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## Transition behavior in fatigue of human dentin: Structure and anisotropy

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### Abstract

The influence of tubule orientation on the transition from fatigue to fatigue crack growth in human dentin was examined. Compact tension (CT) and rectangular beam specimens were prepared from the coronal dentin of molars with three unique tubule orientations (i.e.,  $0^{\circ}$ ,  $45^{\circ}$  and  $90^{\circ}$ ). The CT specimens (N = 25) were used to characterize fatigue crack initiation and steady-state cyclic extension, whereas the rectangular beams (N = 132) were subjected to 4-pt flexure and used in quantifying the stress-life fatigue response. The transition behavior was analyzed using both the Kitagawa–Takahashi and El Haddad approaches. Results showed that both the fatigue crack growth and stress-life responses were dependent on the tubule orientation. The average Paris Law exponent for crack growth perpendicular ( $90^{\circ}$ ) to the tubules ( $m = 13.3 \pm 1.1$ ) was significantly greater (p < 0.05) than that for crack growth oblique ( $45^{\circ}$ ) to the tubules ( $m = 11.5 \pm 1.87$ ). Similarly, the fatigue strength of dentin with  $90^{\circ}$  tubule orientation was significantly lower (p < 0.05) than that for the other two orientations, regardless of the range of cyclic stress. The apparent endurance strengths of specimens with  $0^{\circ}$  (44 MPa) and  $45^{\circ}$  (53 MPa) orientations were nearly twice that of the  $90^{\circ}$  (24 MPa) orientation. Based on these results, human dentin exhibits the largest degree of anisotropy within the stress-life regime and the transition from fatigue to fatigue crack growth occurs under the lowest cyclic stress range when the tubules are aligned with the cyclic normal stress ( $90^{\circ}$  orientation). ( $\mathbb{O}$  2007 Elsevier Ltd. All rights reserved.

Keywords: Anisotropy; Dentin; Fatigue; Fracture; Tubules

#### 1. Introduction

Despite continued improvements in restorative dentistry, the failure of restored teeth has remained commonplace. The majority of failures are attributed to secondary caries, bulk fracture and fracture of the tooth [1-4]. Tooth fractures are often considered a problem of the past, but cracked teeth and tooth fractures are still prevalent today, particularly in seniors [5] and in teeth receiving endodontic treatment [6].

Tooth fractures typically involve failure of the dentin and/ or enamel. Dentin comprises the bulk of the human tooth and is a hard tissue that is approximately 45% mineral, 35% organic matter, and 20% water by volume [7]. On a microscopic scale dentin is traversed by tubules, which exist as open channels  $(1-2\mu m \text{ internal diameter})$  and extend radially from the pulp throughout the dentin towards the dentin-enamel junction. A highly mineralized cylindrical cuff of tissue encloses each tubule lumen and is regarded as the peritubular dentin. Intertubular dentin occupies the space between the cuffs and is comprised of a collagen fibril matrix that is bound by apatite crystallites. The collagen fibrils are distributed in planes that are essentially perpendicular to the tubule lumens [7]. According to this arrangement, the mechanical behavior of dentin would be expected to be a function of the tubule orientation.

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When examined on a microscopic scale, both the hardness and elastic modulus of peritubular dentin are reportedly greater than those of the intertubular dentin [8]. Yet, on a macroscopic scale there is only a small degree of elastic anisotropy with the largest elastic modulus evident perpendicular to the tubules [9–11]. The tensile strength of dentin is largest in the direction perpendicular to the tubules [12–16] and the shear strength is a function of tubule orientation as well [17]. Though the tubules and their surrounding mineralized cuffs are the most obvious structural components, differences in the mechanical properties of dentin are now believed related to the orientation of the collagen fibrils rather than the tubules [18].

Tubule orientation is also important in fracture of dentin. The work of fracture is lowest for crack extension perpendicular to the dentin tubules [19]. Iwamoto and Ruse [20] reported that the fracture toughness of human dentin was approximately 1.13 and  $2.0 \text{ MPa m}^{0.5}$  for fracture perpendicular and parallel to the tubules, respectively. The variation in resistance to fracture with tubule orientation has been attributed to differences in the contribution of toughening mechanisms [21]. Microscopic observations suggest that toughening results predominantly from a combination of collagen fibrils and unbroken ligaments of tissue that "bridge" the crack and reduce the effective stress intensity at the crack tip through closure forces. Cracks extending parallel to the tubules (i.e., transverse to the collagen fibrils) experience the highest degree of bridging and gives rise to the larger fracture toughness for this orientation.

Fracture is often preceded by the initiation of a welldefined crack from incipient damage under cyclic loading (i.e., fatigue) [22]. Consistent with the fracture behavior, the fatigue strength of dentin is lowest when cyclic stresses are oriented along the tubules, which results in coalescence of damage and development of a crack perpendicular to the tubules [23]. However, only minor differences have been identified in the fatigue crack growth properties of dentin as a function of tubule orientation [24]. Collectively these studies suggest that the importance of tubule orientation in fatigue changes with the degree of damage and/or flaw size. The transition from stress-life fatigue to cyclic crack growth in human dentin was recently examined using the Kitagawa–Takahashi diagram [25]. While that evaluation presented a new means of assessing the fatigue responses of mineralized tissues, it did not address anisotropy in the transition behavior. Therefore, the objective of this study was to examine the effects of tubule orientation on the transition from stress-life fatigue to cyclic crack growth in human dentin.

