On the fracture of human dentin: Is it stress- or strain-controlled?

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Abstract: Despite substantial clinical interest in the fracture resistance of human dentin, there is little mechanistic information in archival literature that can be usefully used to model such fracture. In fact, although the fracture event in dentin, akin to other mineralized tissues like bone, is widely believed to be locally strain-controlled, there has never been any scientific proof to support this belief. The present study seeks to address this issue through the use of a novel set of *in vitro* experiments in Hanks' balanced salt solution involving a double-notched bend test geometry, which is designed to discern whether the critical failure events involved in the onset of fracture are locally stress- or strain-controlled. Such experiments are further used to characterize the notion of "plasticity" in dentin and the interaction of cracks with the salient microstructural features. It is observed that fracture in dentin is indeed locally strain-controlled and that the presence of dentinal tubules does not substantially affect this process of crack initiation and growth. The results presented are believed to be critical steps in the development of a micromechanical model for the fracture of human dentin that takes into consideration the influence of both the microstructure and the local failure mode. © 2003 Wiley Periodicals, Inc. J Biomed Mater Res 67A: 484–495, 2003

Key words: dentin; fracture; double-notched bend test; fractography; microstructure

INTRODUCTION

Dentin, the most abundant mineralized tissue in the human tooth, is located between the hard exterior enamel and the softer interior pulp. There is very limited understanding of its structural behavior, despite the fact that this is essential to the prediction of how microstructural alterations due to dentin pathologies and their treatment can degrade the strength and structural integrity of the tooth. Indeed, although there are some five decades of research on the mechanical properties of dentin (e.g., Refs. 1–16), there is still little fundamental comprehension of some of the basic questions that dictate its structural behavior. Notable among these is how fracture occurs in dentin. In particular, no micromechanical model for the fracture of dentin, which incorporates a local fracture criterion with consideration of the role of the salient microstructural features, has ever been developed. Such a model is essential for the understanding of how microstructural alterations can influence the toughness of dentin and thus affect the useful life of the tooth.

To date, only a handful of quantitative studies have been reported in archival literature on the fracture toughness* of human dentin. The earliest of these was by Rasmussen et al.[10,11] who used a "work of fracture" (defined as the work per unit area to generate new crack surface) to quantify the resistance to fracture. Unfortunately, the work of fracture, so defined, can be highly dependent on the geometry and sample size. Therefore, the results reported cannot be compared

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*The fracture toughness is a fracture mechanics-based parameter that is used to describe the onset of fracture. For an elastic body, it is defined at fracture either in terms of a critical value of the strain-energy release rate, G_c, defined as the change in potential energy per unit increase in crack area, or in terms of a critical value of the stress-intensity factor, $K_c = Q\sigma_{app}(\pi a_c)^{1/2}$, which characterizes the local stress and displacement fields ahead of a sharp crack (σ_{app} is the applied stress, a_c is the critical crack length, and Q is a geometry factor of order unity). Under linear-elastic conditions for mode I (tensile opening) loading, $K_c = (E'G_c)^{1/2}$, where E' is the appropriate elastic modulus for plane stress or plane strain.

quantitatively with any subsequent measurements. However, their studies did suggest that the toughness varies with orientation. Specifically, the work of fracture tended to be lower for cracking perpendicular to the dentinal tubular direction (i.e., in the plane of the mineralized collagen fibrils), compared with all other directions. This finding suggests that crack bridging by collagen fibrils could enhance the toughness along directions parallel to the tubule axes. Indeed, Ref. 10 does indicate that crack propagation perpendicular to the tubules is more energetically favorable, consistent with the notion that there can be no fiber bridging in that direction, although the excessive scatter in their results make definitive conclusions difficult.

A subsequent study by el Mowafy and Watts[6] was the first to use fracture mechanics, using compact-tension specimens to measure the fracture toughness, K_c, of dentin. For an orientation parallel to the long axis of the tubules, these authors reported a toughness of $K_c = 3.08$ MPa$\sqrt{m}$ (SD 0.33 MPa$\sqrt{m}$) for dentin, which was found to remain constant from 0° to 60°C. However, their experiments were conducted on machined notches, rather than with atomically sharp (e.g., fatigue) precracked samples, and it is well known for a wide variety of materials including metals, ceramics, and composites (e.g., Refs. 17–25) that the presence of a notch of finite radius, rather than a sharp precrack, can lead to severe overestimates of the fracture toughness.

More recently, Iwamoto and Ruse[13] reported fracture toughness values and an effect of tubule orientation for human dentin, using the so-called notchless triangular prism specimen geometry. However, the accuracy of these data may be deemed somewhat questionable in light of the nonstandard nature of the toughness test. Imbeni et al.,[15] conversely, used fatigue-precracked three-point bend bar samples (nominally conforming to ASTM standards) to determine an accurate measure of the *in vitro* fracture toughness of human dentin and to further assess the influence of notch acuity on such results. Measurements made in an orientation perpendicular to the tubules to determine a worst-case value yielded a fracture toughness of $K_c = 1.79$ MPa$\sqrt{m}$ (SD 0.1). This value is considerably lower than the earlier el Mowafy et al.[6] result, consistent with the observations of Imbeni et al.[15] that toughness values are significantly increased with increasing root radius.

The aim of the present study is to gain a further understanding of the factors that contribute to the *in vitro* fracture properties in human dentin by discerning the nature of the microscopic local failure criteria for the onset of cracking and characterizing how the subsequent crack growth is affected by microstructure.

BACKGROUND

Qualitatively, local fracture events that result in macroscopic fracture can be described as either locally stress- or strain-controlled. This is an important distinction in understanding the nature of fracture from a rounded notch or sharp crack (as in a toughness test), because in the presence of any degree of plastic deformation or inelasticity, the maximum local strains are *at* the crack or notch tip, whereas the maximum local stresses are *ahead* of the tip (Fig. 1).

