In vitro fracture toughness of human dentin

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Abstract: The *in vitro* fracture toughness of human dentin has been reported to be of the order of 3 MPa$\sqrt{m}$. This result, however, is based on a single study for a single orientation, and furthermore involves notched, rather than fatigue precracked, test samples. The present study seeks to obtain an improved, lower-bound, value of the toughness, and to show that previously reported values may be erroneously high because of the absence of a sharp crack as the stress concentrator. Specifically, the average measured critical stress intensity, K_c, for the onset of unstable fracture along an orientation perpendicular to the long axis of the tubules in dentin is found to be 1.8 MPa$\sqrt{m}$ in simulated body fluid (Hanks' balanced salt solution), when tested in a three-point bending specimen containing a (nominally) atomically sharp precrack generated during prior fatigue cycling. This is to be compared with a value of 2.7 MPa$\sqrt{m}$ measured under identical experimental conditions except that the bend specimen contained a sharp machined notch (of root radius 30–50 μm). The effect of acuity of the precrack on the fracture toughness of human dentin is discussed in the context of these data. © 2003 Wiley Periodicals, Inc. J Biomed Mater Res 66A: 1–9, 2003

Key words: dentin; fracture toughness; fatigue; fractography; notch acuity

INTRODUCTION

Dentin is the most abundant mineralized tissue in the human tooth, physically located between the exterior enamel and the interior pulp (Fig. 1). However, there is only limited understanding of its structural performance, despite the fact that an accurate assessment of its mechanical properties is crucial for the prediction of how such factors as caries, sclerosis and aging, not to mention restorative processes, can degrade the strength of the tooth. Although more than five decades of research has been conducted for the purpose of evaluating these properties,[1–11] there is unfortunately little consistency in the available results.

Resistance to fracture is a critically important issue with teeth. For example, exposed root surfaces often exhibit noncarious notches in the dentin just below the enamel–cementum junction. The etiology for such lesions is believed to involve a combination of erosion, abrasion, and abfraction.[12] Whereas erosion is often the result of exposure to acids, abrasion is caused by the use of inappropriate dentifrice and abfraction by mechanical stresses induced by brushing and chewing. These notches serve as effective stress raisers and are often the sites of failure of the tooth because of fracture. Whereas cusp fractures are common in posterior teeth, the anterior teeth are more susceptible to fracture in the gingiva, severing the crown of the tooth. Although such fractures have not been studied extensively, it is generally believed that they are catastrophic events induced by occlusal stresses.

In light of this, a stress-based fracture mechanics approach can provide a sound basis for the prediction of failure in human teeth. Under linear elastic conditions, an essential feature of this approach is that unstable fracture will occur when the stress intensity developed ahead of the tip of a pre-existing flaw exceeds the fracture toughness, K_c, of the material, i.e.,

$$K = Y\sigma_{app}(\pi a)^{1/2} = K_c \tag{1}$$

where σ_{app} is the applied service stress, a is the crack length, and Y is a function dependent on the geometry and crack size and shape.[13]

Despite the usefulness of this approach, however, there are limited quantitative data on the toughness properties of human teeth; indeed, there seems to have only been few studies to date with respect to the

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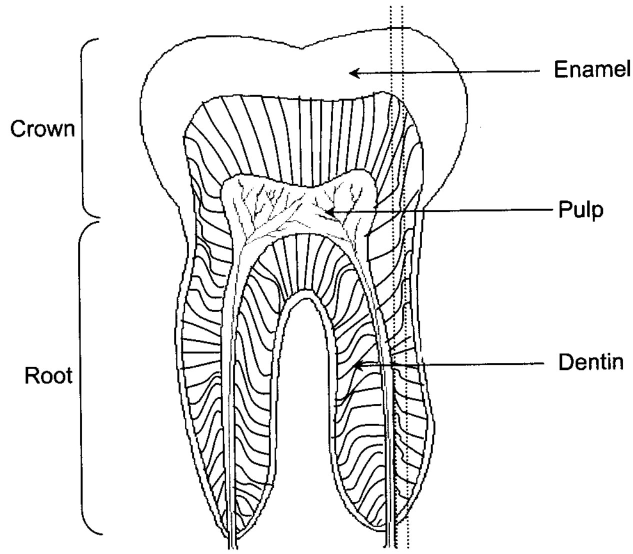

Figure 1. Schematic illustrating a typical human tooth with the section (shown by dotted lines) made for the purpose of specimen preparation. Note the tubules running from the dentin–enamel junction to the pulp of the tooth.

fracture toughness of dentin. The first of these studies, by Rasmussen et al.,[10,11] uses a "work of fracture" (defined as the work per unit area to generate new crack surface) to quantify the toughness. Unfortunately, because this measurement is geometry- and sample-size dependent, their results cannot be compared quantitatively with any subsequent measurements. These authors, however, do report an influence of orientation on the toughness of dentin in that their measured work of fracture was lower for fracture perpendicular to the dentinal tubular direction compared with fracture in the plane of the tubules. Such behavior is unlike bone,[14] where the collagen fibrils are organized in an alternating lamellar structure; in dentin, conversely, the mineralized collagen fibrils are randomly oriented in a plane perpendicular to the tubules. Therefore, as cracks propagating parallel to the tubules would have to cut across the fibrils, it is conceivable that collagen fiber bridging might be a toughening mechanism in this direction. Indeed, the measurement of work of fracture, by Rasmussen et al.,[10] does seem to indicate that crack propagation perpendicular to the tubules is more energetically favorable, possibly because there can be no fiber bridging in that direction. However, it is difficult to find conclusive proof for such anisotropy because the scatter in their results was excessive.

