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Fatigue of dentin–composite interfaces with four-point bend

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ABSTRACT

Objectives. The objective was to determine the fracture and cyclic fatigue properties of composite–dentin beams bonded with a self-etching adhesive in four-point bend.

Methods. Beams of rectangular cross-section were shaped to a size of $\sim 0.87\,\mathrm{mm} \times 0.87\,\mathrm{mm} \times 10\,\mathrm{mm}$ and placed in a four-point bending apparatus, with the loading points 1.8 and 7.2 mm apart, with the interface centered between the inner rollers. Cyclical loading was performed in Hanks' Balanced Salt Solution at 25 °C, with forces between 54% and 99% of the bending strength of the bonded beams.

Results. Solid dentin and solid composite beams [$n = 6$] had bending strengths of 164.4 and 164.6 MPa, respectively, under monotonically increasing loads. Bonded beams [$n = 6$] had strengths of 90.6 MPa. No significant difference was found between solid composite and solid dentin beams, the bonded beams were different (ANOVA, $p < 0.0001$) With long-term cycling, stresses below 49 MPa were tolerated for 10^6 cycles, but with increasing stress up to 90 MPa, beams failed earlier, demonstrating that subcritical fatigue cycling will eventually cause failure.

Significance. Fatigue may be a significant mechanism of dentin–composite bond degradation.

1. Introduction

Dentin–composite bonds are adequate when measured immediately, but deteriorate with time, causing restoration loss and leakage. These limitations have been discussed in recent reviews both from the clinical and laboratory perspectives [1,2]. Two main mechanisms of deterioration have been proposed: mechanical fatigue and hydrolytic degradation. Fatigue can result from stresses placed on the bond by shrinkage of composite due to polymerization, thermal expansion and contraction, or occlusal forces. To improve stability, we need to understand the mechanisms of bond degradation.

Enamel–composite bonds without mechanical undercuts enjoy long-term clinical success, as evidenced by retention of direct composite bonding, porcelain veneers [3,4] and Maryland bridges [5]. However, this is not true of dentin–composite bonds, which are tested by bonding composite into non-carious cervical notches without undercuts. These restorations frequently fail, with failures accelerating after 2 years [2]. The failure rates vary with the bonding system used. Hydrolytic or chemical degradation is assumed to be diffusion- and time-dependent; it takes time to penetrate the interface and cause chemical breakdown. However, fatigue degradation should simply be dependent on the magnitude of stress and number of cycles. Chemical degradation has been studied with NaOCl exposure and found to decrease bond strength in microtensile tests [6].

The cervical areas of teeth are subject to regions of stress concentration during chewing [7]. Cervical bending can generate tensile or compressive stress on the tooth structure or

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the bond. This stress is claimed to be a factor in the formation of non-carious cervical lesions (NCCL) by some authors [8] termed "abfraction", while others discount the possibility and emphasize the role of toothbrush abrasion in the formation of NCCLs [9-12]. A recent systematic review concluded that there is little evidence that abfraction exists [13]. An *in vitro* simulation demonstrated material loss from the dentin surface due to the presence of cyclic (fatigue) stresses, although the magnitude of the loss was small [14]. However, once a notch is formed, it does act to concentrate stress in that location and thus is presumed to be a factor in the eventual de-bonding failure of restorations of NCCLs [15,16]. The fatigue behavior of dentin has been examined in recent studies [17,18], but there are no corresponding studies on the fatigue properties of enamel. However, a study on crack propagation showed the dentino–enamel junction acts as a toughening mechanism and resists crack propagation from enamel to dentin [19].

To improve dentin bonding, we need to understand the contributions of possible degradation mechanisms. If hydrolytic degradation is most important, the strategies must be directed toward making the bonded interface more chemically stable in saliva; if fatigue is most important, then toughening of the interface and inhibition of crack propagation should be pursued.

The commonly reported short-term *in vitro* bond strength is a useful screening test but it tells little about the long-term durability of these bonds. Durability of bonds *in vitro* has been discussed in a recent review [1]. Water storage decreases bond strengths over time [20,21], even in the absence of mechanical fatigue.

The purpose of this study was to test the durability of composite/dentin bonds in four-point bending under cyclic fatigue. The hypothesis to be tested was that cyclic sub-critical loads of sufficient magnitude would eventually lead to failure.