For example, brittle fracture in metallic and ceramic materials (e.g., by cleavage cracking) is invariably modeled as (tensile) stress-controlled, involving the unstable propagation of a microcrack, initiated when the local tensile stresses exceed some critical local fracture stress.[26] Ahead of a sharp crack[27,28] or rounded notch,[29,30] the probability of this local event occurring is generally highest at the location of maximum tensile (or hydrostatic) stress, which in the absence of yielding is *at* the crack or notch tip (e.g., as in ceramics). However, with inelastic deformation, some degree of blunting occurs at a crack tip so that the location of the maximum stresses,[29,31] and hence the most probable site for the initiation of fracture,[32,33] moves *ahead* of the tip (e.g., as in metals). The location of this site depends on several factors, including the applied stress intensity K, yield strength σ_y, Young's modulus, E', and, in the case of a notch, its root radius or included angle. For a sharp crack, it is located at roughly two crack-tip opening displacements from the crack tip[31] (i.e., at a distance on the order of $K^2/\sigma_y E'$) (for a general description, see Refs. 34 and 35); for a rounded notch, it is several orders of magnitude further away from the tip, essentially at, or just behind, the boundary of the plastic zone, [i.e., at a distance on the order of $(K/\sigma_y)^2$].[29,30] Conversely, ductile fracture has been modeled as locally strain-controlled, involving the initiation and coalescence of voids. Ahead of a sharp crack or rounded notch, this event is most likely to occur at the location of maximum equivalent strain, which is at the crack or notch tip.[35–37]

From the foregoing discussion, it is apparent that for materials that display any degree of inelasticity, the locations of the microstructural cracking events that precede the onset of macroscopic fracture ahead of a notch are a definitive indicator of whether the fracture is locally stress- or strain-controlled; strain-controlled fracture will initiate at the notch, whereas stress-controlled fracture will initiate ahead of the notch.[33,35,37–46]

It should be noted here that the separation of the sites of peak tensile stress and plastic strain are the direct consequence of some degree of inelasticity ("yielding") at the notch tip. However, in dentin, such yielding cannot be simply related to pure pressure-

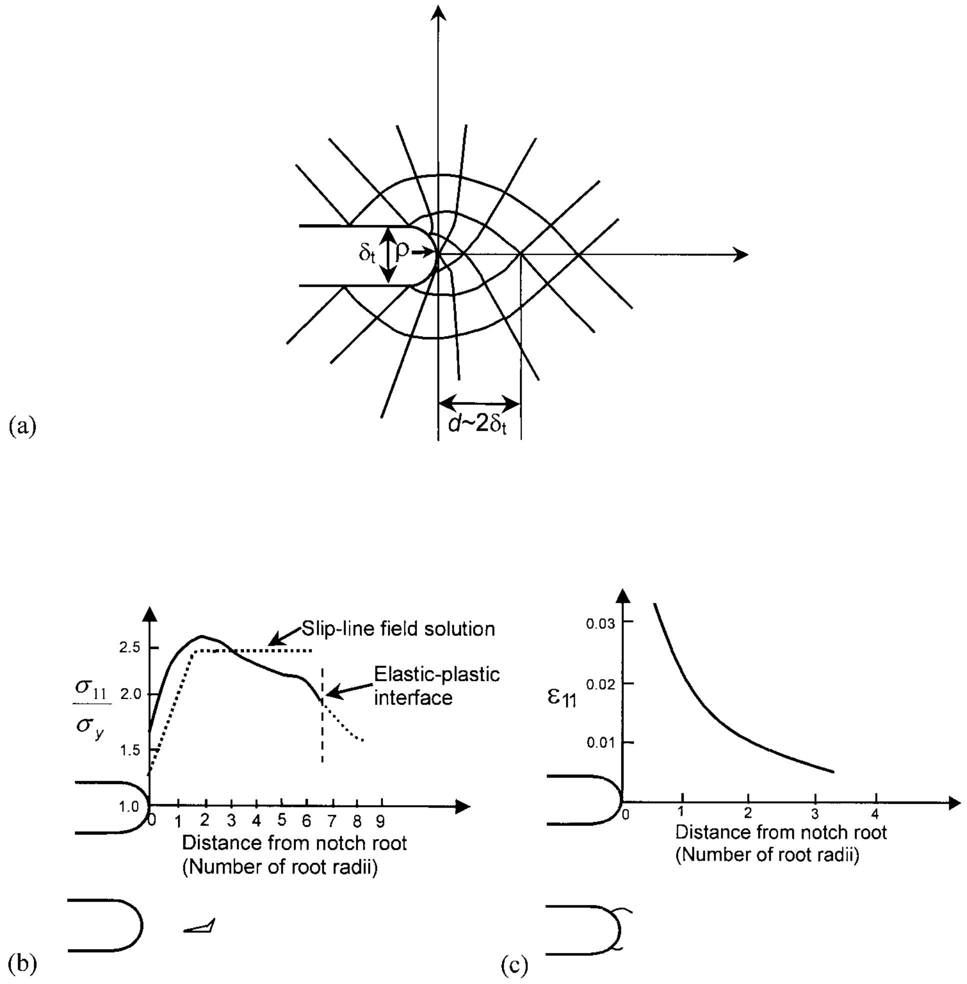

Figure 1. (a) Hill's logarithmic spiral slip-line field for a rounded notch in an elastic-plastic material.[29], and the (b) tensile stress, σ_{11}, and (c) plastic strain, ϵ_{11}, distributions, based on slip-line field analysis[29] and numerical computations,[30] ahead of such a notch. Also shown are schematic illustrations of possible (b) stress-controlled and (c) strain-controlled fracture mechanisms emanating from such notches. Note for stress-controlled mechanisms, the initial fracture event is *ahead* of the notch, whereas it is *at* the notch root for strain-controlled mechanisms.

insensitive, shear-driven plasticity, as in metals, for which the notch-field solutions in Figure 1 were explicitly derived. Although the precise nature of the inelastic constitutive behavior of dentin is not known, it clearly involves processes such as diffuse microcracking damage (e.g., Refs. 47 and 48), plasticity in the highly extendable, low-modulus collagen fibrils (which would be sensitive to both tensile and shear stresses, akin to pressure-dependent yielding in traditional polymeric materials), and poroelasticity (due to the considerable fluid volume distributed in an open porous structure). Despite this, most theoretical models for both deformation and fracture (e.g., Refs. 49 and 50) in mineralized tissues, such as dentin and bone, simply use the von Mises criterion, which is based on pressure-insensitive plasticity theory. However, recent finite element results[51] for inelastic deformation by both pressure-insensitive plasticity and pressure-sensitive microcracking indicate that the notch stress and strain fields are qualitatively similar (i.e., the local stresses peak still ahead of the notch, and the local strains peak at the notch root). It is in this spirit that we use the notch-field solutions, although it must be recognized that the precise quantitative details of these fields have yet to be determined for dentin.