A subsequent study, by el Mowafy and Watts,[6] did utilize fracture mechanics based measurements, specifically using compact-tension specimens to measure an intrinsic fracture toughness in dentin. Using an orientation parallel to the long axis of the tubules, these authors report a K_c value of 3.08 MPa$\sqrt{m}$ (standard deviation 0.33 MPa$\sqrt{m}$) for dentin, which was found to remain constant over the temperature range 0°–60°C. However, their experiments were performed on notched, rather than fatigue-precracked, samples, and it is known from previous studies[15] on brittle bio-implant materials, specifically pyrolytic carbon for prosthetic heart valves, that the absence of a sharp precrack can severely overestimate fracture toughness values. Because recent studies at Berkeley[16] have shown that fatigue cracks can be grown subcritically under cyclic loading in dentin, it was the objective of the present work to perform proper fracture toughness tests using appropriately fatigue precracked samples to determine an accurate measure of the fracture toughness of human dentin *in vitro* and to further assess the influence of notch acuity on such results. Although the anisotropy of toughness values in dentin is still somewhat unproven, measurements were made in an orientation perpendicular to the tubules in an attempt to determine a lower-bound value.

MATERIALS AND METHODS

Recently extracted human molars were used in the present study. Each tooth was sterilized using gamma radiation after extraction.[17] Sections (~1.5–2.0 mm thick) were prepared from the central portion of the crown and the root vertically through the tooth (Fig. 1) such that the plane of the fracture was nominally perpendicular to the long axis of the tubules in dentin. In actuality, it is almost impossible *a priori* to align this fracture plane precisely with the tubule axes because, with the exception of the root, the tubules in dentin do not run a straight course from the enamel to the pulp; rather, from the cervical margin through the crown, the tubules have a complex, S-shaped curvature.[18] Consequently, the orientation of the crack plane was determined precisely from examination of the fracture surfaces. In the present study, all toughness data were collected on samples in which the fracture plane was nominally perpendicular to the average tubule direction.

Beams of dentin measuring approximately 0.90 × 0.90 × 10.0 mm were then obtained from these sections by wet polishing up to a 600-grit finish. These beams were then stored in Hanks' balanced salt solution (HBSS) at ambient temperature for no longer than 1 week until testing. Although mineral can be leached into solution when storing dentin in deionized water, which can result in changes in elastic properties with time, no such changes could be detected after short time storage in HBSS. However, the precise effects of storage solution on the fracture properties *per se* have not been investigated, and further study is warranted.

A micrograph of the typical structure of dentin is shown in Fig. 2; a brief description of this microstructure is given below. *In vitro* first yield (σ_y) and maximum flexural (σ_F) strength levels were measured in HBSS in bending to be $\sigma_y \sim 75$ MPa and $\sigma_F \sim 160$ MPa, respectively.

Fracture toughness testing was performed in general accordance with the ASTM standard E-399 for plane-strain

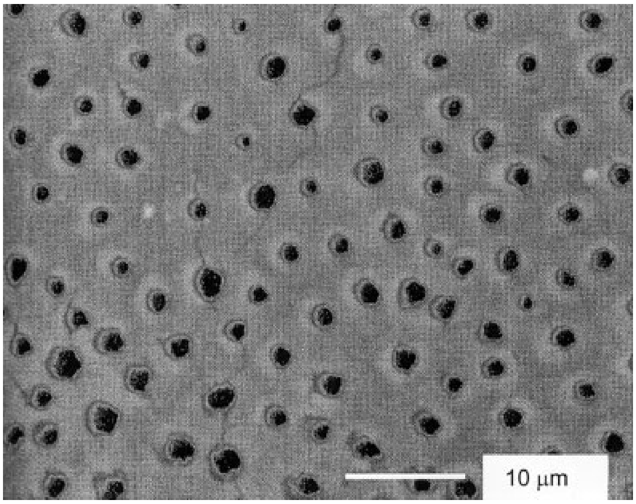

Figure 2. Micrograph illustrating the typical microstructure of human dentin. The most striking feature is the pseudo-periodically placed 1–2 μm diametered tubules.

fracture toughness,[19] with the exception that the full thickness for a state of plane strain could not be achieved with the 0.9-mm-thick dentin samples. According to ASTM E-399, a state of plane strain is achieved when the sample thickness is >2.5 $(K_c/\sigma_y)^2$, i.e., significantly larger than the plastic or damage zone size of $r_y \sim 1/2\pi\ (K_c/\sigma_y)^2$. For dentin, this would require sample thicknesses greater than approximately 1.4 mm to yield a plane-strain K_c value. However, because this criterion is generally quite conservative and the damage zone was well contained within the specimen boundaries, it is believed that the toughness values measured with the current test specimens would be very close to this lower bound.

