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The importance of microstructural variations on the fracture toughness of human dentin

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ABSTRACT

The crack growth resistance of human dentin was characterized as a function of relative distance from the DEJ and the corresponding microstructure. Compact tension specimens were prepared from the coronal dentin of caries-free 3rd molars. The specimens were sectioned from either the outer, middle or inner dentin. Stable crack extension was achieved under Mode I quasi-static loading, with the crack oriented in-plane with the tubules, and the crack growth resistance was characterized in terms of the initiation (K_o), growth (K_g) and plateau (K_p) toughness. A hybrid approach was also used to quantify the contribution of dominant mechanisms to the overall toughness. Results showed that human dentin exhibits increasing crack growth resistance with crack extension in all regions, and that the fracture toughness of inner dentin (2.2 ± 0.5 MPa$\cdot$m$^{0.5}$) was significantly lower than that of middle (2.7 ± 0.2 MPa$\cdot$m$^{0.5}$) and outer regions (3.4 ± 0.3 MPa$\cdot$m$^{0.5}$). Extrinsic toughening, composed mostly of crack bridging, was estimated to cause an average increase in the fracture energy of 26% in all three regions. Based on these findings, dental restorations extended into deep dentin are much more likely to cause tooth fracture due to the greater potential for introduction of flaws and decrease in fracture toughness with depth.

1. Introduction

Dentin is a mineralized tissue that occupies the majority of human teeth by both weight and volume. In the crown, dentin occupies the area between the pulp chamber and the Dentin–Enamel Junction (DEJ). One of the most distinct features of this tissue is the network of tubules (approx. 0.5–1.5 μm in diameter) that radiate outward from the pulp cavity to the DEJ [1]. Regarded as the dentin tubules, each is embodied by a collagen free, hyper-mineralized cuff of peritubular dentin. The interstitial space between the peritubular cuffs, i.e. intertubular dentin, consists of a collagen fibril matrix reinforced by nanoscale crystals of apatite [2,3]. Based on this complex composition and microstructure, dentin is often regarded as a hierarchical biological composite.

Microscopic evaluations show that there are substantial spatial variations in the number, diameter and orientation of the tubule lumens in the tooth crown. These characteristics vary with distance from the pulp, physiology and traumatic history of the tooth [4,5]. The tubule density and the tubule diameters are lowest at the DEJ and highest in deep dentin, nearest the pulp chamber [6]. Pashley et al. [4] estimated that tubules occupied approximately 22% of the evaluated cross-sectional area near the pulp, and only about 1% near the DEJ.

Consistent with the microstructural variations about the tooth, the mechanical properties of dentin have been reported to vary widely with location [7–12]. Most early studies concerning spatial variations in mechanical behavior have concentrated on the hardness and strength, showing that there is a decrease in these two properties with proximity to the pulp. A recent evaluation of the fatigue behavior of coronal dentin [13] distinguished that that there is also a significant reduction in the fatigue crack growth resistance with distance from the DEJ. This aspect of the mechanical behavior is highly relevant to restorative dentistry as flaws are introduced within dentin during the cutting of cavity preparations [14]. Damage caused by cutting may coalesce into well-defined cracks and undergo cyclic extension [15,16].

Fracture is one of the primary forms of restored tooth failure [17], and is often coined as the “cracked tooth syndrome” after [18]. This process is facilitated by cyclic crack growth and occurs when the crack length promotes a stress intensity that reaches the local

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fracture toughness of the tissue. Thus, spatial variations in the fracture toughness of dentin are important to the practice of restorative dentistry and treatment planning. An early study concerned with fracture of dentin [19] estimated a “work of fracture, W_f” using beams subjected to flexure loads, and found that W_f increases with proximity to the DEJ. But the aforementioned study did not evaluate the fracture toughness due to the absence of a well-defined crack. El Mowafy and Watts [20] were the first investigators to report the fracture toughness (K_{IC}) of human coronal dentin, finding an average of 3.08 $\mathrm{MPa \cdot m^{0.5}}$ for cracks aligned with the tubule orientation. It was later criticized that these experiments were performed on notched samples, rather than having sharp cracks, which can cause an overestimate of the toughness. Using sharp cracks with orientation perpendicular to the tubules, the fracture toughness was reported to be 1.8 $\mathrm{MPa \cdot m^{0.5}}$ [21]. Complimentary studies have analyzed the importance of lumen orientation [22,23] and patient age [24,25] on the fracture toughness, with values ranging from roughly 1 $\mathrm{MPa \cdot m^{0.5}}$ to over 2.5 $\mathrm{MPa \cdot m^{0.5}}$. The path of lowest crack growth resistance is perpendicular to the tubules and the fracture toughness of dentin decreases with patient age. However, no study has considered the importance of spatial variations in microstructure on the fracture toughness of dentin.

Most recent studies concerning the fracture behavior of dentin have reported that it exhibits a rising crack growth resistance with crack extension (i.e. rising R-curve) [22,24–26]. This is an important quality, and is exhibited by most materials with hierarchical microstructure [27,28]. A number of studies have evaluated the process of crack extension in dentin and the mechanisms contributing to toughening [26,29–32]. These studies have argued that dentin is primarily extrinsically toughened, and is achieved largely by crack bridging by uncracked ligaments of tissue [25,31,32]. But an alternate evaluation of fracture in dentin suggests that inelastic deformation arising from the organic content plays a major role in the fracture process, and that the toughness should be estimated using elastic–plastic fracture mechanics [33]. Owing to the spatial variations in microstructure of dentin, both theories could be correct, albeit applicable to different regions of the tooth.

