in which a dentist made finishing passes on beams and controlled the depth of cut by tactile sense. Then the resulting depth of cut was measured, and used in controlled cutting operations performed by attaching

the handpiece to a miniature milling machine (Dyna Mechatronics, Model Dynamyte 2400 CNC milling machine, San Jose, CA) that provided controlled feed of the bur across the dentin specimens. That process ensured that a realistic depth of cut was used in the bur preparations and that cutting was performed uniformly, and without development of surface craters that would interfere with the flexure loading. A new bur was used after every 10 specimens to ensure that the bur was sharp. The sequential specimens obtained from each bur (i.e. 1st, 2nd, .. etc) were distributed randomly for testing within the low and high cycle regimes of the appropriate treatment groups. The total time involved in treatment (involving preparation and cutting) was less than 5 minutes. The second treated group received an acid-etching treatment (i.e. ET) after the diamond abrasive sectioning. Etching was performed with a 37.5 % phosphoric acid gel (Kerr Gel Etchant, Kerr Co., Lot #4574966). The gel was distributed on one surface of the beam for 15 seconds, and then removed by rinsing with distilled water for 15 seconds as recommended by the manufacturer. After etching and rinsing, the beams were lightly dried by gentle air blowing according to the product instructions.

The two additional treated groups of specimens involved a combination of bur and etching treatments. The first consisted of sectioning using the diamond slicing operation, bur treatment of one surface as described and followed by etching of that surface (i.e. BT+ET) according to the aforementioned conditions. The second group consisted of the combination of treatments (BT+ET), followed by application of a commercial primer and adhesive resin, and is referred to herein as BT+ET+Ad. The dentin primer (OptiBond FL Prime, Kerr Co., Lot # 4346590) was applied to the moist dentin surface by a light scrubbing motion for 15 seconds using a disposal applicator. After gently air-drying for few seconds, resin adhesive (OptiBond FL Adhesive, Kerr Co., Lot # 4346594) was applied to the primed surface. To ensure a thin and even layer of adhesive resin that would not interfere with the flexure testing, the excess resin was wiped off gently with clean tissue paper. Light curing of the resin was then performed using a quartz-tungsten-halogen light-curing unit (Demetron VCL 401, Demetron, CA, USA) with output intensity of 600 mW/cm² and with tip diameter wider than 10 mm. A twenty-second light exposure was overlapped at three different locations (center and both sides) to achieve a sufficient degree of polymerization over the entire dentin beam. The prepared beams were then placed into the HBSS bath at room temperature for 24 hours before testing.

The average surface roughness (R_a) and peak to valley height (R_y) of the prepared surfaces were assessed using contact profilometry (Model T8000, Hommelwerke, Jena, Germany). Profiles were obtained with direction parallel to the long axis of the beams using a 10 μ m diameter probe. This profile orientation characterizes the state of the surface topography resulting from each method and provides a means of assessing the potential influence of surface defects to the fatigue response. The surface roughness parameters were calculated according to the standard ANSI B 48.1 using a traverse length and cutoff length of 4.8 mm and 0.8 mm, respectively. The values of R_a and R_y were compared using a one-way ANOVA with Tukeys HSD and the critical value (alpha) was set at 0.05. Thereafter, the specimens were returned to a bath of HBSS and soaked for a period of at least two hours for rehydration prior to testing. It is important to note that the final thickness of the beams from all five groups was approximately 0.5 mm after completion of the preparations.

Nevertheless, the exact cross-section geometry of each beam was measured and recorded prior to testing for accurate estimation of the bending stress that resulted from the flexure loading.

Quasi-static and cyclic four-point flexure testing was conducted at room temperature (22 °C) within a bath of HBSS using a universal testing system (Model 3200, BOSE ElectroForce, Eden Prairie, MN, USA) and routine methods described elsewhere [25]. Quasi-static flexure was performed under displacement control loading at a rate of 0.06 mm/min (Figure 1(b)) according to previous studies [22-25]. Fifteen dentin specimens were evaluated from each of the five groups. For the four treated groups of dentin specimens, that surface representing the outermost dentin (closest to the dentin enamel junction) received the treatments. The beams were then oriented in the flexure arrangement such that the treated surface was always subjected to tension (in both quasi-static and cyclic loading experiments). The instantaneous load and load-line displacement were monitored at a frequency of 2 Hz to failure, with the average test requiring slightly less than 5 minutes. The strength (S) was determined using conventional beam theory [Popov, 1978] in terms of the maximum measured load (P) and beam geometry (b, h; Figure 1(b)) according to $S=3Pl/bh^2$, where l is the loading span (l=2 mm). The flexure strengths were compared using a one-way ANOVA with Tukeys HSD and the critical value (alpha) was set at 0.05.