Given this difference between the notch stress and strain distributions, the distinction between stress- or strain-controlled fracture can be achieved through the use of the double-notched four-point bend sample (Fig. 2), which has two important features to enable the evaluation[42–44,51–53]:

- Rounded notches with a large root radius ($\rho >$ 200 μm) are used (rather than sharp cracks) to maximize the difference between the locations of maximum stress and maximum strain ahead of the notch. Hill's well-known logarithmic slip-line

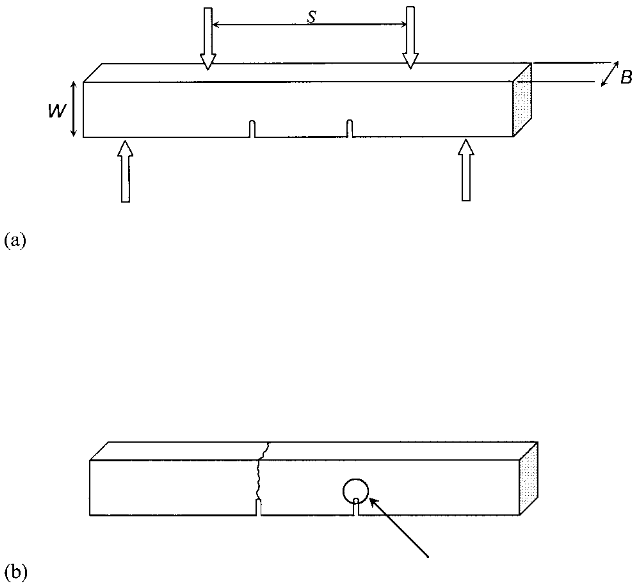

Figure 2. (a) Schematic illustration of the double-notched four-point bend test used to discern whether fracture is stress- or strain-controlled. Between the inner two loading points, the bending moment is constant; thus, when one notch breaks, the other is "frozen" at a point just before fracture instability. The region beneath this unbroken notch [as indicated by the arrow in (b)] is then carefully examined to determine the site of the precursor microscopic events involved in the fracture process.

  field solution[29] for the notch in a perfectly plastic solid [Fig. 1(a)] gives the location of the maximum tensile stress (and maximum degree of triaxiality) at the elastic-plastic interface. Subsequent finite-element solutions for a linear work-hardening solid by Griffiths and Owen[30] show that this location is actually behind the elastic-plastic interface; depending on the nominal (far-field) applied stress, it is typically at distances of 0.7–3 times the notch root radius ahead of the notch [Fig. 1(b)]. Conversely, the maximum strains are at the notch root and decrease monotonically with distance ahead of the notch.[30]

- Two notches in four-point bending are used because there is a constant bending moment between the inner two loading points, hence both notches see exactly the same stress and strain states. For a nominally brittle material, such as dentin, this means that when unstable fracture ensues from one notch, the other notch will be literally at the point of instability, thereby "freezing in" the local microstructural events that precede fracture. Thus, examination of the microstructure below this unbroken notch enables an evaluation of how and where the critical cracking processes initiate and in doing so define whether the process is stress- or strain-controlled.

It might be noted here that even though the actual measurement of the fracture toughness must involve fracture from a nominally atomically sharp crack, this particular experiment is best carried out with rounded notches because the distinction between the sites of the maximum tensile stress and strain substantially diminishes as the root radius, $\rho \rightarrow 0$.[29–31]

Micromechanical models for fracture incorporating such local failure criteria have been widely developed for metallic systems [e.g., the so-called RKR model for stress-controlled cleavage fracture[18] and the stress-state modified strain-controlled fracture model for ductile (microvoid coalescence) fracture[23–25]]; however, such deliberations have not been undertaken for the fracture of biomaterials such as dentin.[†] This is somewhat surprising because the strain-based criteria have been widely used to model such materials [e.g., bone (e.g., Refs. 47, 54, and 55)]. Indeed, because dentin displays marked "yielding" and postyield behavior, in the form of irrecoverable damage, as shown below in the section on Inelasticity or "Yielding" in Dentin, a strain-based fracture criteria would appear to be most probable, although none of this has ever been proved conclusively.

The prime objective of the current work is to use the philosophy described above to determine systematically the nature of the local fracture mechanisms in human dentin, by using the double-notched bending test described above. In addition, the aim is to address several unanswered questions concerning the interaction of the crack with the microstructural features in dentin, specifically: 1) whether the tubules actually affect the macroscopic crack path, 2) the nature of the interaction as the crack tip encounters a tubule, and 3) the role of the mineralized peritubular dentinal cuff in enhancing the toughness.[15]

It is believed that, in addition to the mechanistic understanding gained, this work is of importance from a clinical perspective. Notches resulting from natural caries, and possibly dental repair processes, are common in human teeth, and as such, a mechanistic understanding of dentin fracture from such notches may be considered critical for the development of a methodology for prediction of tooth failures.

EXPERIMENTAL PROCEDURES

Materials

Recently extracted human molars, sterilized with use of gamma radiation after extraction,[56] were used in the present study. Sections (~1.5–2.0 mm thick) were prepared from the

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[†] Recent studies using the double-notched geometry have shown that fracture in human cortical bone is consistent with a strain-based criterion.[51,53]

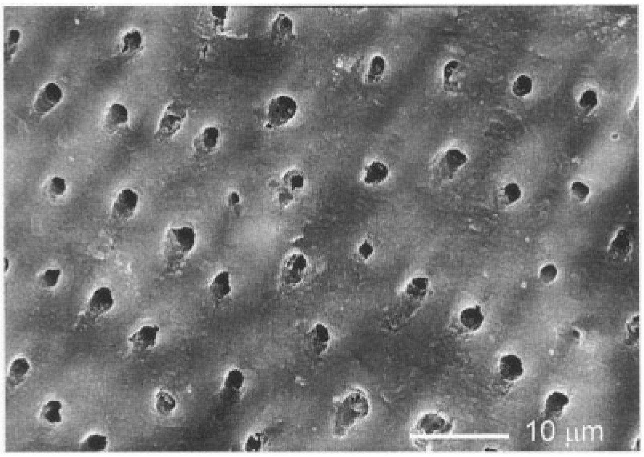

Figure 3. A scanning electron micrograph of the typical "microstructure" of human dentin. The tubules that are the characteristic feature of the microstructure are evident.

central portion of the crown and the root vertically through the tooth. The typical microstructure of dentin is shown in Figure 3, with the tubules being the most characteristic feature; this structure is described in more detail below in the section on Microstructure of Dentin. Although specimens were cut to define a specific orientation of the microstructure with respect to the crack path, in actuality it is almost impossible *a priori* to align the fracture plane precisely with, for example, 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.[57] This is well illustrated by the scanning electron micrograph in Figure 4 showing the side of a notch and the fracture surface of the crack emanating from it; despite the fact that these two surfaces are mutually perpendicular, the orientation of the tubules appears to be identical on the two planes. Consequently, the orientation of the crack plane had to be determined from examination of the crack paths and the fracture surfaces.