#### 2. Materials and methods

Extracted second and third non-carious molars were acquired from dental practices in Maryland according to an approved protocol issued by the Institutional Review Board of the University of Maryland. The teeth were classified in terms of the patient's age and gender. The molars were maintained at 2 °C in a bath of Hanks balanced salt solution (HBSS) until being selected for the preparation of specimens. Each tooth was molded in a polymer resin and then sectioned using a numerical controlled slicer/ grinder and diamond abrasive slicing wheels under continuous delivery of water-based coolant. Two different specimen types were prepared.

Fatigue crack growth properties were determined using compact tension (CT) specimens (Fig. 1(a)) that were prepared from the coronal dentin of selected molars with tubule orientations ( $\theta_1$ ) of 0° (N = 8), 45° (N = 8) and 90° (N = 9) as described in Fig. 1(b). A total of 25 specimens were prepared from the molars of 25 different patients with 17  $\leq$  age  $\leq$  35. Details concerning specimen preparation have been presented elsewhere [26].

The CT specimens were subjected to fatigue loads using a universal testing system.<sup>1</sup> Cyclic loading was performed at 5 Hz under load control actuation with the specimens immersed in an HBSS bath at room temperature. Crack initiation was achieved by cyclic loading with a stress ratio ( $R = \sigma_{\min}/\sigma_{\max}$ ) of 0.5 [27] and typically required between 300 and 500 kcycles of loading. Following initiation, the crack was extended an appreciable length beyond the notch ( $\approx 0.5$  mm) using R = 0.1 and a maximum cyclic load between 8 and 14 N. Thereafter, the change in crack length ( $\Delta a$ ) was documented after specific increments of fatigue ( $\Delta N$ ) until the specimen fractured. Inspection of the notch tip and crack length measurements were performed using an optical microscope ( $100 \times$ ) with scaled reticule.

The incremental crack growth rate (da/dN) was examined in terms of the stress intensity range over the entire growth history. The steady-state (Region II) response was identified and then quantified using the Paris Law [28] according to

$$\left. \frac{\mathrm{d}a}{\mathrm{d}N} \right|_{\theta_1} = C(\Delta K)^m,\tag{1}$$

where  $\Delta K$  is the stress intensity range, and da and dN represent the incremental crack extension ( $\Delta a$ ) and number of cycles ( $\Delta N$ ), respectively. The quantities *C* and *m* are the fatigue crack growth coefficient and exponent. Owing to the size of human teeth, the CT specimen geometry did not conform to standardized dimensions [29]. Therefore, the stress intensity range was estimated in terms of the opening load range ( $\Delta P = P_{\text{max}} = P_{\text{min}}$ ) according to [26]

$$\Delta K = \frac{\Delta P}{B^* \sqrt{W}} \left(\frac{B^* + 1}{B + 1}\right)^{1/2} (0.131 + 0.320\alpha + 0.211\alpha^2) \text{ (MPa m}^{0.5}), \qquad (2)$$

where W,  $B^*$  and B are dimensions of the specimen as defined in Fig. 1(a). The quantity  $\alpha$  is a numerical value equal to the ratio of the average crack length over the growth increment  $((a_{i+1} + a_i)/2)$  to W. The crack growth rate (da/dN) and Paris Law parameters were defined with respect to the out-of-plane tubule orientation ( $\theta_1$  in Fig. 1(b)) and a comparison of these parameters was conducted using a one-way ANOVA to identify significant differences (p < 0.05). The apparent stress intensity threshold ( $\Delta K_{\text{th}}$ ) was also estimated for  $da/dN \le 1 \times 10^{-7}$  mm/cycle. Experimental results for specimens with 90° tubule orientation were previously reported and used in examining the importance of patient age on fatigue crack growth in dentin [26]. Results for the 0° and 45° orientations have not been previously reported for human dentin.

Rectangular beams were also prepared from the coronal dentin of selected molars with tubule orientations ( $\theta$ ) of 0°, 45° and 90°. The orientation ( $\theta$ ) distinguishes the angle between the plane of maximum normal stress resulting from loading and the primary axis defined by the tubule lumens (Fig. 2(a)). The convention results in a consistent definition of tubule orientation in both the CT and rectangular beam specimens. Note that the tubules in beams with 0° orientation were in the plane of fracture and parallel to the expected direction of crack extension, whereas in the 0° CT specimens the tubules were parallel to the plane of fracture and perpendicular to the direction of crack extension. This difference was necessary due to the limited tissue available and consequent difficulties in

<sup>&</sup>lt;sup>1</sup>EnduraTEC Model ELF 3200, Minnetonka, MN, USA.

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