Cyclic loading experiments were conducted with the same loading arrangement used for quasi-static evaluation (Fig. 1(b)) with a stress ratio (R=min load/max load) of 0.1 and frequency of 5 Hz. These conditions are consistent with previous studies [22, 25]. Each beam was subjected to cyclic loading until failure at a cyclic stress that resulted in failures in between 100 cycles and 1,200 kcycles. For specimens that withstood 1,200 kcycles without failure, the experiment was discontinued as that is near the apparent endurance limit identified in previous studies [21,25]. The fatigue life distribution of the specimens that underwent fatigue failure in each group was modeled using a Basquin-type model [26] according to

$$\sigma=A(N)^B \quad (1)$$

where A and B are the fatigue-life coefficient and exponent, respectively, and were obtained from a regression of the fatigue responses plotted on a log-normal scale. For convenience of comparison, the apparent endurance limit was estimated from the models for a fatigue limit defined at 1×10^7 cycles.

The fatigue evaluation required a large number of samples and some results were recruited from previous studies. The control specimens (N=75) consisted of a combination of previously reported data [25] and additional specimens that were prepared to validate the fatigue behavior. The bur treated (BT) specimens (N=41) consisted of previously reported data [22] and one additional specimen. For the ET (N=29) and BT+ET (N=25) groups, all of the specimens were prepared specifically for this investigation. The same is true for all specimens of the BT+ET+Ad group (N=35). The fatigue strength distributions for the five

groups were compared over the defined range of cycles to failure using a Mann Whitney U test with the critical value (α) was set at 0.05.

The treated surfaces resulting from the bur and etching preparations, as well as adhesive bonding, were analyzed using a Scanning Electron Microscope (SEM: JEOL Model JSM 5600, Peabody MA, USA) in secondary electron imaging mode. The fracture surfaces of representative specimens from all groups were examined using the SEM and optical microscopy to identify flaws, the origin of failure or other features that could be important to the mechanical behavior.

Results

Following application of the surface treatments, the prepared surfaces of the dentin specimens were evaluated using microscopy. Electron micrographs documenting the surface characteristics of representative specimens are shown in Figure 2. The surfaces resulting from the bur treatment exhibited a smear layer, as evident in Figure 2(a). Similar features were noted on the surfaces of the control specimens prepared using the diamond abrasive slicing equipment (not shown). The surface of a specimen receiving the etching treatment is shown in Figure 2b. In contrast to the BT specimens, there is no evidence of a smear layer and the most distinguishing feature was the distribution of open lumens. The specimens subjected to bur treatment and followed by etching (BT+ET; Figure 2(c)) exhibited surface characteristics that were similar to those that received an etching treatment only. Nevertheless, a small portion of the peritubular cuff or smear layer debris remained visible within the lumens of the BT+ET specimens. A representative surface of the BT+ET+Ad specimens is shown in Figure 2(d). As evident in this figure, the lumens are filled with resin adhesive and there is only a small degree of adhesive debris remaining on the surface. Hence, the gentle wiping of the resin prior to curing was successful in removing the excess resin. Based on the microscopic evaluation, the surfaces of the specimens receiving bur treatment only, and the BT+ET+Ad specimens appeared to have the largest roughness.