Tests were conducted using the three-point bending geometry [Fig. 3(a)] with a span between the lower two loading points equal to 5–5.5 times the width of the beam (S = 5–5.5 W). Specimens were machined with the anticipation that the fracture occurred in the plane perpendicular to the long axis of the dentinal tubules; this was confirmed by postfailure analysis. Two sets of experiments were performed. In the first set, a sharp notch was carefully introduced into the top surface of the bend specimen using a razor blade [Fig. 3(b,d)]. Typical notch depths were of the order of 50–125 μm, with a root radius, ρ, of approximately 30–50 μm. In the second set, in accordance with ASTM E-399, a precrack was grown out of the notch by cycling in fatigue [Fig. 3(c and e)]; this was achieved at a load ratio (ratio of minimum to maximum loads) of R = 0.1 and loading frequency of 2 Hz, with a final maximum stress intensity of $K_{max} \sim 1$ MPa$\sqrt{}$m, i.e., well below the estimated fracture toughness of dentin. The final precrack length (notch plus precrack) was generally of the order of 100–200 μm, with a presumed atomically sharp crack tip.

In vitro fracture toughness testing of both types of specimens was conducted with an ELF® 3200 series voice-coil mechanical testing machine (EnduraTEC Inc., Minnetonka, MN). Samples were loaded to failure under displacement control in HBSS at ambient temperature (to simulate physiological conditions) at a cross-head displacement rate of 0.01 mm/s. A record of the applied loads and the corresponding displacements was simultaneously monitored during the test and analyzed for determining the fracture toughness. The displacements were obtained continuously from the stroke transducer on the testing frame. Three separate specimens were tested for each type of experiment.

Linear-elastic stress intensities, K, were computed from handbook solutions, specifically from the ASTM standard E-399 for plane-strain fracture toughness.[19] For the three-point bending specimen used:

$$K = \left(\frac{PS}{BW^{3/2}}\right) f(a/W), \tag{2a}$$

where P is the applied load, S is the distance between the outer loading pins, a is the crack length, B and W are, respectively, the specimen thickness and width, and $f(a/W)$ is dimensionless function of a/W given by:

$$f(a/W) = \frac{3(a/W)^{1/2}\{1.99 - (a/W)[1 - (a/W)] \times [2.15 - 3.93(a/W) + 2.7(a/W)^2]\}}{2[1 + 2(a/W)][1 - (a/W)]^{3/2}} \tag{2b}$$

Postfailure observations of the fracture surfaces were made both optically and using a scanning electron microscope, operating in the secondary electron mode.

RESULTS

Microstructure of dentin

Human dentin is a hydrated composite material composed of nanocrystalline carbonated apatite mineral (~45% by volume), type-I collagen fibrils (~30% by volume), and fluid (~25% by volume), with the mineral being distributed in the form of 5-nm-thick crystallites in a scaffold created by the collagen fibrils (typically 50–100 nm diameter). The distinctive feature of the "microstructure" of the dentin is a distribution of 1–2 μm diameter cylindrical tubules that run from the dentin–enamel junction to the soft, interior pulp (Figs. 1 and 2). These dentinal tubules are the paths of the odontoblast cells during tooth formation and are surrounded by a collar of highly mineralized peritubular dentin (~1 μm thick) and are embedded within a matrix of mineralized collagen called intertubular dentin. The mineralized collagen fibrils, which are between 50–100 nm in diameter, form a planar felt-like structure oriented perpendicular to the tubules.[20] The tubules can be considered to be randomly displaced about a periodic lattice,[21] but with a distribution that depends on location within the tooth.

Fracture toughness evaluation

Results of the fracture toughness testing, in the form of typical test records of the load-versus-displacement