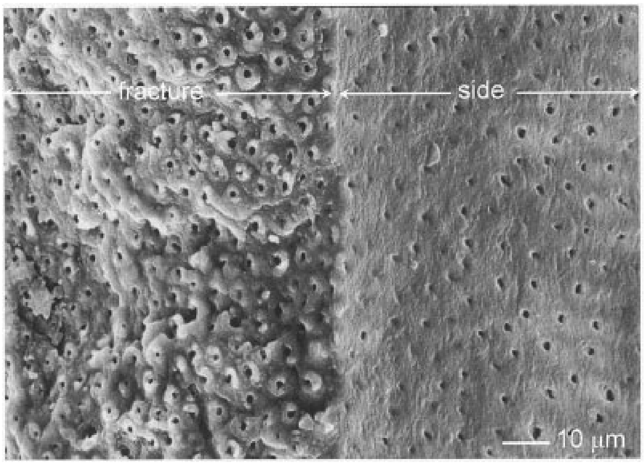

Figure 4. Scanning electron micrograph of the fracture surface (left side) and the side of a failed notch (right side), showing the strikingly similar orientation of the tubules on the two mutually perpendicular surfaces.

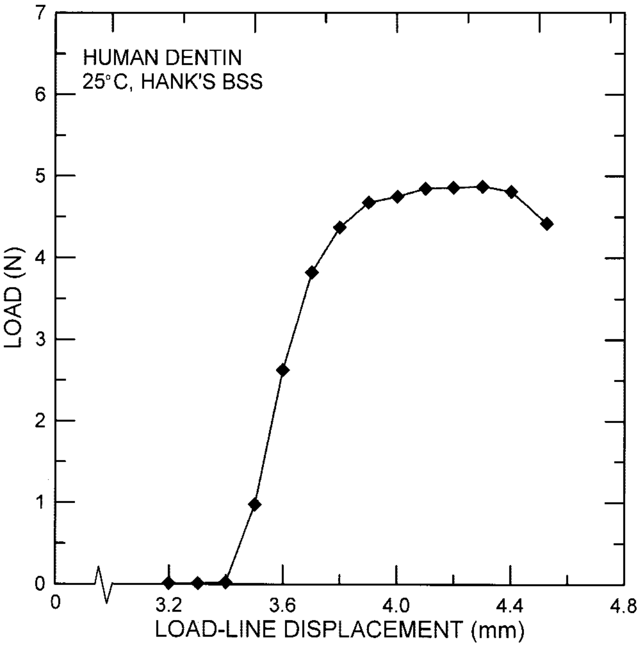

Figure 5. A typical load-displacement curve for human dentin tested in cantilever bending in HBSS solution at 25°C. Note the apparent "yielding" and postyield behavior. Tests were conducted in displacement control at a constant crosshead movement rate of 0.01 mm/s.

Beams of dentin measuring roughly 1.0 × 1.0 × 12.0 mm were then obtained from these sections by carefully further sectioning using a slow-speed diamond saw and subsequent wet polishing up to a 600 grit finish. These beams were then stored in Hanks' balanced salt solution (HBSS) at ambient temperature. Although mineral can be leached into solution when storing dentin in deionized water, which can result in changes in elastic properties with storage time, no such changes could be detected after a short-time storage in HBSS.[58] However, the precise effects of storage solution on the fracture properties have not been investigated *per se*.

In vitro first yield (σ_y) and maximum flexural (σ_F) strengths were measured under displacement control (crosshead displacement rate of 0.01 mm/s) in ambient-temperature HBSS in bending to be, respectively, $\sigma_y \sim 75$ MPa and $\sigma_F \sim 160$ MPa. A typical *in vitro* load-displacement curve is shown in Figure 5. Macroscopically, there is clear evidence of "yielding," akin to that observed commonly in many metallic systems. As noted above, it is believed that such inelasticity is the result of irrecoverable microdamage induced during loading, similar to that reported for trabecular bone[47]; this issue is addressed in more detail below in the section on Inelasticity or "Yielding" in Dentin.

Mechanical testing

Tests were conducted in HBSS by using a symmetric four-point (pure) bending geometry with a double notched configuration [Fig. 2(a)], as described above; this sample creates a constant bending moment between the inner two loading points, which in the present tests had a span *S* equal to 2–4 times the width *W* of the beam. Rounded notches, of root radius, $\rho \sim 200$–$300\ \mu$m, and depth, $a \sim 0.3$–$0.4\ W$, were introduced carefully with a slow-speed saw; care was taken to maintain the specimens in a hydrated state throughout the specimen preparation process. The depths of both notches in each specimen were kept as identical as possible to ensure similar stress/strain fields at the notch tips. A total of 10 such tests were conducted. Conditions can be considered to be nominally plane strain because, in accordance with the ASTM Standard E-399 for fracture toughness testing, the thickness of the bend specimens was comparable to $2.5\ (K_c/\sigma_y)^2$ (~1.4 mm).

All testing was conducted at ambient temperature on an ELF® 3200 series voice coil-based mechanical testing machine (EnduraTEC Inc., Minnetonka, MN). The bend bars were loaded to failure under displacement control at a constant cross-head movement rate of 0.02 mm/min in HBSS at 25°C.

Microstructural characterization

The area around the unfractured notch in the fractured double-notched samples was examined by using a high-power optical microscope and (after coating with a gold-palladium alloy) with a scanning electron microscope (SEM) operating in the back-scattered electron mode. To minimize the possibility of any damage during specimen preparation, the dentin beams were kept hydrated during all preparation and testing procedures, and for SEM imaging until the actual coating was performed. Moreover, given the further possibility of dehydration-induced cracking during the SEM imaging itself, some specimens were first observed in a hydrated condition in an environmental SEM and an atomic-force microscope to verify that the cracking configurations observed were not artifacts of the experimental procedure. However, the micrographs presented in this article were obtained from a conventional SEM because this provided the maximum resolution. In addition, postfailure observations of the fracture surfaces of the broken ligaments were conducted by using the same techniques.

