Four-point bending evaluation of dentin-composite interfaces with various stresses

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Abstract

Fracture properties of composite-dentin beams bonded with a self–etching adhesive were tested following short term pretreatments to simulate potential degradation mechanisms (thermal cycling, immersion in 5% NaOCl, or fatigue cycling). Beams of rectangular cross-section were shaped to a size of ~0.87 x 0.87 x 10 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. Testing was performed in Hanks' Balanced Salt Solution at 25 ˚C .

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 56.3 MPa. Thermo-cycling (5˚ to 55˚C), NaOCl solution immersion, or 10^5 of pre-fatigue cycles did not decrease the strength. Conclusion: Thermal stress, exposure to NaOCL, or 10^5 cycles of mechanical stress does not decrease bond strength of composite bonded to dentin as tested in four-point bending.

Key words: *Dentin, composite, adhesion, four-point bending, fracture, fatigue.*

Introduction

Most dentin bonding agents exhibit acceptable strengths when measured immediately, but deteriorate with time, causing restoration leakage and loss . The deterioration of the bonds can be measured in laboratory studies and also observed clinically as loss of restorations in non-retentive preparations. (1, 2) In general, laboratory studies are more useful for elucidating the mechanisms of deterioration. Two major sources have been identified, namely mechanical fatigue and hydrolytic degradation.

The dentin-composite bond can be stressed mechanically by shrinkage of composite due to polymerization, thermal expansion and contraction, or occlusal forces. Understanding the mechanism of bond degradation will help in the design of new materials and procedures to better resist this degradation.

Enamel-composite bonds without mechanical undercuts have enjoyed 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 two years (2). The failure rates vary with the bonding system used. Hydrolytic or chemical degradation is assumed to be diffusion- and time-dependent; accordingly, 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 the bond. This stress is claimed to be a factor in the formation of non-carious cervical lesions (NCCL) by some authors (8), which is termed "abfraction"; others discount the possibility and emphasize the role of toothbrush abrasion in the formation of NCCL's (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 is thus presumed to be a factor in the eventual debonding failure of restorations of NCCL's (15, 16).

To improve dentin bonding, it is important to discern which mechanism of degradation is most important. If it is hydrolytic degradation, then strategies should be directed towards making the bonded interface more chemically stable in saliva; if it is fatigue, 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. This issue of the durability of bonds in vitro has been discussed in a recent review (1); results show that water storage decreases bond strengths over time (17, 18), even in the absence of mechanical fatigue.

The purpose of this study was to test the strength of composite/dentin bonds in four-point bending under various conditions. The hypothesis to be tested was that thermal stress, NaOCl exposure or mechanical fatigue will decrease the ultimate bond strength of the composite/dentin couple.

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 (19). Teeth were sectioned in a bucco-lingual direction, first to create a slab and then a rectangular cross-section beam with dimensions of ~1.1 x 1.1 x 6 mm. The end of each beam was finished with 600 grit wet abrasive paper. 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 sec, then gently air dried, (ii) bonding resin (SE Bond, Kuraray, Osaka, Japan, lot 00551A) was applied for 20 sec and light cured for 10 sec, 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. After light polymerization and 24 hr water storage the beam was finished on 600 grit wet abrasive paper to 0.87 x 0.87 x 10 mm. The specimens were stored under various conditions prior to testing. Control specimens of identical size and shape were prepared from solid dentin and solid composite.

All testing was performed on an ELF 3200 mechanical testing machine (EnduraTEC, Minnetonka, MN) under force control in a custom-built four-point bend rig, made from Delrin (Figure 1), in Hanks' Balanced Salt Solution (HBSS) at 25˚C. The loading points were spaced 1.8 and 7.2 mm apart; the interface was centered between them. Bending strengths, σb (in MPa), were computed from the maximum load P (in N), to cause failure, using the standard relationship (ASTM E855/1984):

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

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).