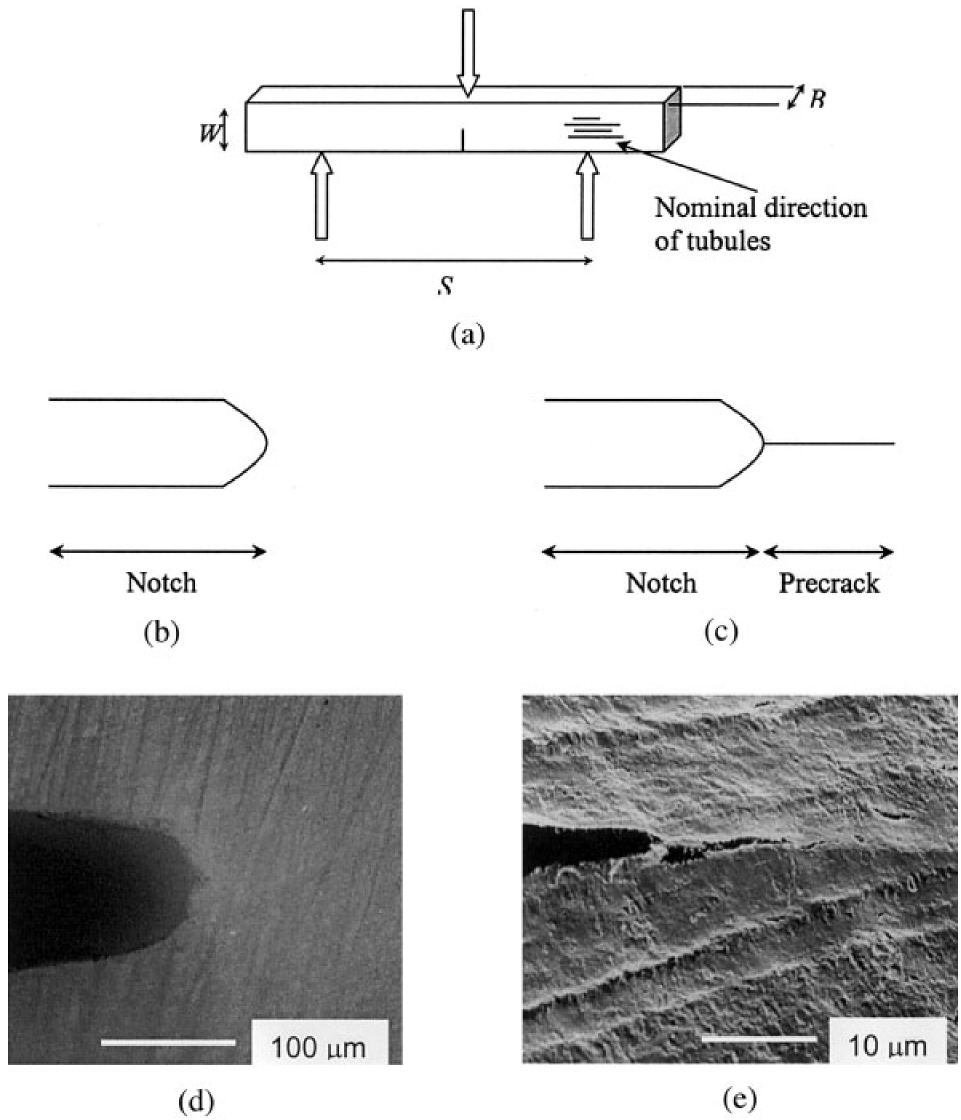

Figure 3. (a) Three-point bending configuration used for the measurement of the fracture toughness, showing schematic illustrations of (b) the rounded-notch and (c) the sharp-crack configurations. (d) Optical micrograph of the notch tip for a notch used for notch toughness tests. (e) Scanning electron micrograph of the tip of a typical precrack used for the crack-toughness tests. The nominal orientation of the tubules in relation to the specimen geometry is shown in (a).

curves, are shown in Figure 4 for the notched and fatigue precracked samples. Fracture toughness, K_c, values were determined from the measured critical loads, P_Q, for the onset of unstable fracture using Eq. (2). These calculations yielded average values of 2.72 MPa$\sqrt{m}$ for the (apparent) fracture toughness of the notched samples and K_c = 1.79 MPa$\sqrt{m}$ for the (real) fracture toughness of the fatigue precracked samples of human dentin; the individual values obtained are shown in Table I. Clearly, the absence of a sharp stress concentrator, specifically a fatigue precrack, acts to elevate the measured toughness of this material. Based on these results, it would seem that an appropriate value for the toughness of human dentin, in the orientation perpendicular to the tubules, is K_c = 1.8 MPa$\sqrt{m}$.

Fractography

Postfailure fractography was performed using a scanning electron microscope (secondary electron mode) for both types of test specimens. As shown in Figures 5–7, fracture did indeed occur in the plane perpendicular to the long axis of the dentinal tubules. The distinction in the two different types of initial stress concentrators used can be clearly seen in Figure 5, the typical fractography of a notched sample being shown in Figure 5(a) and for the fatigue-cracked samples in Figure 5(b). These can be compared with the fracture morphology produced by cyclic fatigue in dentin in Figure 6, and by overload failure in Figure 7.

