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Degradation in the fatigue crack growth resistance of human dentin by lactic acid☆

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ABSTRACT

The oral cavity frequently undergoes localized changes in chemistry and level of acidity, which threatens the integrity of the restorative material and supporting hard tissue. The focus of this study was to evaluate the changes in fatigue crack growth resistance of dentin and toughening mechanisms caused by lactic acid exposure. Compact tension specimens of human dentin were prepared from unrestored molars and subjected to Mode I opening mode cyclic loads. Fatigue crack growth was achieved in samples from mid- and outer-coronal dentin immersed in either a lactic acid solution or neutral conditions. An additional evaluation of the influence of sealing the lumens by dental adhesive was also conducted. A hybrid analysis combining experimental results and finite element modeling quantified the contribution of the toughening mechanisms for both environments. The fatigue crack growth responses showed that exposure to lactic acid caused a significant reduction ($p \le 0.05$) of the stress intensity threshold for cyclic crack extension, and a significant increase ($p \le 0.05$) in the incremental fatigue crack growth rate for both regions of coronal dentin. Sealing the lumens had negligible influence on the fatigue resistance. The hybrid analysis showed that the acidic solution was most detrimental to the extrinsic toughening mechanisms, and the magnitude of crack closure stresses operating in the crack wake. Exposing dentin to acidic environments contributes to the development of caries, but it also increases the chance of tooth fractures via fatigue-related failure and at lower mastication forces.

1. Introduction

Human dentin is a fascinating structural material. It serves as an elastic foundation for the enamel and as a protective enclosure for the central pulp of teeth. This tissue occupies the majority of the tooth by volume and is a composite of collagen (~35%v), apatite minerals (~45%v) and fluid (~20%v) [1]. Dentin is considered a porous material as thousands of cylindrical tubules (ranging from roughly 0.5 to 2 μm in diameter) are distributed from the pulp outwards to the Dentin-Enamel Junction (DEJ). Each lumen is surrounded by a highly mineralized peritubular cuff (~1 μm thick) of hydroxyapatite that is embedded within a matrix of mineralized collagen fibrils regarded as the intertubular dentin [2,3]. The mechanical behavior of human dentin is controlled by its microstructure, which is dependent in the spatial variations (e.g. [4,5]), loading orientation (e.g. [6–8]), and biological aging (e.g. [9–12]).

Resin composites have become the primary material for restorations placed in anterior and posterior teeth [13–15]. However, several studies indicate that the average service life of resin composites is lower than that of the materials they have replaced. The principal failure modes of teeth restored with resin composites fillings are degradation of the restorative margins leading to secondary caries [16–18] and tooth fracture [19,20]. Recurrent caries result from the acid production of biofilms in the oral cavity. Cariogenic bacteria can invade and colonize the interface between the resin and dentin via micro-gaps and debonding that result from degradation of the interface between the restorative material and hard tissues [21,22]. Resin composites are reported to accelerate growth and accumulate more biofilm/plaque than other restorative

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materials and, therefore, are more likely to produce acid (mostly lactic) [23,24]. Moreover, cyclic loading from mastication facilitates biofilm formation at the interface between the composite restoration and tooth [25], thereby exposing the dentin and enamel to localized acidic conditions. Synergy between the mechanical and acidic challenges could accelerate the mechanical aspects of degradation and increase the likelihood for restored tooth failure.

Cyclic loads resulting from mastication can activate fatigue crack growth in dentin, which initiates at flaws introduced via restorative practices [26,27]. Reviews on the fatigue and fracture properties of dentin have been presented [28,29]. Tubule orientation is important to fatigue crack growth in this tissue [7] and spatial variations in the microstructure are important to the crack growth resistance. Studies have shown that cracks initiate more easily in deep dentin, i.e. tissue closer to the pulp, [5] due to a reduction in potency of the extrinsic mechanisms of toughening (i.e. unbroken ligaments of tissue, collagen fibril bridging, etc.) [30]. Changes to the microstructure of dentin caused by the acidic secretions of biofilms could degrade the potency of these mechanisms that are crucial to the fatigue crack growth resistance. That would reduce the durability of the hard tissue foundation supporting the restoration.

The fatigue strength and fatigue crack growth resistance of dentin is significantly reduced by lactic acid exposure [31]. However, the mechanisms responsible for the reduction in fatigue crack growth resistance are not clear. Furthermore, contributions from the presence of resin adhesive filling and sealing the dentinal tubules on crack growth resistance, which is achieved by clinical treatment, is not understood. Therefore, in the present study we investigate the influence of a clinically relevant acidic condition and the importance of sealing the dentinal tubules with dental resin adhesive on fatigue crack growth within human dentin. The overall objective is to determine the reduction in fatigue crack growth resistance caused by acid exposure, and identify the changes in toughening mechanisms.

