

Durability of adhesive bonds to tooth structure involving the DEJ<sup>☆</sup>Enas Elbahie<sup>a</sup>, Dylan Beitzel<sup>a</sup>, Mustafa Murat Mutluay<sup>a,b</sup>, Hessam Majd<sup>a</sup>, Mobin Yahyazadehfar<sup>a</sup>,  
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## ABSTRACT

The importance of the Dentin Enamel Junction (DEJ) to the durability of adhesive bonds to tooth structure is unclear. In fact, no investigation has been reported on contributions of the DEJ to the fatigue resistance of the bonded interface. In this study, the durability of adhesive bonds to tooth structure involving the DEJ was quantified and compared to that of adhesive bonds to enamel only, not including the DEJ. Two different configurations of enamel bonding were considered, including when tensile stress is focused on the outer enamel (occlusal configuration) or the inner decussated enamel (decussated configuration). The resistance to failure for all bonded interfaces was assessed under both static and cyclic loading to failure. Results showed that the durability of the bonded interfaces was primarily a function of their resistance to crack initiation and growth. The bonded interface strength involving the DEJ was significantly ( $p \leq 0.05$ ) greater than that of bonds to enamel only with occlusal configuration, under both static and cyclic loading. While the fatigue strength of bonds involving the DEJ was approximately 20% greater than that for enamel bonds with occlusal configuration (7.7 MPa) it was lower than that of enamel with the decussated configuration. The DEJ deterred cracks from extending readily into the dentin but it did not prevent fatigue failure. These results suggest that the durability of bonds to enamel are most dependent on the enamel rod decussation and that the DEJ plays a minor role.

## 1. Introduction

In the field of restorative dentistry, tooth structure that is undermined by caries is removed using dental burs and replaced with a restorative material of appropriate color and mechanical properties (Fig. 1). In today's practice, resin composites are the most common restorative material. These materials are light-curing particulate reinforced monomers. They are bonded to tooth structure, including the dentin and enamel, using resin adhesives.

One of the primary contributions to the failure of adhesive bonds to tooth structure is degradation by cyclic loading and fatigue (Van Meerbeek et al., 2003; Spencer et al., 2010; Pashley et al., 2011). Surprisingly though, the performance of adhesive bonds to dentin and enamel has been assessed primarily by microtensile tests and static loads to failure (Pashley et al., 1999; De Munck et al., 2005a). Kotousov et al. (2011) performed an interesting analytical analysis concerning the initiation and propagation of adhesion failures in composite restorations. The fatigue properties of the bonded interface, which define

its resistance to failure under cyclic loading, has received rather limited attention overall. In addition, contraction stresses develop in the resin composite as a result of light curing and generate residual tensile stresses (Yamamoto et al., 2011). These stresses can facilitate cracking and further decreases the fatigue resistance of the bonded interface between the restoration and tooth structure.

The durability of adhesive bonds to tooth structure is a major concern in the field of restorative dentistry and the majority of studies on this topic have focused on dentin bonding (Arola, 2017). In comparison, relatively few studies have been focused on the fatigue properties of adhesive bonds to enamel (Ruse et al., 1995; De Munck et al., 2005b; Erickson et al., 2006; 2008; 2009a; Barkmeier et al., 2009). In studies comparing the durability of adhesive bonds to dentin and enamel, the fatigue resistance of enamel bonds is substantially lower than that for dentin (De Munck et al., 2005b; Yahyazadehfar et al. 2013a). For instance, Yahyazadehfar et al. (2013a) reported that the apparent endurance limit of the bonded interface to enamel was nearly 40% lower than that achieved in bonding to dentin. Surprisingly, fatigue failure

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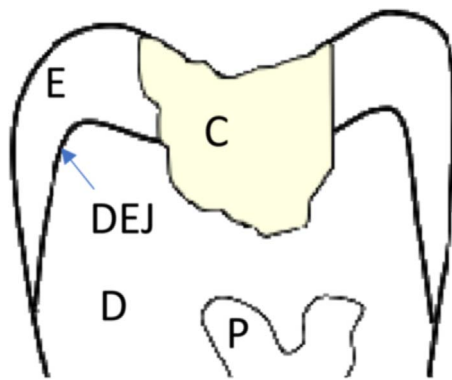


Fig. 1. Schematic diagram for the crown portion of a restored tooth. Highlighted in this diagram are the resin composite restoration (C), the enamel (E), the dentin (D), the pulp (P) and the Dentin Enamel Junction (DEJ). The interface between the composite and surrounding tooth structure is the bonded interface.

occurred through the initiation and propagation of cracks in the enamel and not by failure of the adhesive. If that is the predominate mode of failure in bonds involving enamel, using restorative approaches that are capable of arresting the damage and preventing crack propagation from the enamel to the underlying dentin could have clinical impact.

Cracks that initiate in the enamel on the occlusal surface of teeth must pass through the Dentin Enamel Junction (DEJ) to reach the underlying dentin and pulp. The DEJ is a complex interface that has been considered one of the most interesting in nature (Marshall et al., 2001; Marshall et al., 2003). The width of this interface is under debate, with measures of just over a few microns (Habelitz et al., 2001) to an average of 10  $\mu\text{m}$  and more (Gallagher et al., 2003; Fong et al., 1999). Studies focused on the region surrounding the DEJ using nanoindentation have shown that the elastic modulus and hardness undergo a gradual transition from the hard and stiff qualities of the enamel to those of the more compliant and soft dentin (Fong et al., 1999; Marshall et al., 2001). Perhaps the most interesting quality of the DEJ is its apparent contribution to the resisting crack extension from the enamel towards the dentin (Lin and Douglas, 1994; Xu et al., 1998; Marshall et al., 2003; Imbeni et al., 2005). Prior evaluations concerned with the fracture resistance of the DEJ were not performed on restored tooth structure. Furthermore, although the mechanics of fracture at the adhesive interface between tooth structure and resin composite has been examined, no study has identified how the DEJ participates in resisting the growth of cracks within or near the bonded interface in restored teeth.