RESULTS AND DISCUSSION

Microstructure of dentin

Human dentin is a hydrated composite of nanocrystalline carbonated apatite mineral (~45% by volume), type I collagen fibrils (~30% by volume), and fluid (~25% by volume). The mineral component is distributed in the form of 5-nm-thick crystallites in a scaffold created by the fibrils (typically 50- to 100-nm diameter). The most distinctive feature of this "microstructure" is a unique distribution of 1- to 2-μm-diameter cylindrical tubules (Fig. 3) that run from the dentin-enamel junction (DEJ) to the soft pulp in the interior of the tooth. These dentinal tubules are actually the paths of the odontoblast cells during tooth formation. They are surrounded by a collar of highly mineralized peritubular dentin (~ 1 μm thick) and are embedded within a matrix of mineralized collagen (intertubular dentin). The mineralized collagen fibrils form a planar felt-like structure oriented perpendicular to the tubules.[59] The tubules are randomly displaced about a periodic lattice[60] but with a distribution that depends on location within the tooth (e.g., Ref. 8). The interaction of these tubules with cracks in the dentin is of obvious interest from the perspective of an understanding of the failure of dentin; this issue is addressed below in the section on Double-Notch Experiments.

Inelasticity or "yielding" in dentin

Because the concept of the double-notched four-point bend test to distinguish between stress- and strain-controlled fracture is based on the premise of inelasticity or plastic yielding, it is important to establish that inelastic deformation does indeed occur in human dentin. Although such deformation is well understood for traditional materials (e.g., in the form of dislocation activity in metals), the mechanism of "yielding" in dentin is far less characterized, but it can be considered in terms of regions of "microdamage," as in bone (e.g., Ref. 47). Moreover, the nonlinear, nonrecoverable nature of the load/displacement curve measured for dentin in the current work (Fig. 5) is indicative that some form of "plastic" yielding does indeed occur.

With respect to localized yielding in the presence of cracks, the notion of a plastic zone is invariably used, where the stresses locally exceed the "yield" strength, σ_y, of the material. The plastic-zone size can be roughly estimated from continuum arguments (e.g., Ref. 34) to scale with the square of the ratio of the fracture toughness to the "yield" strength; specifically, at the onset of fracture, the maximum dimension is approximately $r_y \sim 1/2\pi\ (K_c/\sigma_y)^2$, which in dentin is on the order of 100 μm.[#] The boundary of this zone is drawn around a typical crack in human dentin in Figure 6(a). It is evident that within this zone [Fig. 6 (c) and (d)], "microdamage" can be seen in the form of cracked dentinal tubule cuffs, whereas no such damage is apparent outside the zone [Fig. 6(b)]. Thus, the

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[#] Note that the relationship used to estimate the plastic-zone size, $r_y \sim 1/2\pi\ (K_c/\sigma_y)^2$, pertains roughly to the maximum extent of the zone in plane strain and the forward extent in plane stress. Thus, it is a reasonable approximation for all stress states.

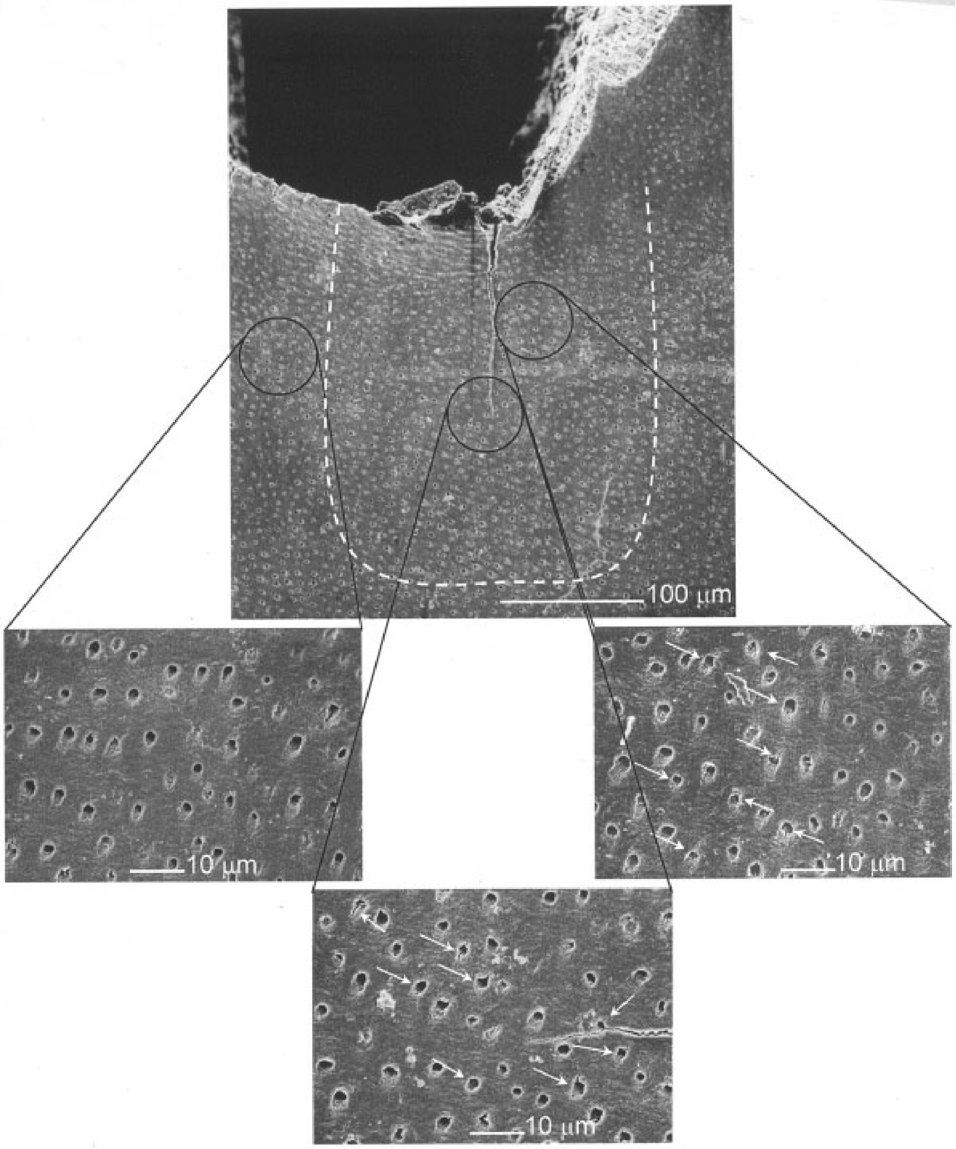

Figure 6. Scanning electron micrographs of the concept of a "yielding" or "damage" zone is shown, based on the regions where the peritubular cuffs are cracked. (a) An overview of a crack emanating from a notch, with the nominal damage zone indicated as a dotted line based on the continuum solution for the "plastic-zone" size (see text), and high magnification micrographs of areas (b) outside the zone, (c) inside the zone, and (d) at the notch tip. Note the extensive cracking of peritubular cuffs in the "damage zone" in (c) and (d) (indicated by white arrows).

existence of a nonlinear load/displacement curve and formation of a "microdamage" zone in the high-stress regions surrounding a crack are strong evidence that dentin does display inelastic deformation, consistent with the notion of microcracking of the peritubular cuffs. It is appreciated, however, that other factors, e.g., "plasticity" in the collagen fibers, may also play a role.