2. Materials and methods

The dentin specimens [$n = 12$] were prepared from recently extracted human molars collected according to a protocol approved by the UCSF Institutional Review Board and sterilized by gamma radiation [22]. Teeth were sectioned with a rotating diamond blade in a bucco-lingual direction, first to create a slab and then a rectangular cross-section beam, approximately parallel to the long axis of the tooth, with dimensions of ~1.1 mm × 1.1 mm × 6 mm. The end of each beam was finished with 600 grit wet abrasive paper. The surface for bonding was the occlusal end of each beam, near the middle of the crown. Composite was bonded as follows: (i) the surface was treated with a self-etching primer (SE Bond Primer, Kuraray, Osaka, Japan, lot 00408A) for 20 s, then gently air dried, (ii) bonding resin (SE Bond, Kuraray, Osaka, Japan, lot 00551A) was applied for 20 s and light cured for 10 s, and (iii) composite resin (Filtek Z-250, shade A3, 3M ESPE, St. Paul, MN, Lot #: 3AE 2006-01) was added to the surface and shaped as an extension of the beam. The shaping of the composite was accomplished by using microscope glass slides below and on the sides of the beam. Care was taken to avoid pooling of the bonding resin on the surface; any excess resin was removed by a gentle air stream. After light polymerization and 24 h water storage the beam was finished on 600 grit wet abrasive paper to 0.87 mm × 0.87 mm × 10 mm. Great care was taken during the polishing process to avoid any undue stress on the bonded specimens. Since the bonded surface was a flat square area measuring 1.1 mm × 1.1 mm, the C-factor was very low, resulting in minimal stress from polymerization [23,24]. The specimens were stored in Hanks' Balanced Salt Solution (HBSS) at 25 °C prior to testing. Control specimens of identical size and shape were prepared from solid dentin and solid composite.

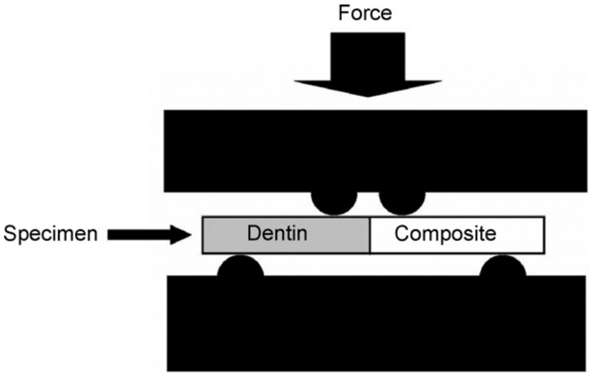

Fig. 1 – Diagram of four-point bend apparatus. Distance between loading points—1.8 mm for upper and 7.2 mm for lower.

All testing was performed on a factory-calibated ELF 3200 mechanical testing machine (EnduraTEC, Minnetonka, MN) under force control in a custom-built four-point bend rig, made from Delrin (Fig. 1), in HBSS at 25 °C. The loading points were spaced 1.8 and 7.2 mm apart; the interface was centered between them. The spacing of the loading points was determined by the size of the beams, which are limited by the size of the dentin beam which can be made from a human molar tooth. Each beam was positioned so that the bonded interface, which was visually discernible, was centered between the inner loading points. Bending strengths, σ_b (in MPa), were computed from the maximum load P (in N), to cause failure, using the standard relationship (ASTM E855/1984):

$$\sigma_b = \frac{3Pa}{bh^2} \times 10^6,$$

where a is the spacing (in meters) between upper and lower loading points, b and h are, respectively, the specimen width and thickness (in meters).

In the first set of experiments (Table 1), solid dentin beams, solid composite beams, and the bonded beams [$n = 6,6,6$ respectively] were tested to failure by increasing the force

Table 1 – Strength of beams in four-point bending

Type of sample	Mean fracture stress in MPa (S.D.)	n	Statistical grouping
Solid dentin	164.4 (9.1)	6	A
Solid composite	164.6 (2.4)	6	A
Bonded beam	90.6 (2.5)	6	B

Table 2 – Number of cycles until fracture for bonded beams ($n = 3$ for each group)

Maximum bending stress (MPa)	Number of cycles (Mean)	Standard deviation
49.2 or less	1,000,000 (no fracture)	0
52.3	691,429	53,037
55.4	42,694	5842
67.7	6423	794
80.0	1436	168
89.8	16	17

linearly at 0.01 mm/s. In the second series of experiments (Table 2), bonded beams [$n = 6$] were subjected to cyclic fatigue loading until failure or 10^6 cycles, whichever occurred first. These specimens were stored in HBSS for 24 h prior to testing in force control at the maximum stresses listed in Table 2, with the minimum stress set at 10% of the maximum stress, i.e., at $R = 0.1$.

3. Results

Bending strengths of the solid composite and solid dentin beams were nearly identical 164.4 ± 9.1 and 164.6 ± 2.4, respectively (Table 1). The control bonded beams had a bending strength of 90.6 ± 2.5, ~55% of the solid dentin and composite beams. The results were analyzed by ANOVA. No significant difference was found between the solid composite and solid dentin beams. The composite–dentin bonded beams were significantly different, ($p < 0.0001$) Stereo light microscopy revealed that all bonded beams failed at the interfacial region. In the second series of experiments, bonded beams subjected to cyclic fatigue load all survived 10^6 cycles when the maximum stresses were below 49.2 MPa. At higher stresses, the beams survived progressively fewer cycles as the applied loads increased up to 89.8 MPa. Only an average of 16 cycles were tolerated at this level (Table 2). Thus, the results show that the interface is susceptible to fatigue, as cycling at subcritical loads eventually causes failure.