In this investigation we evaluated the influence of microstructure on the fracture toughness of human dentin using a conventional fracture mechanics approach. A hybrid method was also adopted, involving a combination of experiments and numerical modeling, to estimate the relative contributions of extrinsic and intrinsic toughening mechanisms to the site-specific crack growth resistance. The primary objectives were to quantify the spatial variations in fracture toughness of coronal dentin and to develop a mechanistic understanding of the fracture process related to the microstructure.

2. Materials and methods

The experimental evaluation was conducted using the coronal dentin of caries-free unrestored 3rd molars, which were obtained from young dental patients in Maryland ($16 \leq \text{age} \leq 28$ years). The teeth were extracted according to protocols approved by the Institutional Review Board of the University of Maryland Baltimore County (approval Y04DA23151) and stored in Hank's Balanced Salt Solution (HBSS) with record of patient age and gender.

Within one month of extraction a Compact Tension (CT) specimen was wet sectioned from each tooth using a numerical controlled slicer/grinder (Chevalier, Model SMART-H81811, Taiwan), and diamond abrasive slicing wheels. The specimens were prepared to achieve crack growth in-plane with the dentin tubules (0° orientation) but perpendicular to their length as detailed in Fig. 1. Primary sectioning was performed to obtained specimens from the peripheral ($N = 4$), middle ($N = 4$) and inner ($N = 4$) regions of the teeth, which were located approximately 0.5 mm, 2 mm and 3.5 mm away from the DEJ, respectively (Fig. 1(a)); a total of 12 specimens were prepared for testing. Secondary sectioning was performed to establish the body of the specimen (Fig. 1(b)) and introduce other aspects of the detailed geometry. Briefly, a 1 mm wide channel was introduced on one side of each specimen to guide the direction of crack extension and precision holes were counter-bored using a miniature milling machine to enable application of opening mode (Mode I) loads. Then, a sharp notch was introduced using a razor blade and a diamond particle paste (1 μm diameter) to facilitate crack initiation. The protocols used in preparing the dentin CT specimens have been described in more detail elsewhere [34,35].

A fatigue pre-crack was introduced at the notch tip of the specimens to avoid effects of the notch radius [21]. Mode I cyclic loading of the CT specimens was performed for the crack initiation process using a universal testing system (Bose, Model ELF3200, Eden Prairie, MN, USA) with stress ratio (R) of 0.5, frequency of 5 Hz and using loads of $14 \leq P \leq 25$ Newtons. All loading was performed within an HBSS bath at room temperature (22 °C). After initiation, the crack was extended approximately $a \leq 0.5$ mm from the notch tip. Stable crack growth experiments were conducted using a dedicated universal testing system complemented with a microscopic imaging system [35]. Quasi-static loading was performed using

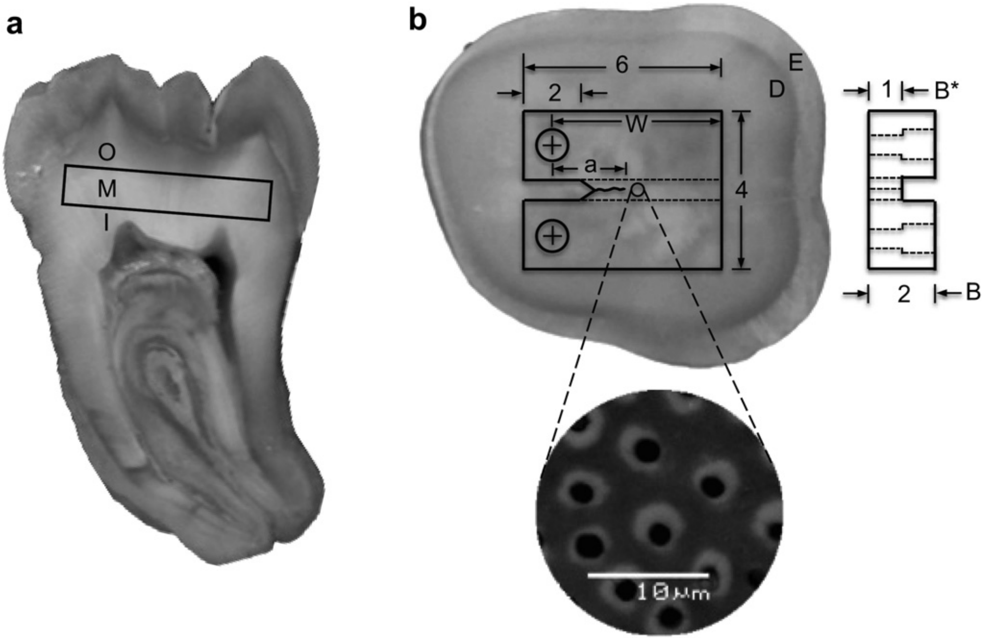

Fig. 1. Preparation of a compact tension (CT) specimen machined from a human 3rd molar. (a) section of a human 3rd molar indicating the three coronal regions where the specimens were obtained. I, M and O represent inner, middle and outer, respectively; (b) view of a sectioned tooth and potential specimen. The dentin (D) and enamel (E) are evident from the difference in gray scale. Note that the crack front is in-plane with the tubules, but perpendicular to their axes.