Bending strength

For comparison, solid dentin beams, solid composite beams, and the bonded beams [n = 6] were tested to failure by increasing the force linearly at 0.01 mm/sec. The bonded beams were exposed to various conditions prior to testing: (i) control specimens were stored for 24 hr in HBSS [n=6], (ii) another set was thermo cycled between water baths at 5º, 37º and 55ºC for 5, 20 and 5 sec, respectively, for 1000 cycles [n=6], (iii) a third set was placed in 5% NaOCl for 1200 sec [n=6], and (iv) a fourth group was subjected to 10^5 fatigue cycles in the four-point bend apparatus at loads of 0.22 to 2.2 N (i.e., at a stress or load ratio of R ~ 0.1) at a frequency of 5 Hz (force control) which translates into bending stresses of ~ 2.35 and 23.5 MPa (varying slightly with specimen dimensions)[n=6], the maximum stress corresponding to ~40% of the single-cycle bending strength of the control group in Table 1.

Fatigue of dentin-composite interfaces

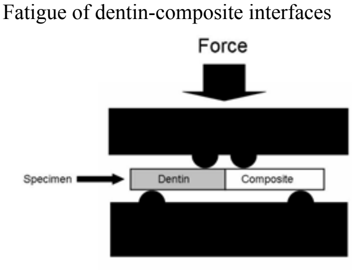

Fig. 1. Diagram of four-point bend apparatus. Distance between loading points - 1.8 for upper and 7.2 mm for lower.

Table 1. Strength of beams in four-point bending.

Type of sample	Pre-test Condition	Mean fracture stress in MPa (SD)	*n*	Statistical grouping
Solid dentin	None	164.4 (9.1)	6	A
Solid composite	None	164.6 (2.4)	6	A
Bonded beam	Stored 24 hr at 37°C	54.1 (8.9)	6	B
Bonded beam	1000 Thermocycles	56.8 (10.5)	6	B
Bonded beam	NaOCl storage for 1200s	49.6 (8.8)	6	B
Bonded beam	Fatigue	56.3 (8.3)	6	B

Results

Bending strengths of the solid composite and solid dentin beams were nearly identical (Table 1). The control bonded beams, which were only stored in fluid(HBSS) and not stressed, had a bending strength of ~33% of the solid dentin and composite beams. Thermo-cycling, exposure to 5% NaOCl solution, or 10^5 fatigue cycles (at stresses of roughly 40% of the single-cycle strength) prior to testing did not decrease the bending strength significantly (one-way ANOVA, $p<0.05$). Stereo light microscopy revealed that all bonded beams failed at the interface.

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). The mean interface strength of 56.3 MPa (Table 1), represents a value of only ~32% of the bending strength of either solid dentin or composite. Bonds vary with the operator, different operators will have different bond strength results, apparently because of minor variations in technique of application of the adhesives to the dentin surface (20-23)., The same is true when restorations placed by different operators are examined for size and frequency of gaps between composite and dentin (22, 24).

The four-point bend test is not commonly used for studying bond strengths. However, in the present study, none of the specimens failed cohesively in dentin; all failed adhesively in the interface. Shear tests performed with point loading sometimes result in cohesive dentin failures, which is likely an artifact of the test geometry associated with stress concentrations at the loading point and corresponding crack propagation into dentin. (25). However, cohesive dentin failures seldom occur with tensile tests. (26) Thus, shear tests have been criticized as difficult to interpret and to relate to inherent properties of the interface (27). The four-point bend specimens in this study also failed at the interface, showing that the dentin/composite interface is far less fracture resistant than either dentin or the composite.