The studies concerning fracture of the DEJ suggest that its complex microstructure results in a number of unique toughening mechanisms

that operate in concert to promote crack arrest (White et al., 2005). Attempts at characterizing the fracture toughness of the DEJ have resulted in values ranging from roughly 0.5  $\text{MPa m}^{0.5}$  to over 3  $\text{MPa m}^{0.5}$  (Lin and Douglas, 1994; White et al., 2000; Dong and Ruse, 2003; Imbeni et al., 2005). These values are not substantially greater than those reported for dental enamel (Bajaj and Arola, 2009a; Bechtle et al., 2010; Yahyazadehfar et al., 2013b). Therefore, it is not clear whether the DEJ or the enamel plays a more important role on the bonded interface durability of teeth restored with resin composite restorations.

The DEJ appears to bestow the tooth with resistance to fracture from cracks initiating within the enamel. It could serve an equally important role in resisting fatigue failure of resin composite restorations bonded to enamel. However, to the authors' knowledge, no study has evaluated the contributions of the DEJ to the durability of resin-enamel adhesive bonds under cyclic loading. In this investigation, a combination of efforts involving experiments and a finite element analysis were performed to determine: 1) if the DEJ increases the durability of adhesive bonds to dental enamel, and 2) if the DEJ is capable of arresting fatigue damage that develop in the enamel of restored teeth. The overall objective was to develop new understanding concerning the contribution of the DEJ to the durability of adhesive bonds to tooth structure.

## 2. Materials and methods

Twin Bonded Interface (TBI) specimens were prepared from sections of caries-free human third molars that were obtained from participating clinics in the state of Maryland. The teeth were obtained with record of donor age ( $18 \leq \text{age} \leq 30$ ) and gender according to a protocol approved by the University of Maryland Baltimore County (#Y04DA23151). The teeth were stored in Hank's Balanced Salt Solution (HBSS) for less than 1 month, and then 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 spray. Sections of enamel, or dentin and enamel encompassing the DEJ, were obtained from the cuspal regions of the donor teeth as shown in Fig. 2(a).

The specimens were prepared using a special molding technique after Mutluay et al. (2013a, 2013b) and Yahyazadehfar et al. (2013a). Briefly, the lingual and buccal aspects of the enamel cubes were etched for 15 s (34% phosphoric acid gel), followed by application of primer to these surfaces according to the manufacturer's instructions. Adhesive was applied to the primed surfaces (Clearfil SE Bond, Lot 062127, Kuraray America) and cured for 10 s with a quartz-tungsten-halogen light-curing unit (Demetron VCL 401, Demetron) with calibrated output intensity of 600  $\text{mW}/\text{cm}^2$  and tip diameter wider than 10 mm. The

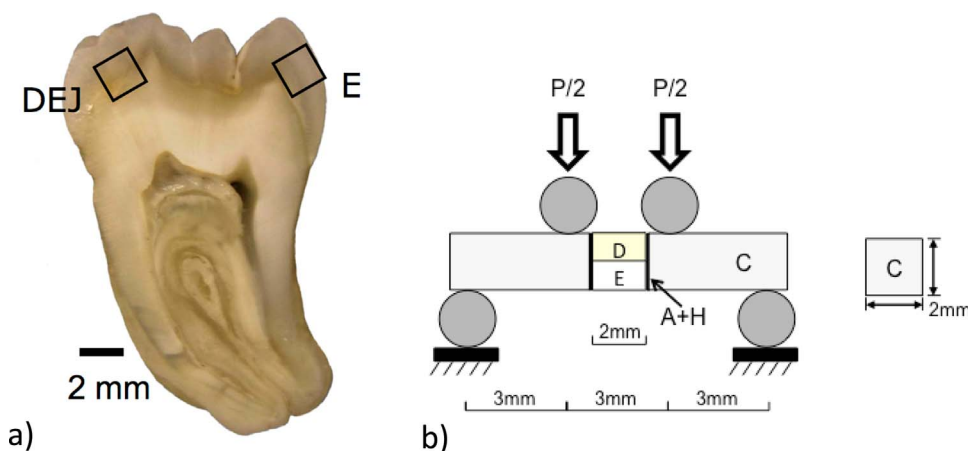


Fig. 2. Preparation of the twin bonded interface specimens and flexural loading. a) A primary tooth section from the bucco-lingual plane with 2 mm thickness. Secondary sectioning was used to prepare  $2 \times 2 \times 2 \text{ mm}^3$  cubes of material consisting of enamel only (E), or the Dentin Enamel Junction (DEJ). The specimen geometry after preparation was approximately  $2 \times 2 \times 12 \text{ mm}^3$ . (b) the four-point flexure configuration for evaluating the bonded interface. In the twin bonded interface specimen (TBI) there are two bonded interfaces that consist of the adhesive and hybrid layer (A+H). For the enamel specimens loading was conducted with either the outer enamel (nearest the occlusal surface) subjected to tension or the inner enamel (nearest the DEJ) subjected to tension. These two configurations are regarded as the "occlusal" and "decussated" arrangements, respectively. The DEJ specimens (as shown) were arranged for cyclic loading such that the enamel was subjected to cyclic tension and the

dentin (D) was on the compression side.

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