A comparison of the average surface roughness (R_a) and peak to valley height (R_y) of the dentin specimens resulting from the individual treatments is shown in Figures 3(a) and (b). The R_a of the bur treated specimens ($0.59 \pm 0.13 \mu\text{m}$) was larger, but not significantly greater ($p > 0.05$) than that of the control ($0.36 \pm 0.11 \mu\text{m}$) or the ET treated specimens ($0.33 \pm 0.15 \mu\text{m}$). Furthermore, the surface roughness of the BT specimens was not significantly different ($p > 0.05$) than that of the ET group. The R_a of the beams receiving bur and etching treatments was the largest of all groups ($0.73 \pm 0.35 \mu\text{m}$), which was significantly greater than the control ($p < 0.05$), but not significantly different ($p > 0.05$) from the R_a resulting from bur treatment alone. In addition, the application of primer and adhesive did not cause a significant change in the roughness (BT+ET+Ad; $R_a = 0.52 \pm 0.24 \mu\text{m}$) from that resulting from bur treatment alone, or after secondary etching (BT+ET). Results for the relative measures of R_y were consistent with those obtained for the R_a . The R_y resulting from the BT+ET treatment was significantly greater ($p < 0.05$) than that resulting from the BT and ET treatments alone. However, there were no other significant differences in the R_y between the groups.

A comparison of the strength of the dentin specimens assessed by quasi-static loading to failure is shown in Figure 4. The average flexure strength of the flaw free control specimens was 154 ± 24 MPa. Surprisingly, there was no significant difference ($p > 0.05$) in the strength between the flaw free control and any of the groups of specimens receiving bur, etching or combinations of those two treatments. Hence, the average strength of dentin was not influenced by any of the surface treatments performed.

A comparison of the fatigue life distribution of the control dentin specimens with results for the two groups receiving single surface treatment (BT and ET) is presented in Figure 5. Specifically, the fatigue life diagram for the flaw free control specimens is presented with the fatigue life distribution for the BT specimens in Figure 5a. Each data point in this diagram represents the fatigue response of one specimen; data points with arrows represent those that did not fail within a prescribed number of cycles and the experiment was discontinued. An equivalent comparison of results for the flaw free control and the ET specimens is shown in Figure 5b. Power law equations describing the mean fatigue responses are presented in each diagram for reference, along with a description of the goodness of fit in terms of the coefficient of determination (R^2). According to the Mann Whitney U test, the fatigue strength distribution of the specimens receiving BT ($Z = -5.6$; $p = 0.0001$) and ET ($Z = -21.3$; $p = 0.0001$) treatments were both significantly different from that of the flaw free control. There was no significant difference between the responses of the BT and ET groups ($p > 0.05$).

Fatigue life diagrams for the two groups of dentin specimens receiving a combination of treatments are presented in Figure 6. Results for the BT+ET group of treated specimens are shown in Figure 6a, along with results of the controls specimens. As apparent from this comparison, the fatigue strength of the BT+ET specimens was significantly lower ($Z = -5.5$; $p = 0.0001$) than that for the control group. Similarly, the fatigue life distribution for the BT+ET+Ad group is shown in Figure 6b. These results are compared with the responses of the BT+ET group previously shown in Figure 6a. According to the Mann Whitney U test, there was no significant difference in the fatigue life distributions of the BT+ET group and after application of the adhesive ($Z = -1.0$; $p = 0.312$).

Using the power law equations developed from the least-squares error analysis of the cumulative fatigue responses, the apparent endurance limit for each group of specimens was estimated at 1×10^7 cycles. For the control specimens that value is 44 MPa. Similarly, for the two groups of BT and ET specimens, the apparent endurance limit is 28 and 35 MPa, respectively. In evaluation of the BT+ET and BT+ET+Ad groups the apparent endurance limits were found to be 31 MPa and 36 MPa, respectively. An alternate way of comparing the responses is in terms of the fatigue life rather than the strength. All four of the treated groups exhibited a mean fatigue life less than one tenth that achieved by the control group.

The fracture surfaces of specimens from each group were evaluated using the SEM. A compression curl was identified on the compressive side of the neutral axis for all groups, thereby indicating that failure initiated from the tensile surface regardless of the method of treatment. Failure progressed along the plane of maximum principal stress to the compressive side of the specimens. Consequently, the majority of the fracture surface was

