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On the Mechanistic Role of the Dentin-Enamel Junction in Preventing the Fracture of Human Teeth

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On the Mechanistic Role of the Dentin-Enamel Junction in Preventing the Fracture of Human Teeth

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The dentin-enamel junction (DEJ), which is the interface between the dentin and outer enamel coating in teeth, is known for its unique biomechanical properties that provide a crack-arrest barrier for flaws formed in the brittle enamel. In this work, we re-examine how cracks propagate in the proximity of the DEJ, and specifically quantify, using interfacial fracture mechanics, the fracture toughness of the DEJ region. Additionally, we show that the vital function of the DEJ, in preventing cracks formed in enamel from traversing the interface and causing catastrophic tooth fractures, is not necessarily associated with the crack-arrest capabilities of the DEJ itself, but rather with the development of crack-tip shielding, primarily from uncracked-ligament bridging, in the mantle dentin adjacent to the DEJ. Measurements of the toughness of the DEJ region give estimates of $G_c \sim 115$ J/m^2, i.e., ~5 to 10 times higher than enamel and ~75% of that of dentin.

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The dentin-enamel junction (DEJ) in teeth represents the zone between two distinct calcified tissues with very different biomechanical properties: enamel and dentin[1]. Enamel is the hard and brittle outer portion of the tooth that envelops the softer dentin; it is comprised of defective carbonate-rich apatite (AP) crystals arranged in enamel rods (4-5 μm in diameter) or prisms that lie nearly perpendicular to the DEJ2[2,3]. Its fracture toughness is typically K_c~0.7-1.3 MPa√m in respective directions parallel and perpendicular to the enamel rods[4,5]. Dentin, conversely, is a biological composite which is tougher than enamel and similar at the nanostructural level to bone. It has a unique architecture consisting of dentinal tubules, ~1 μm in diameter, surrounded by peritubular dentin, consisting of ~0.5-1 μm thick cylinders of randomly-oriented apatite crystallites. These tubular units are embedded in a collagen matrix-apatite reinforced composite. Since the tubules are the formative tracks of the odontoblastic cells that move inward and reside on the pulp chamber surface, there are substantial variations in morphology and structure of the dentin from the DEJ to the pulp chamber[6]. Dentin has a K_c toughness that varies between 1.0 and 2.0 MPa√m in directions perpendicular and parallel to the tubules[7,8]. The toughness of dentin adjacent to the DEJ, so-called mantle dentin, is supposedly higher due to its lower mineral content and reduced modulus[9]; the tubules in this region are comparatively rare or absent.

The DEJ itself has a hierarchical microstructure with a three-dimensional scalloped appearance along the interface[1]. It is an anatomically thin region with a broader functional width; the enamel and dentin close to the interface have slightly different microstructures and properties than the more distant bulk phases. Specifically, the morphology of the collagen is such that type-I fibrils emanate from the dentin and project fibrils (~100 nm in diameter) perpendicular to the DEJ[10]; such Von Korf's fibrils cross the DEJ and appear to be inserted directly into the enamel. In contrast, collagen fibrils in bulk dentin are either parallel or at angles less than 90^{o} to the plane of the junction[1].

Although the fracture-resistant properties of the DEJ are believed to originate from a gradual change in microstructure and properties of dentin and enamel rather than from an abrupt transition between two dissimilar materials[11,12], the DEJ appears as a 'line' when imaged microscopically. This apparently sharp interface, the "optical DEJ", is thought to represent the original position of the basement membrane of the ameloblasts and odontoblasts, where they contact in the embryological tooth bud. Generally, interfaces between materials with dissimilar elastic and mechanical properties are "weak links" in a structure; the DEJ, however, acts to successfully transfer applied loads (e.g., masticatory or impact) from the enamel to the dentin and can inhibit cracks in enamel from propagating into the dentin and causing catastrophic fracture of the tooth[13]. Although there have been numerous attempts to explain this latter function[5,11,13-20], including that the DEJ is tougher than either dentin or enamel[1] because it is less mineralized[19] and contains more collagen[1,13], that it may reduce the stress concentration[1,20], or that it promotes crack deflection[14] due to microhardness[2,19] or modulus[11] differences across the interface, there is little consensus on the origin of the DEJ's crack-arrest properties.