In the first part of this study, there were no differences between the fracture strength of the control group of samples which were pre-exposed to HBSS for 24 hr, and the groups that were subsequently subjected to NaOCl solution or thermo-cycled. This likely is because these degradation treatments were too short to cause a measurable breakdown of the interface. Thermo-cycling stress is commonly used prior to measurement of microleakage or bond strength. The ISO/TS 11405 standard is 500 cycles (28) and we used 1000, but others have suggested that 10,000 cycles is closer to the equivalent of 1 year in vivo (29), which in view of our results seems more appropriate . Also, the effect of thermal cycling is dependent on the geometry of the specimens and the coefficient of thermal expansion of the composite. Thermal changes can produce dimensional changes of gaps around large composite restorations (30); however, the specimens used in the present study were small rectangular beams that were not confined to cavities. Contraction and expansion along the length of the beams should have only minimal effect on the bond. The effect of dimensional changes across the width of the beam should be minimized by the relatively small cross-sectional dimension.

NaOCl was used to attack any collagen in the interface, exposed due to incomplete penetration of polymer into the demineralized collagen. Toledano et al. (2006) found that NaOCl immersion of bonded samples degraded bond strength. However, we detected only a slight trend toward lower bond strengths following a 1200 sec 5% NaOCl immersion. Longer immersion times might affect the bond significantly, as happens with long-term water storage (17). However, the mechanism of hydrolytic or chemical degradation of composite-dentin bonds is not clearly understood and requires further investigation, as either may prove to be a primary mechanism for the observed deterioration of bonds both in vivo and in vitro (1, 2).

No effect of 10^5 subcritical fatigue loading cycles on the eventual bending strength of the bonded specimens was observed; however, the loads were relatively low (roughly 40% of the single-cycle bending strength). 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 (31).

Previous studies have applied fatigue testing to tooth-composite interfaces in shear (32). More recently, micro-rotary fatigue testing was applied to microtensile stick specimens (33, 34) 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.

Conclusions

In conclusion, it was found that the bending strength of dentin-composite interfaces is approximately 32% of the (single-cycle) bending strength of solid dentin or composite beams. These strengths were not significantly affected by short-term thermo-cycling, NaOCl exposure, or 10^5 fatigue cycles at subcritical loads corresponding to stresses of the order of 40% of the bending strength.