in-plane and parallel to the dentin tubules due to the orientation of sectioning and flexure loading (Figure 1). Micrographs from selected specimens are shown in Figure 7. All the micrographs in this figure were obtained just beneath the surface that was subjected to the maximum tensile stress. The fracture surface from a representative BT specimen is shown in Figure 7a. Evident in this figure is the smear layer at the surface as highlighted with arrows, as well as its extension beneath the surface within the tubule lumens. A region of damaged material appears just beneath the surface proper that extends to a depth of approximately 10 μm . These features are substantially different from those for specimens that received an etching treatment. For example, for the ET group (Figure 7(b)) there was no evidence of damage to the surface. The most notable feature was the expansion in lumen diameter, which appeared to begin at approximately 10 μm depth and then increased with proximity to the surface. The fracture surfaces of specimens received bur and etching treatments (BT +ET; Figure 7c) appeared nearly identical to those of the ET group. The depth of demineralization, as apparent from the broadening of the tubule lumen diameter, extended approximately 10 μm beneath the surface. A fracture surface from the BT+ET+Ad group is shown in Figure 7d. Consistent with the ET and BT+ET specimens there is no evidence of damage or smear layer at the surface. However, the lumens are filled with resin adhesive that penetrated to an average depth of approximately 40 μm beneath the surface. That distance is well beyond that window of visibility provided by the level of magnification used in obtaining the micrographs in Figure 7.

Discussion

The objective of this investigation was to identify the changes in strength and fatigue behavior of dentin caused by bur preparations, etching and placement of a resin adhesive using a 3-step (etch-and-rinse) system. When evaluated under quasi-static loading to failure (Figure 4), there was no significant difference in the apparent strength between the control group, the groups receiving a single surface treatment (BT or ET) or the two groups treated with a combination of processes (BT+ET or BT+ET+Ad). The results from quasi-static loading distinguish that neither the bur or etching treatments introduced defects of adequate size to reduce the strength of dentin.

Results from cyclic loading of the specimens showed that both the bur and etching surface treatments caused a significant reduction of the fatigue strength with respect to the control (Figure 5). Furthermore, when the bur treated specimens were subsequently subjected to etching (i.e. BT+ET), the surface integrity was not substantially improved; the fatigue strength of the BT+ET group was significantly lower than that of the flaw free control. Even after the addition of primer and adhesive to the BT+ET surfaces, the fatigue strength was significantly lower than that of the flaw free controls; it was not significantly different from that of dentin receiving the BT+ET treatment (Figure 6b). Therefore, the null-hypothesis must be rejected.

The reduction in fatigue strength of the BT specimens could result from changes to the surface roughness or surface integrity. There was an increase in the average surface roughness (Ra) after bur treatment from approximately 0.36 ± 0.11 to 0.59 ± 0.13 μm (Figure 3) but the change was not significant. Though the increase is nearly a factor of two, it is

undoubtedly much less than would occur when cutting is performed via hand control of the bur and handpiece. It is important to note that the surface profiles of the BT specimens primarily represent characteristics of the smear layer [27] and do not characterize the underlying subsurface damage. According to a previous evaluation of the surface topography of dentin, the profile valley radii resulting from cutting of dentin are much greater than the magnitude of surface roughness [28]. Thus, while bur treatment did result in an increase in the surface roughness, there is only minimal difference in the degree of stress concentration realized by the larger roughness of these specimens. Therefore, the most likely cause for reduction in fatigue life of the BT dentin is the introduction of subsurface flaws to the tissue (not identified by the surface profiles) and the flaw sensitivity of dentin [24].

If there are subsurface flaws beneath bur cut surfaces of dentin, a simple solution is to simply remove them. A previous evaluation of bur treated surfaces suggested that the apparent length of subsurface flaws (i.e. cracks) in dentin after bur treatment was approximately 70 μm , in comparison to less than 30 μm for the flaw free control [22]. There was evidence of a damaged region beneath the smear layer of the BT specimens from evaluation by electron microscopy (Figure 7a). However, no specific subsurface flaws were identified, thereby suggesting that the reduction in fatigue strength results from subsurface defects extending beyond the smear layer and underlying damaged region ($\approx 10 \mu\text{m}$). In the worst-case scenario the aforementioned region of damaged tissue is no longer load-bearing (i.e. the smear layer and damaged region has no stiffness), and the effective dimensions of the beams are reduced by the thickness of the damaged region (10 μm). Then according to simple beam theory [29] there would be an increase in surface stress of less than 5%. That is far less than the difference in fatigue strength between the flaw free control and bur treated group, which is a minimum of 20% difference. Therefore, the flaws resulting from bur treatment of dentin must extend beyond the visible damaged region and removing these flaws by etching is unlikely.