There is also inconsistent information on the toughness of the DEJ region compared to that of enamel and dentin. Specifically, the range of DEJ toughnesses reported differ by a factor of three or more – from 336 J/m^2 [15,16] to 988 J/m^2 [13] when described in terms of energy (G_c) and from 0.6-0.9 MPa√m [2,6] to 3.4 MPa√m [13] when presented as a stress intensity (K_c); indeed, there have been only a few realistic measurements[13,14]. Accordingly, in this work, we apply a novel technique involving propagating indentation cracks into the interfacial region in human teeth to quantitatively assess the toughness of the DEJ; further, we identify the microstructural mechanisms by which the DEJ functions to inhibit cracks from traversing the interface to cause catastrophic tooth fracture.

To first assess the variation in properties across the interface, we measured Vickers hardness and indentation toughness profiles under hydrated conditions normal to the DEJ (Fig. 1). Similar to previous results[11,13,16,18,20], this indicated that the hardness of the enamel falls quite rapidly within a millimeter of the optical DEJ to reach a minimum in the mantle dentin in close proximity to the DEJ. Corresponding indentation toughness measurements show that that the toughness of the enamel also has a minimum close to the DEJ, but then rises steeply over the final ~200 μm into the DEJ. *These profiles clearly indicate that cracks in the enamel experience a region of decreasing hardness yet increasing toughness as they impact the DEJ.*

To quantitatively evaluate the toughness of the DEJ, we placed a series of Vickers microhardness indents (~40-50 per tooth) in polished sections of 13 non-carious extracted human molars (11 axial and two occlusal sections), each tooth being unique to a single patient. Indents were made under hydrated conditions at ~20-50 μm from the optical DEJ on the enamel side (Fig. 2) such that cracks emanating from the corners of the indents would propagate toward the dentin and impinge onto the DEJ at differing angles of incidence. We then observed whether the cracks penetrated the interface, arrested or deflected along the DEJ. Knowing the modulus and toughness of the two phases on either side of the interface, we deduced the interface toughness using an "interface impingement" technique developed by Becher *et al.* for ceramics[21]. The basis of this method is the linear-elastic solutions of He and Hutchinson[22] which govern whether a crack, which is incident on a bimaterial interface, will deflect along, or penetrate through, the interface; this event depends specifically on (i) the angle of incidence, (ii) the elastic mismatch across the interface (which is a function of the relative elastic moduli), and (iii) the ratio of fracture toughnesses of the interface and the material on the far side of the interface ($G_{c,interf}/G_{c2}$) towards which the crack is propagating (Fig. 3).

Based on a total of 172 indentation cracks examined, we found that more than 75% of the cracks actually penetrated the (optical) DEJ a short distance, only to arrest after propagating ~10 μm or less into the mantle dentin. As the absolute resolution of the optical metallograph (2000X) used to make this assessment was ~500 nm, there was some degree of uncertainty the remaining cracks, which either arrested at the interface or penetrated it by less than ~500 nm. No evidence was seen of substantial interfacial delamination at the DEJ (except on dehydration in the conventional SEM). In general, the vast majority of these cracks were normally incident, as cracking in the enamel occurs by separation of the enamel rods and these are aligned roughly perpendicular to DEJ. A few cracks which impinged on the DEJ at angles of between ~30 and 75 degrees to the interface were found to penetrate.