4. Discussion

This study examined the effects of bending stresses on the failure resistance of dentin–composite bonds. Results indicate that the interface is weaker than either of the separate materials (when tested as a solid beam).

In the four-point bend test, not commonly used for studying bond strengths, none of the specimens failed cohesively in dentin; all failed adhesively. Shear tests performed with point loading, such as a knife edge, sometimes result in cohesive dentin failures, likely an artifact of the test geometry associated. The point loading results in stress concentrations in a small area and may induce corresponding crack propagation into dentin [25]. However, cohesive dentin failures seldom occur with tensile tests [26] or with the lap shear test which distributes the load more evenly with an enclosure surrounding the composite sample [27]. Shear tests have been criticized as difficult to interpret and to relate to inherent properties of the interface [28]. In the four-point bend geometry, the bottom part of the beam specimen is subjected to tensile stresses which are highest at the surface of the specimen at the interface, and this is where cracks are generally initiated.

The four-point bend specimens in this study failed at the interface, showing that the dentin/composite interface is far less fracture resistant than either dentin or composite. We did not carry out a detailed analysis of the failed interface, only a visual microscope examination. A more detailed analysis might show the more exact travel path of the crack with respect to the layers at the interface, such as the location within or near the hybrid layer.

Cycling at higher stresses, above ~50% of the single-cycle strength, clearly limits the endurance of the interface and demonstrates how subcritical fatigue loading will eventually lead to failure, with the durability of the bond related to stress magnitude.

Previous studies have applied fatigue testing to tooth-composite interfaces in shear [29]. More recently, micro-rotary fatigue testing was applied to microtensile stick specimens [30,31] and it was found that the load at which 50% of the specimens fail after 10^5 cycles was about 30–40% lower than the corresponding micro–tensile bond strength. This particular test is different from the four-point bend in that the beam specimen is machined to a cylindrical shape in the middle of the specimen and the applied force is constantly changing direction.

In our study, cyclic loads of increasing magnitude were used until failure occurred. Fig. 2 shows a typical stress/life (*S/N*) curve, where the number of loading cycles to failure diminishes with increasing stress. Fatigue failure occurred when the applied stress was above 52.3 MPa, or ~58% of the bending strength, although it is possible that failure could have occurred at lower stresses if we had continued the testing beyond 10^6 cycles. The form of this curve is not unlike many other structural materials, with an apparent "fatigue limit", i.e., lower plateau in the *S/N* curve for lives exceeding ~10^6 cycles, at approximately 50% of the single-cycle strength.

While our results suggest that fatigue is a significant mechanism of dentin–composite bond degradation, it does

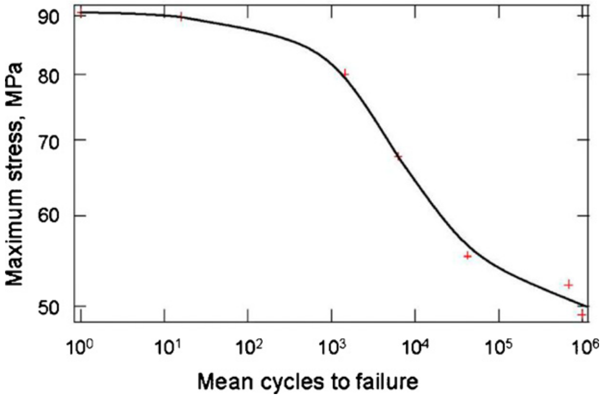

Fig. 2 – Stress/Life (S/N) curve, data corresponding to Table 2: the mean number of cycles survived ($n = 3$ each data point) vs. maximum stress applied.

not answer the question of the relative importance of fatigue versus hydrolytic degradation. Future studies should address this problem by combining water storage with fatigue.

In conclusion, the bending strength of dentin–composite interfaces was found to be approximately 55% of the (single-cycle) bending strength of solid dentin or composite beams. At cyclic loads above 50% of the bending strength, our results clearly show that fatigue damage will eventually break the bond. However, all samples survived for at least 10^6 cycles at stresses of $\sim$50 MPa, which is encouraging, since typical masticatory stress levels that a human tooth experiences are on the order of 20–42 MPa [32,33]. However, these physiologic loads were calculated to estimate the forces on cusps of human molars. Restored teeth with bonded interfaces are subject to different distribution of stresses, some of which may be concentrated at the interface. This apparent endurance strength also compares well with fatigue endurance strengths for human dentin reported in the literature, which range from 25 to 45 MPa, depending upon the cyclic frequency [17].

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