Double-notch experiments

Results from the double-notched four-point bend tests, which were used to detect the microstructural events before macroscopic fracture in dentin specimens, are shown in the macroscopic images in Figure 7, before and after failure at one of the notches, and in the microscopic SEM images of the crack paths in Figures 8 and 9. Macroscopic and microscopic images of the fracture surfaces are shown in Figure 10. As described above, when one notch breaks, the other is "frozen" at a point immediately preceding unstable fracture. Examination in the scanning electron microscope of this region, marked by the white circle in Figure 7(b), clearly indicates that all precursor cracks form directly at the notch root, generally with evidence of multiple initiation, as shown in Figure 8. To provide absolute proof that these cracks actually

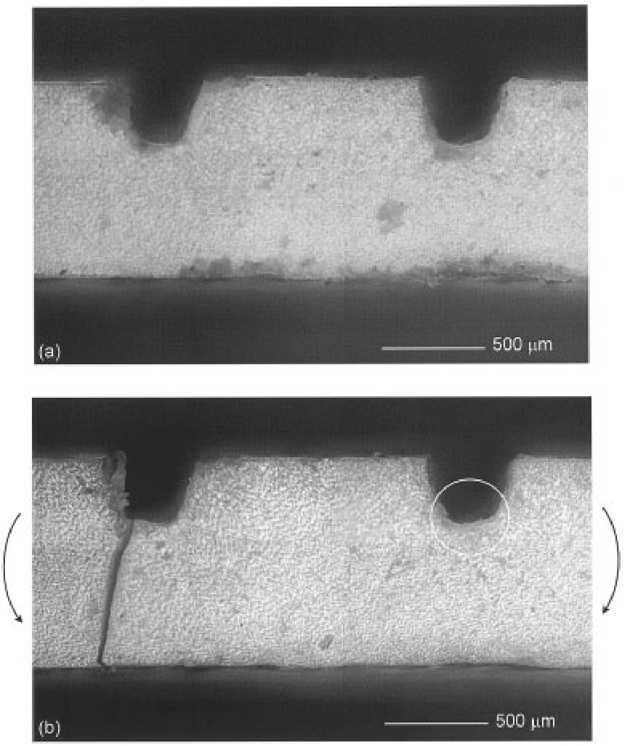

Figure 7. Optical microscope images of the double-notched four-point bend specimen (a) before and (b) after fracture. The critical area of interest, indicated by the white circle in (b), is in the region beneath the unbroken notch.

formed *at* the notch, serial sectioning should be used, although this was not feasible with the present samples because they were so fragile. However, the extremely small (<10 µm) size of the precursor cracks that are imaged (e.g., Fig. 9, where a ∼2- to 3-µm crack can be seen at the notch root surface) leaves little doubt that crack initiation is *at* the notch and not ahead of it.

For the specimens tested, the nominal elastic bending stress, σ_{nom}, at the notch tip at maximum load was in the 50–95 MPa range ($\sigma_{nom}/\sigma_y = 0.67–1.27$). According to the numerical analysis of Griffiths and Owen,[30] the maximum tensile stress, σ_{11}, occurs at ∼140–330 µm ahead of the notch tip (i.e., roughly at a distance of 0.7–1.1 times the notch root radius). Because absolutely no evidence of precursor cracking was found in this region and all cracking was detected exactly at the notch root, we can conclude that fracture in human dentin is indeed consistent with a locally strain-based criteria.

Microstructure/crack path interactions

SEM images of the subsequent propagation of the crack and its interaction with the salient microstructural features in human dentin are shown in Figures 11 and 12. Figure 11 shows of one such crack, which has propagated some 200 µm from the notch. The plane of the crack and the crack front are nominally parallel to the axis of the tubules, which are spaced roughly 10 µm apart. It is evident that at this scale of observation, the crack path is relatively deflection free, implying that the most recognizable feature of the microstructure (i.e., the tubules) do not have a major influence on the fracture process or the path taken by the growing crack. However, as noted above, there is ample evidence of damage in the form of cracking of the peritubular dentin cuff for the tubules in the vicinity of the growing crack, as indicated by the arrows and the insets in Figure 11. Because these subcracks form ahead of the main crack tip [Fig. 11(c)], using the arguments outlined above, their formation may be presumed to be stress-controlled. Where these cracked peritubular cuffs are within a few micrometers of the main crack

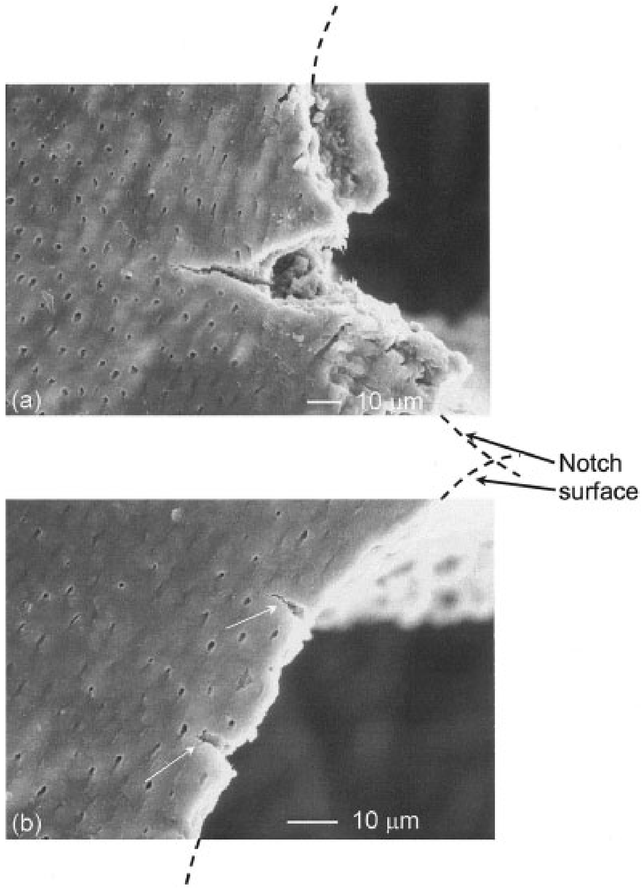

Figure 8. Scanning electron micrographs of the microscopic cracking events immediately before unstable fracture in a double-notched four-point bend test, showing micrometer-scale cracks emerging from the root of the unbroken notch. Multiple initiation sites are evident in (b). The fact that these precursor cracks form at the notch, as opposed to a few hundred micrometers ahead of it, is definitive proof that fracture in dentin is locally strain-controlled.