The path followed by the fatigue crack is relatively

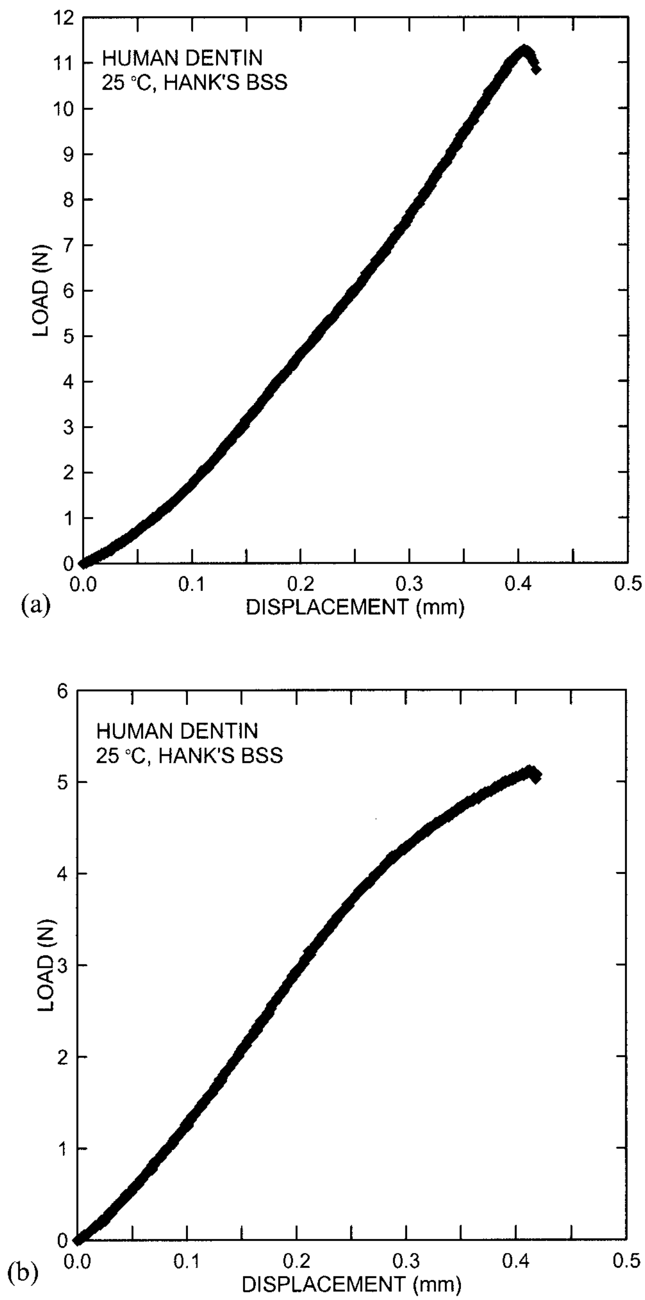

Figure 4. Representative load/displacement curves obtained for the measurement of the fracture toughness ahead of (a) a machined notch and (b) a fatigue precrack.

free of tortuosity with no evidence of any severe influence of the tubules on the crack front (generally evidenced by extensive crack front "bowing" around the tubules or by cavitation). However, there does seem to be some evidence, albeit not totally conclusive, of pullout behavior of the peritubular dentin cuff surrounding the tubules (indicated by arrows in Figs. 6 and 7), suggesting the possibility of some degree of extrinsic toughening. No evidence could be found for the presence of fatigue striations, however, in contrast to many metallic and polymeric materials. Furthermore, unlike such materials in which there are generally significant differences between the fracture modes, and hence the fractography, of fatigue and

TABLE I
Measured *In Vitro* Fracture Toughness Values of Human Dentin

Precrack Type	Root Radius, ρ (μm)	Fracture Toughness (MPa√m)
Notch	39	2.65
Notch	46	2.88
Notch	38	2.63
Notch[a]	165	3.08
Fatigue crack	→0	1.73
Fatigue crack	→0	1.85
Fatigue crack	→0	1.79

[a]Average data after el Mowafy and Watts.[6]

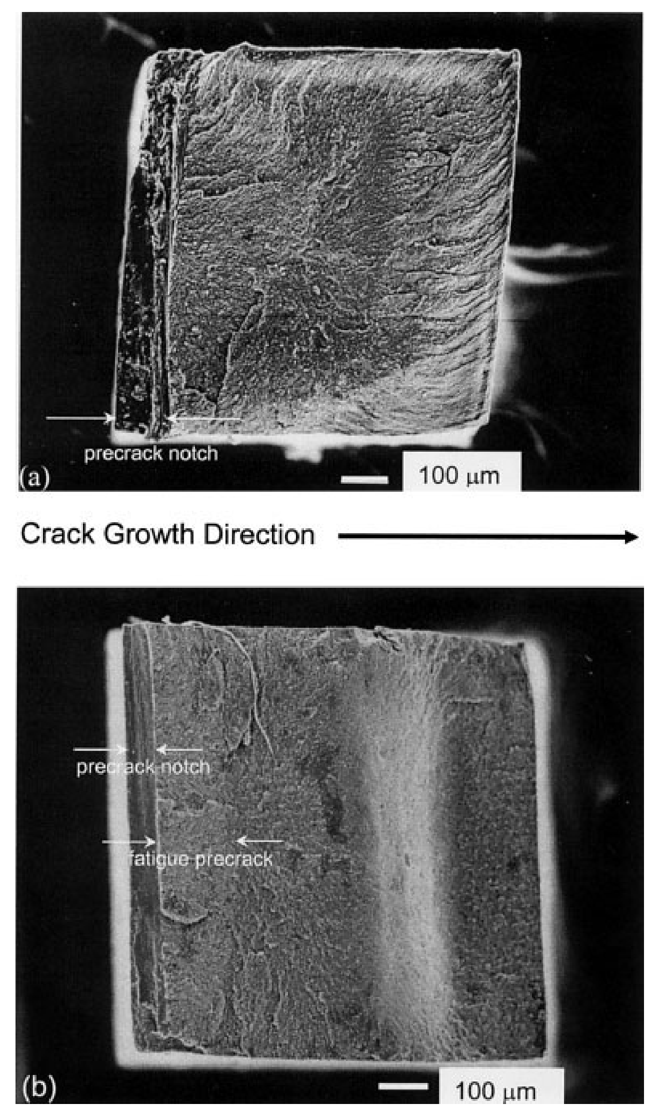

Figure 5. Scanning electron micrographs showing an overview of the fracture surfaces obtained by fracture ahead of (a) a machined notch and (b) a fatigue precrack. The nominal direction of crack growth is also indicated.

