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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 $\sim 0.87 \text{ mm} \times 0.87 \text{ mm} \times 10 \text{ mm}$ 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.

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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 $\sim 1.1\text{ mm} \times 1.1\text{ mm} \times 6\text{ mm}$. 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

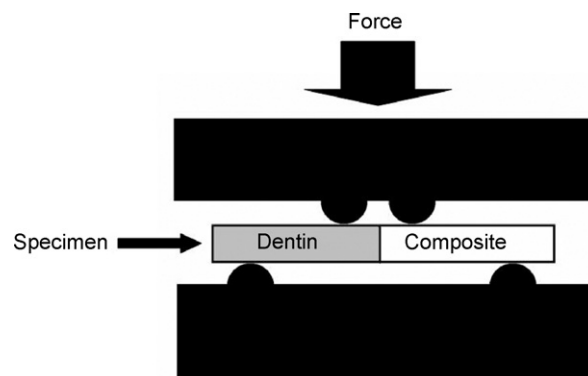


Fig. 1 – Diagram of four-point bend apparatus. Distance between loading points—1.8 mm for upper and 7.2 mm for lower.

and 24 h water storage the beam was finished on 600 grit wet abrasive paper to $0.87\text{ mm} \times 0.87\text{ mm} \times 10\text{ mm}$. 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 $1.1\text{ mm} \times 1.1\text{ mm}$, 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.

All testing was performed on a factory-calibrated 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

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