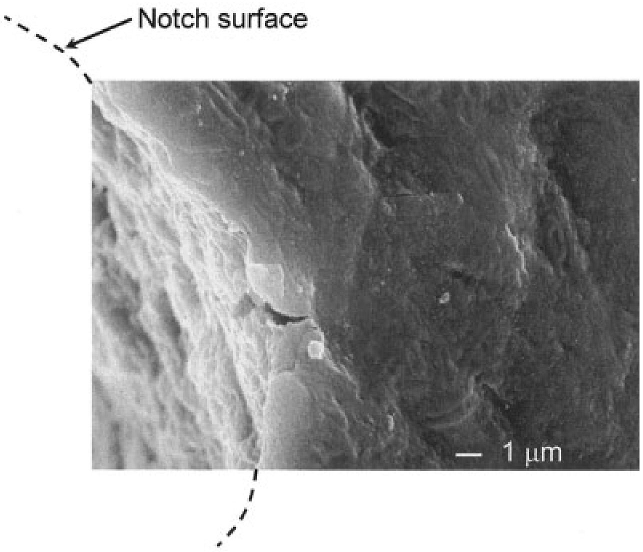

Figure 9. A scanning electron micrograph of a 1- to 2-μm precursor crack initiating at the root of the unbroken notch in a double-notched four-point bend test.

path, minor out-of-path deflections can be seen [Fig. 11(c)]. However, in general, because the crack path neither seeks out nor avoids these regions, as indicated by the high magnification SEM micrographs in Figure 12, it would appear that the role of the cracked peritubular cuffs or tubules themselves is minimal in influencing the toughening of human dentin.

These observations imply that the tubules may not, as has previously been suggested, play a causative role in the reported anisotropy in the mechanical properties of human dentin (e.g., Refs. 10, 11, and 13). Although not specifically examined in the present work, it is more likely that such anisotropy results from the orientation of the collagen fibril network (which may act to "bridge" the crack), as has been suggested for bone (e.g., Refs. 51, 53, 61, and 62), and more importantly from crack bridging due to the formation of uncracked ligaments.[63] It should be noted that investigations into such effects in elephant dentin (see Ref. 63 for further discussion) yielded a 55–65% higher toughness for an orientation where the long axis of the tubules was in the plane of crack propagation, compared to an orientation with the long axes perpendicular to both the plane and direction of crack propagation. Such results are consistent with a contribution to the toughness from crack bridging by collagen fibrils and uncracked ligaments, as discussed in detail elsewhere.[63]

CONCLUSIONS

On the basis of an experimental study of the *in vitro* fracture behavior of human dentin (at 25°C in HBSS), specifically to examine the character of the deformation, local fracture mechanisms, and the role of the principal microstructural features in influencing the process of crack initiation and growth, the following conclusions can be made:

1. Through the use of double-notched four-point bend testing to determine the location (in relation to the notch) and nature of the microstructural cracking events immediately before failure, fracture in human dentin has been shown to be consistent with a locally strain-controlled failure criterion.
2. Metallographic examination of a growing crack in dentin revealed the existence of a "microdamage" zone ahead and in the wake of the crack tip; the size of this zone was

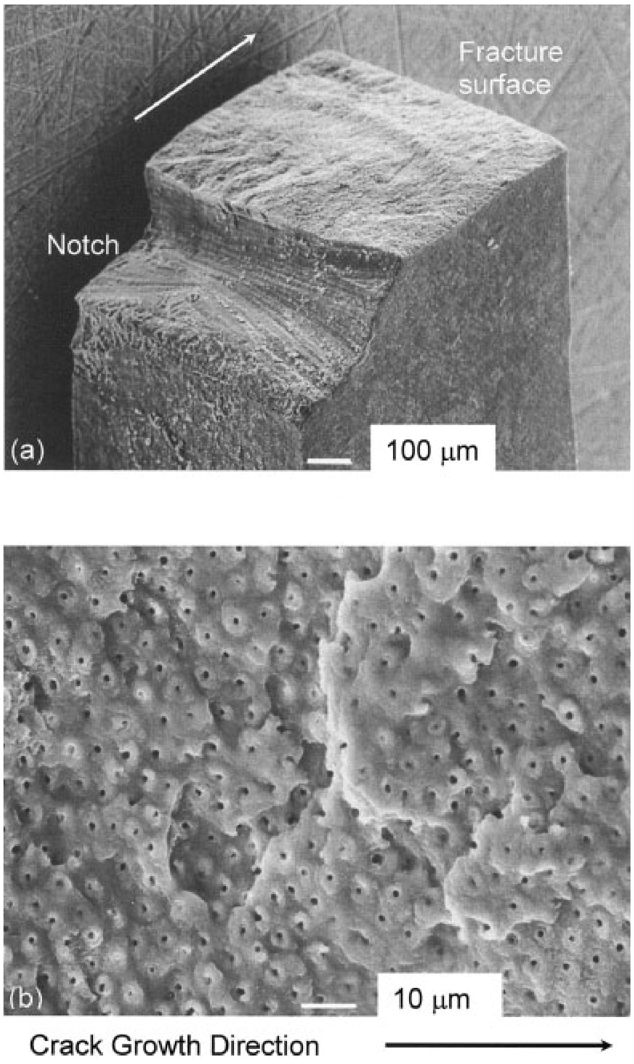

Figure 10. (a) A low-magnification scanning electron micrograph of the broken notch in a double-notched four-point bend test. The white arrow indicates the nominal direction of crack growth. (b) A high-magnification scanning electron micrograph of the morphology of the resulting fracture surface.