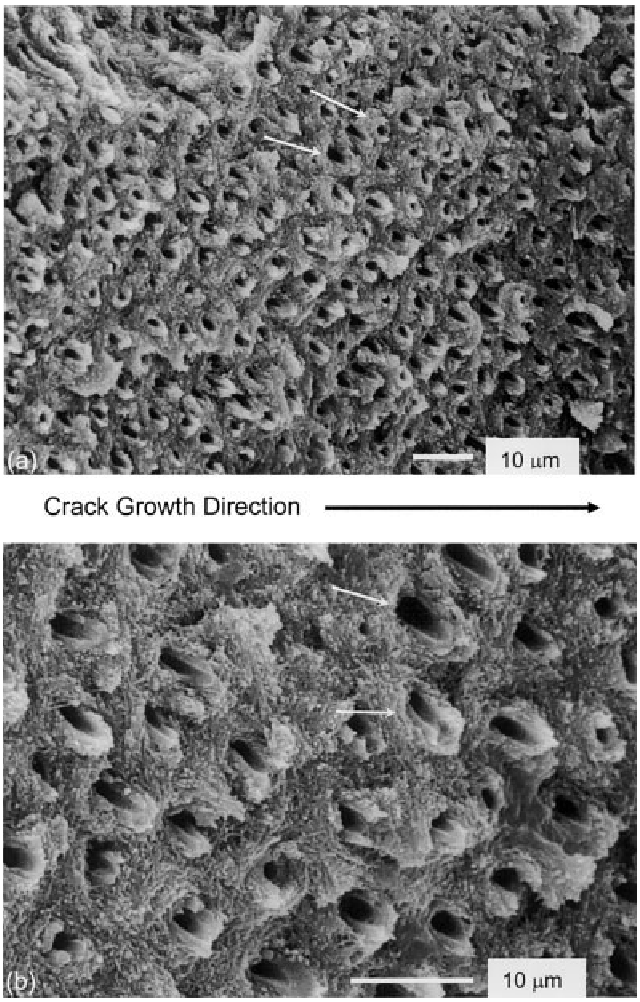

Figure 6. Low (a) and high (b) magnification scanning electron micrographs of the cyclic fatigue region of the precrack used for the true K_c determination. There is some evidence of pullout of the peritubular dentin cuffs (indicated by white arrows). The nominal direction of crack growth is also indicated.

overload fracture,[22] there was little difference in dentin between the morphology of fracture surfaces obtained during cyclic fatigue-crack growth and final overload (fast) fracture (Figs. 6 and 7). This is typical of most brittle solids, for example, ceramic materials such as silicon nitride and pyrolytic carbon,[15,22] in which the mechanisms of crack advance *ahead* of the crack tip are essentially identical under alternating and single-cycle (overload) loading. In such brittle materials, the mechanistic effect of cyclic fatigue loading is invariably seen *behind* the crack tip in the form of a progressive degradation in the operative crack-tip shielding (toughening) mechanisms.[23] Whether these mechanisms are relevant to the fatigue of dentin is currently under study.

In this regard, it is interesting to speculate whether the tubules in dentin serve as effective stress raisers and hence as crack-initiation sites. Although this issue has never been addressed adequately, support for this possibility is provided by the common occurrence of microcracks in the peritubular dentin surrounding the tubules (Fig. 8) on the fracture surfaces obtained at the higher stresses associated with final overload failure.

DISCUSSION

The present work seems to be only the second study in the archival literature, after Ref.,[6] to quantify the K_c fracture toughness properties of human dentin. This is somewhat surprising in view of the significant work reported on the mechanical properties of other biological material systems, such as on the fracture toughness of bone.[24–26] Nevertheless, the currently mea-

