FAILURE CRITERIA OF DENTIN-RESIN TENSILE BOND TEST—A FRACTURE MECHANICS APPROACH

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Introduction

Interfacial properties are of crucial importance in dentistry, where adhesive bonding agents are used to bond restorative resin composites to natural tooth materials (enamel and dentin). Interfacial strength has to be evaluated accurately before reliable clinical performance can be predicted or structure-property relationships can be studied. The most popular methods to determine a bond strength in dental research are the shear and tensile tests. In these tests adhesive strength is simply defined as the measured failure load divided by the original cross-sectional area of the bonded surface. This presumes an approximately uniform stress distribution at the cross-section. However, it has been demonstrated with finite element analysis that the stress distribution at the interface is not uniform in these tests (1). The so-called “average stress” or “nominal stress” values of conventional bond strength tests are unlikely to represent the true interfacial failure stress. It has been suggested that a more promising approach to determine the bond strength is based on interfacial fracture mechanics (2,3), which represents the real local effort of the creation in a new crack surface.

In general engineering the attention has already been focused increasingly on the description of mechanical failure based on fracture mechanics rather than on conventional stress-based criteria. There is a large intellectual knowledge base which supports the interpretation of fracture phenomena. The fracture mechanics approach has become even more attractive due to its application to the strength and function of interfaces between dissimilar materials (4). It is reasonable to assume that these engineering ideas will carry over into biology and biomechanics and should help rationalize the biomechanical success and failures of bonded biological structures such as the dentin-enamel complex (5).

The current conventional tensile bond strength test is well established in many dental materials laboratories. The acceptance of a new interfacial strength evaluation technique would be strongly encouraged if the general testing configuration and the ease of use could be retained. The most simple solution to this dilemma is the inclusion of a precrack at the adhesive interface of the tensile test specimen, effectively converting the nominal bond strength test into a fracture mechanics test.

However, it is of first importance that the application of the criteria of fracture mechanics to such a test configuration be theoretically justified before proceeding further towards the experimental stage. This paper presents a theoretical basis for the proposed interfacial fracture toughness test. The specific aim of this paper is to define the failure criteria of a tensile bond test by a fracture mechanics approach.

Figure 1. Diagrammatic representation of the conventional tensile bond test of composite resin bonded to human dentin.

Analytical equations are derived and finite element analysis is used to calibrate the stress intensity factor and the strain energy release rate.

Analytical Fracture Mechanics Parameters Derivation

The two dimensional configuration of the conventional cylindrical dentin-resin tensile bond test specimen is shown in Figure 1. The basic notation depicted in Figure 2 is used to develop the fracture mechanics parameters.

The expression for mode I (tensile) fracture toughness, G_{IC} , is based on the energy theory. The energy required to advance a crack an increment, da , is given as

$$dW = G_{IC} \cdot b \cdot da \quad (1)$$

where G_{IC} is the critical strain energy release rate and b is the instantaneous crack front width at a critical crack length, $a + da$. Irrecoverable work, dW , when the crack advances an amount da , can be expressed in terms of the elastic compliance change while loading the material. Thus the irrecoverable work is expressed as

$$dW = \frac{1}{2} \cdot F_c \cdot dZ = \frac{1}{2} \cdot F_c^2 \cdot dC \quad (2)$$

Figure 2. Geometric illustration of the bonded area. D, the diameter of the original bonded area; a , crack length; b , instantaneous crack front width. Dashed arrow indicates fracture path.

where F_C is the critical failure load, dC is the incremental change in compliance and dZ is the incremental change in loading displacement due to crack advance, da . Combining equations (1) and (2) will give the critical strain energy release rate as the classical compliance equation from linear elastic fracture mechanics (LEFM) (6). That is,

$$G_{IC} = \frac{F_C^2 \cdot dC}{2b \cdot da} \quad (3)$$

b is a function of a and D , where D is the original diameter of the bonded area.

$$b = 2 \cdot \sqrt{\frac{D^2}{4} - \left(\frac{D}{2} - a\right)^2} \quad (4)$$

Consider a dimensionless variable α instead of a , $\alpha = a/D \cdot b$ can be written as

$$b = 2D \cdot \sqrt{\alpha \cdot (1 - \alpha)} \quad (5)$$

Thus, equation (3) can be derived as

$$G_{IC} = \frac{F_C^2 \cdot dC}{4 \cdot D^2 \cdot \sqrt{\alpha \cdot (1 - \alpha)} \cdot d\alpha} \quad (6)$$