References

1. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res. 2005 Feb;84(2):118-32.
2. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater. 2005 Sep;21(9):864-81.
3. Fradeani M, Redemagni M, Corrado M. Porcelain laminate veneers: 6- to 12-year clinical evaluation--a retrospective study. Int J Periodontics Restorative Dent. 2005 Feb;25(1):9-17.
4. Peumans M, De Munck J, Fieuws S, Lambrechts P, Vanherle G, Van Meerbeek B. A prospective ten-year clinical trial of porcelain veneers. J Adhes Dent. 2004 Spring;6(1):65-76.
5. Hikage S, Hirose Y, Sawada N, Endo K, Ohno H. Clinical longevity of resin-bonded bridges bonded using a vinyl-thiol primer. J Oral Rehabil. 2003 Oct;30(10):1022-9.
6. Toledano M, Osorio R, Albaladejo A, Aguilera FS, Osorio E. Differential effect of in vitro degradation on resin-dentin bonds produced by self-etch versus total-etch adhesives. J Biomed Mater Res A. 2006 Apr;77(1):128-35.
7. Geramy A, Sharafoddin F. Abfraction: 3D analysis by means of the finite element method. Quintessence Int. 2003 Jul-Aug;34(7):526-33.
8. Lee WC, Eakle WS. Stress-induced cervical lesions: review of advances in the past 10 years. J Prosthet Dent. 1996 May;75(5):487-94.
9. Litonjua LA, Andreana S, Bush PJ, Tobias TS, Cohen RE. Noncarious cervical lesions and abfractions: a re-evaluation. J Am Dent Assoc. 2003 Jul;134(7):845-50.
10. Litonjua LA, Andreana S, Bush PJ, Tobias TS, Cohen RE. Wedged cervical lesions produced by toothbrushing. Am J Dent. 2004 Aug;17(4):237-40.
11. Litonjua LA, Andreana S, Patra AK, Cohen RE. An assessment of stress analyses in the theory of abfraction. Biomed Mater Eng. 2004;14(3):311-21.
12. Litonjua LA, Bush PJ, Andreana S, Tobias TS, Cohen RE. Effects of occlusal load on cervical lesions. J Oral Rehabil. 2004 Mar;31(3):225-32.
13. Bartlett DW, Shah P. A critical review of non-carious cervical (wear) lesions and the role of abfraction, erosion, and abrasion. J Dent Res. 2006 Apr;85(4):306-12.
14. Staninec M, Nalla RK, Hilton JF, Ritchie RO, Watanabe LG, Nonomura G, et al. Dentin erosion simulation by cantilever beam fatigue and pH change. J Dent Res. 2005 Apr;84(4):371-5.
15. Braem M, Lambrechts P, Vanherle G. Stress-induced cervical lesions. J Prosthet Dent. 1992 May;67(5):718-22.
16. Tyas MJ. The Class V lesion--aetiology and restoration. Aust Dent J. 1995 Jun;40(3):167-70. Review.
17. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, et al. Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res. 2003 Feb;82(2):136-40.
18. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Suzuki K, Lambrechts P. Four-year water degradation of a resin-modified glass-ionomer adhesive bonded to dentin. Eur J Oral Sci. 2004 Feb;112(1):73-83.
19. White JM, Goodis HE, Marshall SJ, Marshall GW. Sterilization of teeth by gamma radiation. J Dent Res. 1994 Sep;73(9):1560-7.
20. Moll K, Fritzenschaft A, Haller B. In vitro comparison of dentin bonding systems: effect of testing method and operator. Quintessence Int. 2004 Nov-Dec;35(10):845-52.
21. Bouillaguet S, Degrange M, Cattani M, Godin C, Meyer JM. Bonding to dentin achieved by general practitioners. Schweiz Monatsschr Zahnmed. 2002;112(10):1006-11.
22. Finger WJ, Balkenhol M. Practitioner variability effects on dentin bonding with an acetone-based one-bottle adhesive. J Adhes Dent. 1999 Winter;1(4):311-4.
23. Sano H, Kanemura N, Burrow MF, Inai N, Yamada T, Tagami J. Effect of operator variability on dentin adhesion: students vs. dentists. Dent Mater J. 1998 Mar;17(1):51-8.
24. Jacobsen T, Söderholm KJ, Yang M, Watson TF. Effect of composition and complexity of dentin-bonding agents on operator variability--analysis of gap formation using confocal microscopy. Eur J Oral Sci. 2003 Dec;111(6):523-8.
25. Eick JD, Robinson SJ, Chappell RP, Cobb CM, Spencer P. The dentinal surface: its influence on dentinal adhesion. Part III. Quintessence Int. 1993 Aug;24(8):571-82.
26. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. Dent Mater. 1994 Jul;10(4):236-40.
27. Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. J Dent. 1989 Apr;17(2):61-7.
28. ISO, Dental materials-Testing of adhesion to tooth sturcture. 2003.
29. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent. 1999 Feb;27(2):89-99.
30. Staninec M, Mochizuki A, Tanizaki K, Jukuda K, Tsuchitani Y. Interfacial space, marginal leakage, and enamel cracks around composite resins. Oper Dent. 1986 Winter;11(1):14-24.
31. Staninec M, Kim P, Marshall GW, Ritchie RO, Marshall SJ. Fatigue of dentin-composite interfaces with four-point bend. Dent Mater. 2007 Nov 8;
32. Ruse ND, Shew R, Feduik D. In vitro fatigue testing of a dental bonding system on enamel. J Biomed Mater Res. 1995 Mar;29(3):411-5.
33. De Munck J, Braem M, Wevers M, Yoshida Y, Inoue S, Suzuki K, et al. Micro-rotary fatigue of tooth-biomaterial interfaces. Biomaterials. 2005 Apr;26(10):1145-53.
34. De Munck J, Van Landuyt KL, Coutinho E, Poitevin A, Peumans M, Lambrechts P, et al. Fatigue resistance of dentin/composite interfaces with an additional intermediate elastic layer. Eur J Oral Sci. 2005 Feb;113(1):77-82.

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