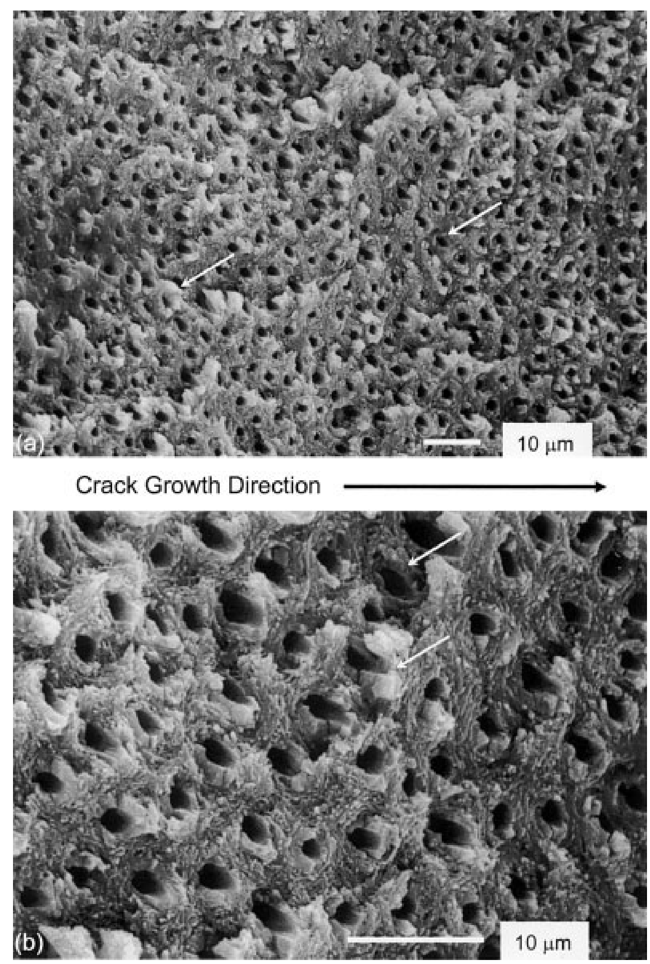

Figure 7. Low (a) and high (b) magnification scanning electron micrographs of the overload (fast) fracture region of a specimen used for true K_c determination. It is evident that although this fracture surface is slightly more "rough" at a macroscopic size scale, it is essentially identical to that obtained by cyclic fatigue at microscopic size scales. There is again some evidence of pullout of the peritubular dentin cuffs (indicated by white arrows). The nominal direction of crack growth is also indicated.

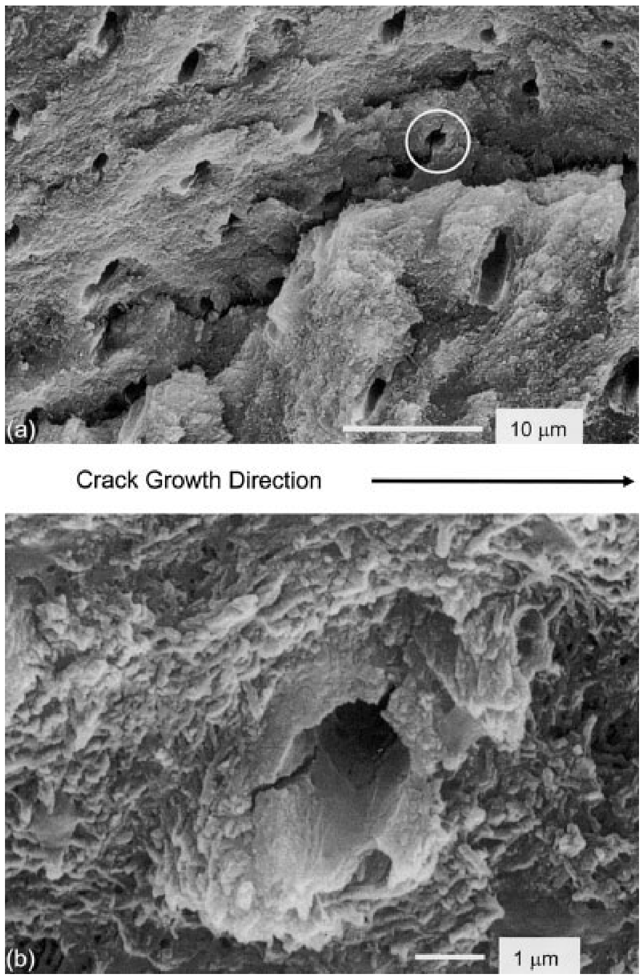

Figure 8. (a) A typical scanning electron micrograph of the overload fracture region is shown, along with (b) a magnified image of the indicated region. The crack formation in the peritubular dentin is apparent. The nominal direction of crack growth is also indicated.

sured fracture toughness of $K_c = 1.79$ ($\pm$0.06) MPa$\sqrt{\text{m}}$ for dentin (measured in a fatigue-precracked sample with fracture occurring perpendicular to the long axis of the tubules) represents a figure that is ~34% lower than the value of 2.72 MPa$\sqrt{\text{m}}$ obtained for dentin using a sharp notched sample. Based on our current results, we have clearly shown that the fracture toughness of dentin can be elevated by using a less than atomically sharp stress concentrator, that is, the apparent toughness was increased by >50% using a sharp machined notch (with a root radius of 30–50 μm). It is therefore our contention that a value of 1.79 MPa$\sqrt{\text{m}}$ would be a reasonable estimate of the actual fracture toughness of dentin in this orientation. It should be noted here that the earlier work of el Mowafy and Watts,[6] in which an average value of 3.08 MPa$\sqrt{\text{m}}$ was reported for an orientation parallel to the tubules, seems to represent an overestimate of the K_c toughness because these authors used a relatively blunt notch (with a root radius, ρ ~165 μm) instead of a sharp fatigue precrack in their fracture toughness test samples. There is a possibility that the difference in K_c values may additionally be associated with orientation, because Rasmussen et al.[10,11] have suggested that tubule orientation and distribution can affect the fracture resistance in dentin. Indeed, these authors measured a ~50% lower work of fracture in the plane of the collagen fibrils (perpendicular to the tubules), compared with the orthogonal direction cutting the fibrils, suggesting that mineralized collagen fibrils and the tubules might have a role in any toughening mechanisms involved; however, as noted above, it is difficult to draw firm conclusions because their toughness measurements were highly size dependent and subject to excessive scatter (specifically, with average measure errors of 12–15% and root mean square deviations of 33–60%).