After some manipulation, equation (6) becomes

$$G_{IC} = \frac{F_C^2 \cdot (E^* \cdot D \cdot dC)}{4 \cdot D^3 \cdot E^* \cdot \sqrt{\alpha \cdot (1 - \alpha)} \cdot d\alpha} \quad (7)$$

$E^* \cdot D \cdot dC$ is a dimensionless compliance. E^* is the combined elastic modulus of the tensile bond test specimen. The results of finite element simulation showed $dC = dC_a + dC_d + dC_r$ and $dC_r \cdot E_r = dC_d \cdot E_d$, where subscripts a , d and r represent adhesive, dentin and resin, respectively. Moreover, dC_a and $dC_a \cdot E_a$ were found to be negligible when the adhesive thickness was about 50 μm . Consequently, the following equations can be obtained:

$$dC \cdot E^* = dC_d \cdot E_d + dC_r \cdot E_r + dC_a \cdot E_a = dC_d \cdot E_d + dC_r \cdot E_r = 2 \cdot dC_d \cdot E_d \quad (8)$$

$$dC = dC_d + dC_r + dC_a = \frac{dC_d \cdot E_d}{E_r} + \frac{dC_d \cdot E_d}{E_d} = dC_d \cdot E_d \cdot \left(\frac{1}{E_r} + \frac{1}{E_d}\right) \quad (9)$$

Thus,

$$E^* = 2 \cdot \left(\frac{1}{E_r} + \frac{1}{E_d}\right)^{-1} \quad (10)$$

Equation (7) can be reduced to

$$G_{IC} = \frac{F_C^2}{D^3 \cdot E^*} \cdot f\left(\frac{a}{D}\right) \quad (11)$$

where $f(a/D) = Y^2$. Y is the dimensionless stress intensity factor coefficient and is a function of crack length. It is independent of the specimen material as long as the scaled specimen configuration remains constant. From equations (7) and (11), Y can be expressed as

$$Y = \left(\frac{E^* \cdot D \cdot dC}{4 \cdot \sqrt{\alpha \cdot (1 - \alpha)} \cdot d\alpha}\right)^{1/2} \quad (12)$$

Figure 3. A 3-dimensional finite element model for the dentin-resin tensile bond test specimen.

and therefore

$$G_{IC} = \frac{F_C^2}{D^3 \cdot E^*} \cdot Y^2 \quad (13)$$

Generally (6),

$$G_{IC} = \frac{K_{IC}^2}{E^*} \quad (14)$$

Thus, combining equations (13) and (14) we may get

$$K_{IC} = \frac{F_C}{D^{3/2}} \cdot Y \quad (15)$$

Calibration of the Dimensionless Stress Intensity Factor Coefficient (Y)—Finite Element Analysis

The use of numerical techniques for the calibration of fracture mechanics parameters is gaining on experimental calibrations (7). Although this shift eliminates the variation inherent to experimentation, it should be kept in mind that numerical techniques are approximate methods. Therefore, special care was taken in the definition of a finite element mesh for a dentin-resin tensile bond test specimen used for this calibration. Error analyses were run to ensure that the used mesh provided sufficient accuracy without loss of detail or structural response, and within reasonable margins of available computational hardware.

A three-dimensional finite element model was constructed using Mentat II v2.1.3 (Nippon Marc Analysis Research Corporation, Tokyo, Japan) on a graphics workstation (Silicon Graphics, Inc., Mountain View, CA). The dimensions of the model were taken from experimental test specimens. A thin strip (50 μm) of adhesive Teflon tape (#5490 extruded PTFE film tape, Scotch, 3M Company, St. Paul, MN, USA) with a punched hole (3.3 mm in diameter) is normally utilized to precisely define the bonding area. Due to symmetry only one half of the specimen was modeled, as shown in Figure 3. Isoparametric eight-node hexahedral elements were utilized in this study. A tensile load of 100N was applied, uniformly distributed at the unbonded composite cylinder top surface. The nodes at the dentin

Figure 4. Stress intensity factor coefficients (Y) as a function of normalized crack lengths (α).

base were restrained for all degrees-of-freedom. Tying was used to connect nodes in the mid-plane of the adhesive layer (all degrees-of-freedom). In this analysis of a dentin-resin tensile bond test the used Young's moduli and Poisson's ratios were: $E_d = 18.3$ GPa, $\nu_d = 0.30$ (human dentin); $E_r = 16.6$ GPa, $\nu_r = 0.24$ (restorative composite resin) (8); and $E_a = 2.1$ GPa, $\nu_a = 0.25$ (dentin bonding adhesive) (3).