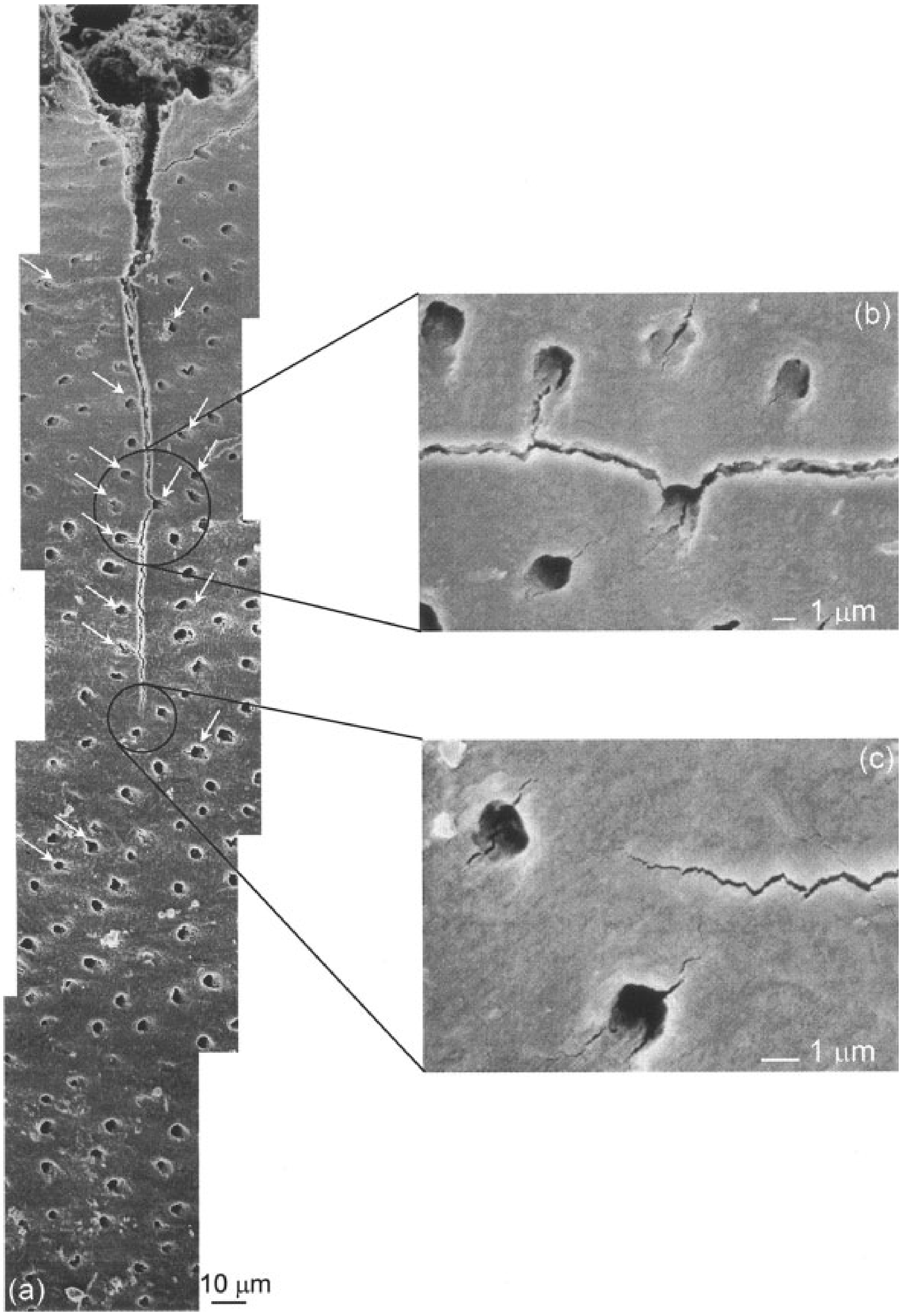

Figure 11. Scanning electron micrographs of an ~200-μm crack emanating from the notch, showing the relationship between the crack path with the salient microstructural features. Insets in (b) and (c) show higher magnification images of the cracking of the peritubular cuffs [also shown by the white arrows in (a)] and the interaction of the subcracks with the main crack path.

comparable with continuum estimates for the plastic-zone size, based on the "yield" strength of dentin. Within this zone, extensive evidence of microcracking of the dentinal tubule cuffs was apparent, consistent with the notion that "plasticity" in dentin involves some form of "microdamage."

3. Examination of the subsequent crack growth revealed only minimal interaction of crack with the presence and orientation of the tubules or the existence of cracked peritubular cuffs. It is concluded that the mild influence of orientation reported for the fracture toughness of human dentin is *not* associated with the tubules *per se* but more probably with the mineralized collagen fibrils that form a planar feltlike structure perpendicular to the tubule axes.

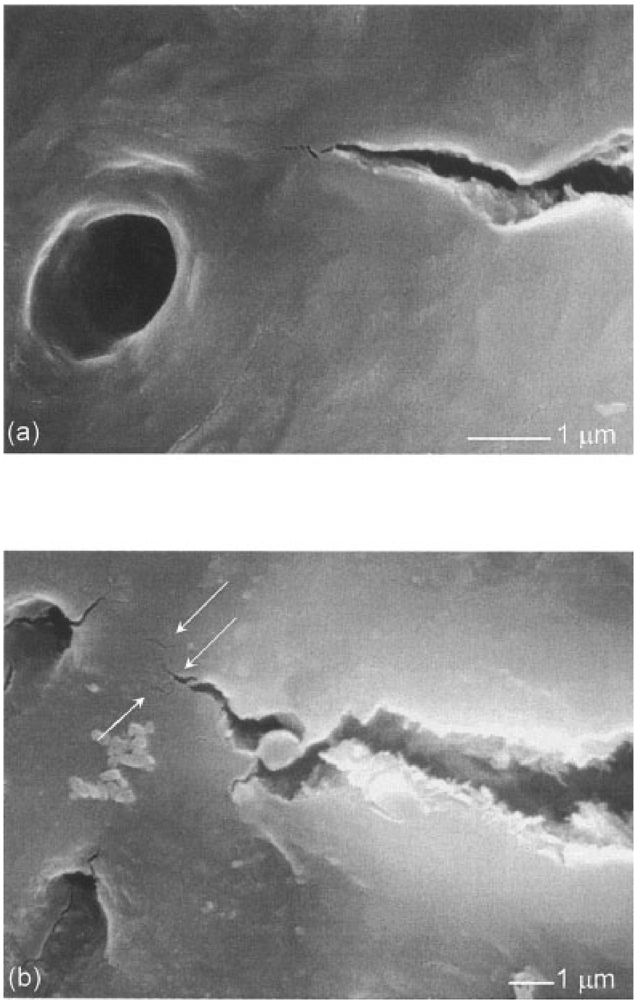

Figure 12. High-magnification scanning electron micrographs of the specific interaction of the tip of the main crack with (a) a tubule and (b) region of microdamage (indicated by white arrows) ahead of a crack tip.

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