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On the durability of resin–dentin bonds: Identifying the weakest links

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ABSTRACT

Fatigue of resin–dentin adhesive bonds is critical to the longevity of resin composite restorations.

Objectives. The objectives were to characterize the fatigue and fatigue crack growth resistance of resin–dentin bonds achieved using two different commercial adhesives and to identify apparent “weak-links”.

Methods. Bonded interface specimens were prepared using Adper Single Bond Plus (SB) or Adper Scotchbond Multi-Purpose (SBMP) adhesives and 3M Z100 resin composite according to the manufacturers’ instructions. The stress–life fatigue behavior was evaluated using the twin bonded interface approach and the fatigue crack growth resistance was examined using bonded interface Compact Tension (CT) specimens. Fatigue properties of the interfaces were compared to those of the resin–adhesive, resin composite and coronal dentin.

Results. The fatigue strength of the SBMP interface was significantly greater than that achieved by SB ($p \leq 0.01$). Both bonded interfaces exhibited significantly lower fatigue strength than that of the Z100 and dentin. Regarding the fatigue crack growth resistance, the stress intensity threshold (ΔK_{th}) of the SB interface was significantly greater ($p \leq 0.01$) than that of the SBMP, whereas the ΔK_{th} of the interfaces was more than twice that of the parent adhesives.

Significance. Collagen fibril reinforcement of the resin adhesive is essential to the fatigue crack growth resistance of resin–dentin bonds. Resin tags that are not well hybridized into the surrounding intertubular dentin and/or poor collagen integrity are detrimental to the bonded interface durability.

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1. Introduction

Fatigue of materials is gaining recognition in the field of restorative dentistry. Indeed, fatigue is now recognized as either the primary mode of failure or a contributing mechanism for both direct and indirect restoratives [e.g. 1,2]. As such, the fatigue properties of dental materials and the durability of their bonds to tooth structure are important metrics of performance.

Fatigue failure of dental composites has received attention for over two decades [e.g. 3–11]. Recent *in vitro* evaluations and clinical outcomes suggest that the fatigue properties of resin composites may be useful in predicting clinical performance [12,13]. Lohbauer et al. [1] emphasized the importance of considering the fatigue strength of resin composites during materials selection, and commented that experimental investigations on fatigue performance should involve clinically relevant geometries and loading conditions.

While chemical degradation of the bonded interface is a longstanding concern, cyclic stresses are considered a contributing factor to its progressive degradation over time [14,15]. In fact, the bonded interface has been regarded as the weakest link of resin–dentin bonds [14]. Studies on fatigue of tooth–resin bonds began almost as early as those on resin composites [16,17]. Yet, relatively few investigations have been reported in this area [18–24]. There has been an attempt at modeling the fatigue behavior of the resin–dentin adhesive interface using the finite element method [25]. Results from prior studies have distinguished that bonded interfaces are not resilient to cyclic loads, with fatigue strengths reported to be as low as 25% of the static strength.

Belli et al. [24] recommended that a combination of experimental approaches should be used to obtain a broader picture of the bonded interface performance. In a comparison of two different adhesive systems, they applied both static and cyclic methods of loading. While there was no difference in strength of the two adhesive systems under static loads, there was a significant difference in their fatigue strength. The difference in fatigue behavior was attributed to an unequal population of flaws within the adhesive layers. If flaws are located at the interface, within either the resin adhesive or the hybrid layer, then the “initiation” phase of the fatigue life is relatively short. In these instances, the bonded interface durability will be related to its resistance to the “propagation” of these defects via cyclic extension. Soappman et al. [26] proposed evaluating the fatigue crack growth resistance of resin–dentin bonds using a conventional fracture mechanics approach. However, that investigation did not perform a complimentary evaluation of the interface via static loading or stress-life fatigue. All of these modes of loading are relevant to the durability of tooth–resin composite bonded interfaces.

The present study adopts three approaches for evaluating the durability of resin–dentin adhesive bonds. Specifically, the flexure strength, stress-life fatigue and fatigue crack growth properties of dentin–resin bonded interfaces were characterized, and the results were compared with those obtained for the individual materials (i.e. dentin, resin adhesive and resin composite). The overall objective of this investigation was to evaluate the fatigue resistance of dentin bonds developed

using two selected commercial materials and to assess the “weakest-link”.

2. Materials and methods

The durability of resin–dentin bonds resulting from two different commercial adhesive systems was evaluated in terms of the stress-life fatigue and fatigue crack growth responses. Both approaches involved sections of human coronal dentin, which was obtained from caries-free third molars. The teeth were obtained from participating clinics in Maryland according to an approved protocol (#Y04DA23151). The only record of the teeth was the age ($18 \leq \text{age} \leq 30$ years) of the donor. The teeth were sectioned using a slicer/grinder (Chevalier Smart-H818II, Chevalier Machinery, Santa Fe Springs, CA, USA) with diamond abrasive slicing wheels (#320 mesh abrasives) and copious water coolant. All sections were obtained from the mid coronal region with necessary geometry for the specimens (Fig. 1). The remaining materials included either Single Bond Plus (SB: 3M ESPE) or Scotchbond Multi-Purpose (SBMP, 3M ESPE) adhesive and Z100 (3M ESPE) resin composite.

The flexure strength and stress-life fatigue behavior was evaluated using the Twin Bonded Interface (TBI) approach. The specimens (Fig. 1(a)) were prepared using a molding process after Mutluay et al. [27]. Briefly, the adhesive (SB or SBMP) was applied to the two opposing surfaces of rectangular beams (roughly $2 \times 2 \times 10 \text{ mm}^3$) of mid-coronal dentin (Fig. 1(a)) according to the manufacturer's recommendations. Bonding with each of the two adhesive systems was preceded by the recommended 15 s etch (SB 37% phosphoric etchant) and rinse. Then the beams were placed within a dedicated mold with the tubules oriented nominally parallel to the bonding interface, akin to the walls of a Class II preparation. Restorative resin composite (Z100, 3M ESPE) was applied incrementally from the dentin beam surface and distributed incrementally to fill the mold cavities on each side of the dentin beam. The composite was cured for 40 s on both sides using a quartz–tungsten–halogen light-curing unit (Demetron VCL 401, Demetron, CA, USA) with output intensity of 600 mW/cm^2 and with tip diameter wider than 10 mm. The bonded sections were released from the mold and sectioned using the slicer/grinder to obtain TBI specimens roughly $2 \text{ mm} \times 2 \text{ mm} \times 12 \text{ mm}$ (Fig. 1(a)). Control specimens consisting of the Z100 resin composite only were prepared using the molding and sectioning process. A total of 105 TBI specimens were prepared, which consisted of 35 for each of the three groups. All of the specimens were inspected, polished lightly with hydration using #600 mesh emery paper, and stored in HBSS at room temperature (22°C) for a minimum of 48 h prior to further evaluation.

Bonded interface Compact Tension (CT) specimens were prepared using identical bonding procedures and a molding technique similar to that used for the TBI specimens. Briefly, sections of dentin were obtained representing half of the completed CT specimen geometry (Fig. 1(b)). Either the SB or SBMP was applied to one edge according to the manufacturer's recommendations; etching preceded application of the adhesives

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