The model was analyzed using the MARC K6.1 finite element program (Nippon Marc Analysis Research Corporation, Tokyo, Japan) on a Silicon Graphics workstation (Indy, Silicon Graphics, Inc., Mountain View, CA) and a Cray J916 at the NCHC (National Center for High-Performance Computing, Taiwan, ROC). An advancing crack was simulated by incrementally releasing the tied nodes at the adhesive mid-plane, starting from one edge. The crack propagation increments to the other end of the adhesive cross-section ranged from $1.64 \mu\text{m}$ to 0.12 mm, depending on the local element refinement. Twenty-four steps of loadline compliance versus crack length were recorded. The output from the finite element analysis was post-processed in Mentat. The dimensionless stress intensity factor coefficients (Y) could then be calculated using equation (12).

Results and Discussion

Pronounced stress concentrations of the normal stresses perpendicular to the interface, maximum principal stresses and equivalent Von Mises stresses are evident at the edge of the adhesive layer during tensile loading. Considering the stress distribution, crack propagation is likely to take place along the adhesive layer.

If the tensile bond test is running under load control, the sample will usually fail catastrophically. However, under displacement control steady state crack growth may be encountered. For both modes the failure of the dentin-resin bond will emanate from the edge of the adhesive layer due to the stress concentration. This observation is another reason to doubt the value of an "average stress" or "nominal stress" approach for interfacial strength determination.

Figure 4 shows the dimensionless stress intensity factor coefficients along the adhesive layer as a function of normalized crack length for tensile loading. This curve increases monotonically with the crack length. This implies that the tensile test specimen is potentially unstable after crack initiation.

The tensile bond test in dental materials research is not only controversial due to its mechanics, but also where reproducibility is concerned. Large variations are reported for bond strength values from

different investigators (9,10). Coefficients of variation are between 65% and 111% (11). The results of this study may partly explain this high variation. As shown in Figure 4, the stress intensity factor coefficients increase rapidly for $a \leq 0.03D$. This suggests that the measurement of bond strength or fracture toughness is strongly affected by the length of the initiating crack as well as the condition of the bond at the edge of the adhesive, and will likely result in high variations of experimental fracture toughness measurements. However, the stress intensity factor coefficients increase at a much lower rate if $a > 0.03D$. Therefore, a modified tensile bond test with a precrack greater than $0.03D$ may reduce the variation of the experimental measurements as well as facilitate the assessment of fracture toughness. The stress intensity factor coefficients as a function of the normalized crack length can be fitted by fourth-degree polynomials as follows ($a > 0.03D$):

$$Y = 0.571 + 1.115\alpha - 2.027\alpha^2 + 5.857\alpha^3 - 2.677\alpha^4 \quad (16)$$

A modified tensile bond test specimen having a straight through notch with a precrack length of $0.05D$ is proposed as an alternative interfacial strength sample. Using composite resin bonded to human dentin as an example, the stress intensity factor coefficient can be calculated by equation (16) as: $Y = 0.62$. Consequently, the equations (13) and (15) which are the general forms for determination of fracture toughness in terms of K_{IC} and G_{IC} can be rewritten in the following forms:

$$G_{IC} = 0.39 \cdot \frac{F_C^2}{E^* \cdot D^3} \quad (17)$$

$$K_{IC} = 0.62 \cdot \frac{F_C}{D^{3/2}} \quad (18)$$

In an experimental setting, a precrack can be created by a thin ($50 \mu\text{m}$) Teflon tape, with the bonding area exactly defined by cutting its projection out of the Teflon tape, using a customized punch.

Conclusions

The conventional tensile bond test, which is used to determine a nominal interfacial strength value, is attractive because of its simplicity. However, the mechanical behavior of this test is likely to generate unrealistic results. In this study failure along the adhesive interface of the tensile bond test has been assessed by a fracture mechanics approach. The equations for the stress intensity factor coefficient, and hence the critical stress intensity factor and the critical strain energy release rate, have been derived as a function of the crack length. The stress intensity factor coefficients have been calibrated by means of finite element analysis. The stress intensity factor coefficient increases monotonically as the normalized crack length increases. This implies that the tensile test specimen is potentially unstable, especially at a crack length $\leq 0.03D$. It is hypothesized that the reported high variation of experimental results is partly due to the length of the precrack and the local conditions at the edge of the adhesive interface. Based on these considerations, a modified tensile bond test specimen with a $0.05D$ precrack has been proposed. Further experimental research should be carried out to validate the concept of this modified testing methodology.

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