2. Materials and methods

Non-carious human 3rd molars were collected according to a protocol approved by the Institutional Review Board of the University of Maryland Baltimore County (Approval Y04DA23151). The teeth were obtained from young adults (17 ≤ donor age ≤ 31 years). Immediately after extraction the teeth were stored in Hanks Balanced Salt Solution (HBSS) with 0.2% sodium azide as an antimicrobial agent at 4 °C. Details regarding specimen preparation have been presented elsewhere [10, 32]. Briefly, compact Tension (CT) specimens were prepared from the coronal dentin (one only from each tooth) within one month of extraction using a numerical grinder (Chevalier Smart-H818II, Chevalier Machinery, Santa Fe Springs, CA, USA) and diamond impregnated slicing wheels (#320 mesh abrasives) under a water-based coolant bath. Primary sectioning was performed perpendicular to the longitudinal axis to obtain specimens from either the outer or middle regions of the crown as shown in Fig. 1(a). Each section was visually inspected and those with signs of visible damage or decay were discarded. Secondary sectioning was performed to obtain the body of the CT specimen of $4 \times 6 \times 2\ mm^3$. Precision holes were counter-bored to a depth equivalent to that of the back channel using a miniature milling machine (Dyna Myte, Model 2400) and required for the application of opening mode loads. The back channel and the notch were machined using the numerical slicer grinder and diamond wheel. The notch tip was sharpened using a razor blade and 1 μm diamond paste, resulting in a notch-tip radius of approximately 20 μm, which was validated using electron microscopy on representative specimens. Additional details on the protocols used to prepare dentin CT specimens have been described in more detail elsewhere [10,33]. A schematic diagram of the specimen geometry is shown in Fig. 1(b).

The specimens were subjected to cyclic loading within a lactic acid solution with pH = 5. The choice of conditions for acid exposure was based on many factors: *Streptococcus mutans* (*S. mutans*), one of the most prevalent bacterial species in oral biofilms [34], produces lactic acid after metabolization of fermentable carbohydrates [35]. *S. mutans* contains a cell-bound protein that adheres to the tooth surface and that is capable of tolerating pH levels as low as 4.5 [36–39]. An *S. mutans* colony increases in number at pH levels between 4.5 and 5.5 [38], whereas a pH below 4.5 causes instability and disruption [34,38]. Thus, a lactic acid solution with pH = 5 was considered a clinically relevant model and is consistent with the acid environment used in previous investigations [31,40]. The lactic acid solution was prepared by adding 7.45 ml of 0.1 M lactic acid to 950 ml of deionized water. A basic solution of NaOH was added to the buffer until the desired pH

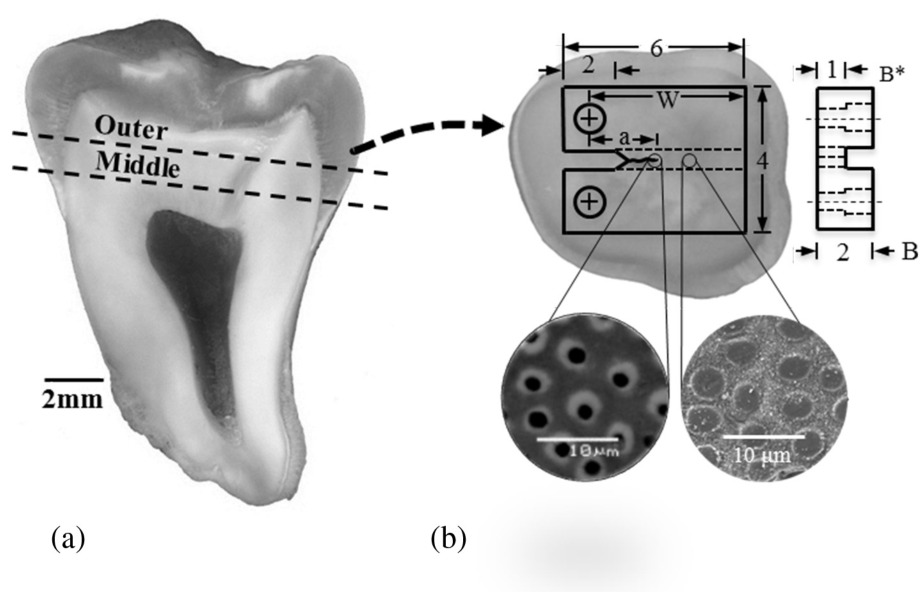

Fig. 1. Preparation of the compact tension (CT) specimens from sectioned human 3rd molars. (a) buccal-lingual section of a tooth showing the two coronal regions where the specimens were obtained (i.e. middle and outer regions); (b) view of a tooth section (perpendicular to its axis) and potential specimen. Note that in this orientation the crack will extend in-plane with the tubules, but perpendicular to their axes.