In an examination of the power law models describing the fatigue life distributions it is apparent that the R^2 values for all the treated groups were relatively low. That is a potential limitation of the experimental protocol; the evaluation was conducted using a large number of stress levels, rather than performing fatigue tests using few stress levels and a larger number of specimens at each. Nevertheless, the distribution of the fatigue data about the mean is an important characteristic of differentiation between the groups. The BT specimens exhibited the lowest fatigue strength overall and the largest degree of scatter in the fatigue responses (Fig. 5a). The coefficient of determination for the BT group ($R^2=0.37$) is less than one half the value of the control specimens and substantially smaller than any of the groups receiving etching treatment. There are specimens of the BT group that performed nearly equivalently to the flaw free control specimens (Figure 5a), but some failed below two standard deviations of the expected life as well. That could suggest that either the subsurface damage is randomly distributed or that material removal occurs in a brittle manner and causes the development of spurious flaws. There are also spatial variations in the microstructure and mineral/collagen ratio of dentin that could contribute to the variability in fatigue responses [24,30]. Nevertheless, the primary point is that the damage imposed during bur cutting of dentin is detrimental. Moreover, the degree of damage imposed to the dentin specimens in this in vitro evaluation is expected to be far less than that

introduced during the placement of cavities. Further study regarding damage of tooth structure during cutting may be warranted.

The etching treatment was conducted according to the manufacturers recommendation and comprised of a 15 sec exposure to 37.5 % phosphoric acid gel, followed by water rinsing. Etching was expected to remove flaws from the machined surfaces and facilitate an increase of the fatigue strength of the bur treated substrate. However, when applied to the flaw free control specimens, etching caused a significant reduction to the fatigue strength (Figure 5b). The surface roughness of the ET specimens was not significantly different from that of the control group (Figure 3). In addition, the microscopic evaluation showed that the ET surfaces appeared smooth and free of notable defects (Figure 2b). A close examination of the fracture surfaces for the ET and the BT+ET groups revealed that the demineralized zone at the top of the surface extended a few micrometers. Admittedly, the depth of apparent demineralization is potentially influenced by the SEM preparation and collapse of the collagen. Also evident is the expansion of the lumens beneath the surface receiving acid treatment and an increase in porosity through opening of the transverse lumens from the interior of the peritubular cuffs and within the intertubular dentin (Figures 7b and 7c). The affected depth of etching (inferred by expansion of the lumen diameter via demineralization) was limited to approximately 10 μm , which is consistent with the range reported for a 15 sec. etch [6, 31-33]. If that layer was no-longer load bearing, simply mechanics shows that the surface stress would increase by only 5%. Thus, consistent with the BT specimens, the reduction in fatigue strength of the ET group (Figure 5b) may result from defects that developed beneath the most immediate etched surface through diffusion of the acid. A reduction in mineral content at the etched surface would also reduce the stiffness of the affected layer and increase the degree of surface strain with flexure loading. Nevertheless, the application of primer and adhesive did not recover the fatigue strength of the dentin subjected to bur and etching treatments, despite the degree of penetration (Figure 6b).

This investigation is the first to identify that acid etching causes a reduction in the durability of dentin when subjected to cyclic loading. If the degradation caused by etching is quantified in terms of the decrease in apparent endurance limit, etching results in at least a 20% reduction in the fatigue strength of dentin. Moreover, if the tissue is initially prepared by a high-speed handpiece followed by acid etching, the reduction in fatigue strength would approach 30%. Biofilm attack of resin composites [34-36] and the bonded interface [37], as well as enzymatic degradation of dentin collagen [e.g. 38-41], are recognized obstacles to achieving bond durability with placement of resin composite restoratives. However, the present results add to this list of concerns: the integrity of the supporting hard tissue foundation is an additional consideration to adhesive bond durability. That is of substantial clinical significance. If acid etching will remain an integral component of dentin bonding, and subsurface demineralization causes the development of spurious defects, then remineralization strategies [e.g. 42-44] to repair these defects in dentin may be essential for extending the durability of the bonded interface.