With respect to the critical issue of the type of stress concentrator used in measuring the toughness, similar observations have been made for pyrolytic carbon, in which the early fracture toughness measurements using machined notches by More et al.[27] (2.79 $\pm$ 0.23 MPa$\sqrt{\text{m}}$) were >50% higher than the values measured ahead of sharp fatigue precracks by Dauskardt et al.[15] (1.29–1.84 MPa$\sqrt{\text{m}}$). Indeed, it is well known that the toughness of both ductile and brittle materials can vary substantially with the root radius of the notch used[28,29]; specifically, early studies suggested that the apparent toughness should scale with the square root of the notch radius, that is, $K_c \propto \rho^{1/2}$. This is evident from the present work in which the apparent toughness of dentin is plotted as a function of the square root of the notch radius (Fig. 9). The data from Ref.[6] are also included for the purpose of comparison. Clearly, the fracture toughness measured ahead of a sharp fatigue precrack not only provides a lower-bound value but also represents a more appropriate quantification of the intrinsic fracture resistance of the material.

Finally, it is of interest to note how this corrected fracture toughness value for dentin of K_c = 1.79 MPa$\sqrt{\text{m}}$ compares with the toughness properties of other biological and bio-implant material systems associated with dentistry. For example, a fracture toughness of 0.23–6.56 MPa$\sqrt{\text{m}}$ has been reported for cortical bone,[30] which is similar in composition to dentin in living tissue, whereas the toughness of human dental enamel is quoted as 0.7–1.27 MPa$\sqrt{\text{m}}$,[31] although the latter values were obtained using micro-indentation techniques which often tend to be approximate. By comparison, most dental cements have toughnesses in the range 0.1–0.5 MPa$\sqrt{\text{m}}$, amalgams in the range 0.1–1.6 MPa$\sqrt{\text{m}}$, and dental composites in the range 0.6–2.0 MPa$\sqrt{\text{m}}$.[32]

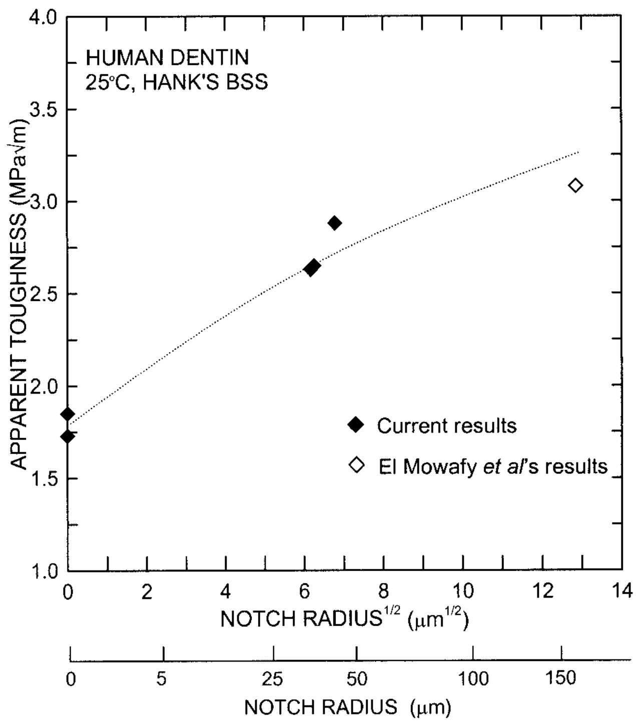

Figure 9. The apparent fracture toughness values for dentin are plotted as a function of the square root of the notch root radius. The corresponding notch radii are also shown. Results are shown for the present investigation along with the single data point of el Mowafy and Watts[6] for comparison.

CONCLUSIONS

Based on an experimental study of the intrinsic fracture toughness of human dentin *in vitro*, the following conclusions can be made:

1. The fracture toughness of dentin has been accurately measured in HBSS to be $K_c = 1.79$ (± 0.06) MPa$\sqrt{m}$ for fracture nominally perpendicular to the tubule orientation.
2. This effect of notch acuity on the measured fracture toughness was demonstrated for dentin by measurements of an apparent increase in fracture toughness value with increasing notch root radius. Indeed, the apparent toughness was shown to be directly proportional to the square root of the notch radius, and was increased by >50% by using a sharp machined notch (with a 30–50 μm root radius) rather than a fatigue precrack.
3. This value (1.79 MPa$\sqrt{m}$) is ~42% lower than the only other fracture toughness value reported for dentin in the literature, specifically $K_c = 3.08$ MPa$\sqrt{m}$ by el Mowafy and Watts.[6] The discrepancy between the current and previous K_c results is attributed to a difference in the orientation investigated and primarily to the effect of the notch acuity on the toughness value. The current study was performed using atomically sharp fatigue precracks in accordance with ASTM standards, whereas the prior result was measured on samples containing machined notches.
4. It is concluded that for an accurate assessment of the intrinsic fracture toughness of biological materials, it is imperative that a sharp crack-like stress concentrator be used to initiate fracture. The use of even sharpened machined notches can significantly, and erroneously, increase the measured fracture toughness value.

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