In addition to the clinical relevance of the investigation, there is an important finding that relates to *in vitro* testing methodologies applied to dental materials. With regards to dentin bonding, there have been concerns regarding the use of microtensile bond strength testing

towards understanding clinical failures [45-47]. Perhaps the prevailing concern is that the results of *in vitro* experiments do not reflect the reality of failures *in vivo*, and there is little correlation to clinical behavior [48]. One direct limitation of microtensile tests is that they utilized quasi-static loading to failure. When the dentin treatments were evaluated under monotonic loading to failure, there was no significant difference between any of treated groups with respect to the flaw free control. However, under cyclic loading the degradation of dentin caused bur treatment and etching treatments was clearly revealed. Considering that mastication is a process of cyclic loading, it would appear that fatigue studies are a critical requirement to identifying the effects of dentin treatments on the durability of the bonded interface.

Based on results of the fatigue studies, the largest degree of damage and size of defects resulted from bur cutting of the dentin specimens. Defects are most commonly introduced when cutting and/or grinding of brittle materials [9]. In coronal dentin the brittleness increases with proximity of the pulp due to the change in microstructure and increasing mineral to collagen ratio [24]. Related to these changes in microstructure within the crown, there is also a reduction in the resistance to fatigue crack growth of dentin with depth [30]. That raises the potential for degradation in the fatigue strength of coronal dentin with depth of the cavity preparation. Furthermore, dentin undergoes a reduction in both the fatigue strength and fatigue crack growth resistance with patient age [49-52], as well as a reduction in fracture toughness [53,54]. An increase in the brittleness due to these changes in microstructure increases the likelihood of introducing flaws to the tooth during cavity preparation, as well as the potential for fatigue to facilitate tooth fracture. Therefore, bur treatments would be expected to be more detrimental to the fatigue properties of old dentin than that identified here.

As with all investigations there are some important limitations to the experimental approach and methods of evaluation that warrant discussion. One concern is the large number of stress levels used in evaluation of the fatigue behavior and the correspondingly small coefficient of determination (R^2). That concern was addressed earlier, with comment that higher coefficients could be obtained by using fewer stress levels and larger number of samples within those levels. But that concern is most relevant to the modeling, and less with respect to the variability in fatigue responses. As evident in Figures 5 and 6, all of the treated groups exhibited larger variation in the fatigue behavior than the “flaw-free” control and this variation is expected to be associated with the type and severity of flaws. In addition, the cyclic loading was conducted using a frequency of 5 Hz, which is approximately three times higher than that of mastication [55]. The fatigue properties of dentin are dependent on loading frequency [21,56], and there is an increase in the apparent fatigue strength of human dentin with increasing testing frequency. A frequency of 5 Hz was used to balance the concerns of clinical relevance and the period of testing required. For reference, the present study required more than one year of experimental evaluation. Reducing the testing frequency to 2 Hz would increase the required time for achieving the results by a factor of 2.5. The importance of this issue is that the influence of surface flaws resulting from the bur or etching treatments on the fatigue strength of dentin could be different if tested at 2 Hz or lower frequency. Hydrated dentin exhibits increasingly brittle behavior with reduction in

loading rate as shown in a previous evaluation on the dynamic fatigue behavior of dentin [57]. That would suggest that testing at 5 Hz frequency could have diminished the degree of reduction in fatigue strength that occurs by flaws introduced by the bur treatments. While the exact degree of reduction may be frequency dependent, results from the experiments clearly show that both bur and etching treatments reduce the fatigue strength of dentin. Equally important, the application of an etch-and-rinse adhesive did not improve the fatigue strength from that caused by the bur or etching treatments.

Another perceived limitation is the narrow range of treatment conditions considered and the definition of control samples as “flaw free”. For example, the fatigue behavior of dentin is a function of tubule orientation and location. Dentin exhibits a larger degree of flaw sensitivity when cyclic stresses are acting parallel to the tubules [23], and within deep dentin [30]. Consequently, the degradation in fatigue strength caused by bur and etching treatments may be more detrimental if applied to deep dentin or in the alternate tubule orientation. These concerns should be addressed considering the importance to clinical practice. Furthermore, in regards to the surface treatments with the high speed bur, a single bur type was used with a very moderate feed rate and narrow range of cutting conditions. Thus, the estimated reduction in fatigue strength resulting from the bur treatments could be very conservative. Lastly, it is necessary to comment on the preparation of the control specimens and reference to these as “flaw free”. These specimens were prepared using a commercial grinder with diamond abrasive slicing process. This process is typically adopted for preparing test coupons of materials (e.g. ceramics, composites, etc), which are then used to characterize the intrinsic mechanical properties. Processing conditions were carefully chosen to minimize the introduction of cutting damage, but it is possible the diamond abrasive slicing process could have introduced small flaws within the surface that reduced the fatigue strength from 100% flaw free. Dentin is also traversed by the tubules, which could be considered a distributed network of flaws. Both of these factors complicate the idea of a “flaw-free” dentin control and the preparation of one that is not subject to scrutiny. Further work is underway to identify the extent of degradation that results from cutting cavity preparations and the methods that can be used to follow the preparation and maximize the durability of dentin.

Conclusions

An experimental evaluation was conducted to systematically evaluate the degradation in fatigue strength of dentin caused by the procedures involved in preparing cavities and placement of resin-composite restorations according to a 3-step bonding procedure. Under quasi-static loading to failure, there was no significant reduction ($p>0.05$) to the strength of dentin resulting from bur treatment, etching or application of primer and resin adhesive. When subjected to cyclic loading, both bur cutting and etching significantly reduced ($p = 0.0001$) the fatigue strength of dentin. Furthermore, the dentin beams receiving treatments involving bur cutting followed by etching, and the latter steps followed by placement of a primer and resin adhesive exhibited a significantly lower ($p = 0.0001$) fatigue strength than the flaw free controls. Overall, the findings show that both cutting of dentin with burs and etching degrade the fatigue properties of dentin. Tooth structure prepared

using these techniques potentially lacks the durability that is desired for maximizing lifelong oral health.

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Figure 1.

Specimen preparation and flexure loading of the dentin beams.

a) location of the crown in which the specimens were obtained and the treatments. The dentin tubules are oriented perpendicular to the beam length (along the y axis). After sectioning, the treated controls were subjected to either bur treatment (BT) or etching (ET). Thereafter, the treated groups were comprised of bur treatment and etching (BT+ET), and bur treatment, etching and application of the primer and adhesive (BT+ET+Ad). All treatments were conducted on the surface facing the DEJ.

b) nominal specimen geometry and flexure loading configuration for both monotonic and fatigue loading. All dimensions in millimeters. The beams were loaded with the treated surfaces subjected to tension.

Figure 2.

Micrographs of the treated surfaces of selected dentin beams.

a) bur treatment, b) etching treatment, c) after bur treatment and etching, d) after bur treatment, etching and application of the adhesive. Note the parallel lines on the surface of the bur treated specimen that result from the direction of the bur rotation. This specimen shows some degree of a smear layer, which obscures details of the microstructure beneath. There is no evidence of smear layer on the surface of the beams treated with etchant in (b) and (c).

a)

b)

Figure 3.

A comparison of the surface texture of the dentin specimens. a) average surface roughness, b) peak to valley height roughness. Columns with different letters are significantly different ($p < 0.05$).

Figure 4.

Results from quasi-static loading of the coronal dentin beams to fracture. The responses for each group are presented in terms of the mean and standard deviation in strength. There was no significant difference in any of the groups evaluated ($p>0.05$).

a)

b)

Figure 5.

A comparison of fatigue life diagrams for the control and treated control groups of dentin specimens. Each data point corresponds to the failure of a single dentin beam and data points with arrows identify beams that did not fail and the test was discontinued. The R^2 accompanying each empirical equation represents the coefficient of determination.

a) flaw free control and Bur Treatment (BT), b) flaw free control and Etching Treatment (ET).

a)

b)

Figure 6.

Fatigue life diagrams for the treated groups of dentin beams.

a) beams receiving bur and etching treatments (ET+BT) compared with the control, b) beams receiving bur and etching treatments an adhesive (BT+ET+Ad) compared with the control.

Figure 7.

Micrographs obtained from the fracture surfaces of selected dentin beams. The treated surface is at the top of these micrographs.

a) bur treatment. Note the smear layer that is evident at the top of the fracture surface (arrows). b) etching treatment, c) after bur treatment and etching. d) after bur treatment, etching and application of the adhesive. Note the thin layer of adhesive that is present at the top of the fracture surface.