Such observations were confirmed by imaging in the SEM (Fig. 4). It is apparent that once a crack penetrates the DEJ and comes to arrest in the mantle dentin, it is significantly bridged close to the crack tip by "uncracked ligaments". Such uncracked ligaments are regions of unbroken material, a few micrometers in dimension, which span the crack in the wake of the crack tip. In general, they are created either by the non-uniform advance of the crack front and/or by the imperfect linking of microcracks, initiated ahead of the crack tip, with the main crack[23]; in dentin, it is believed that they are created primarily by the latter process involving microcrack formation at the tubules ahead of the main crack[24]. The resulting uncracked-ligament bridging is a widely observed toughening mechanism in structural materials[23] and has been identified as one of the prominent contributions to the toughness of dentin[24] (and cortical bone[25]). The bridging mechanism acts to reduce the crack-driving force by sustaining a portion of the applied load that would otherwise contribute to cracking. In the present case, *it is this reduction in crack-driving force due to such bridging which is responsible for bringing the crack to a halt, once it traverses the DEJ and encounters the higher collagen content of the dentin.*

Crack bridging may also be responsible for the lack of incidence of cracks causing delamination along the DEJ. In samples where the DEJ cracked open under vacuum in the conventional SEM, clear evidence was seen of bridging by intact individual collagen fibrils that span the DEJ; indeed, such a mechanism of collagen-fiber bridging has been proposed for the toughening of bone[26]. This notion is consistent with the fact that the DEJ is a complex interdigitation of enamel and dentin, with the enamel side being highly mineralized and the mantle dentin having more collagen, fewer tubules and less overall mineral than the bulk dentin. *We thus believe that collagen fibrils perpendicular to the interface constitute the key reinforcing mechanism at the DEJ, which explains why so few cracking events cause delamination when they impinge on the DEJ.*

For cracks initiated in the enamel, this study shows that such cracks are stopped by penetrating the (optical) DEJ to arrest within ~10 μm into the mantle dentin, an event promoted by the generation of uncracked-ligament bridging. To quantify this, we note that the lack of evidence of interfacial delamination leads to a criticality between penetration and arrest at the interface, which can be used to estimate a lower-bound toughness of the DEJ region.

Considering a crack in the enamel (material-1) propagating into the dentin (material-2) and knowing the toughness of dentin, G_{c2}, and the elastic mismatch at the interface, characterized by the so-called Dundurs' parameter $\alpha = (E_1 - E_2) / (E_1 + E_2)$, where E_1

and E_2 are the respective Young's moduli of the enamel and dentin, the toughness of the DEJ, $G_{c,interf}$, can be estimated from the He-Hutchinson solutions[22] for cracks perpendicular to an interface, specifically the $G_{c,interf}/G_{c2}$ vs. α plot shown in Fig. 3. Since these solutions are based on the strain-energy release rates rather than stress intensities, the value of the fracture toughness of dentin was calculated as $G_{c2} \sim 154$ J/m^2 from the measured K_c value of 1.8 MPa√m determined in ref. [7]. The modulus mismatch for the enamel/dentin interface is $\alpha \sim 0.53$, based on respective values[11] for the Young's moduli of enamel and dentin of 63.6 and 19.7 GPa. From Fig. 3, the critical ratio of the interface and matrix toughness, $G_{c,interf}/G_{c2,}$ for penetration rather than interface arrest of normally incident cracks is ~0.75. This yields a toughness of the DEJ of $G_{c,interf}$ ~115 J/m^2, which is much higher than that of enamel (G_{c1} ~ 10-25 J/m^2) but only ~70% of that of dentin (G_{c2} ~ 154 J/m^2).

In summary, our experiments have shown that the vital function of the DEJ in human teeth, in preventing cracks in the enamel from traversing the interface and leading to catastrophic tooth fractures, may not be necessarily associated with the crack-arresting capabilities of the DEJ *per se*; rather, cracks propagating from the enamel can penetrate the interface and propagate less than 10 μm or so into the mantle dentin before they arrest. Mechanistically, this is associated with the enhancement of bridging forces across the crack, generated by the creation of uncracked-ligament bridges once the crack enters in the mantle dentin. The absence of delamination along the DEJ also can be associated with the occurrence of bridging, from individual collagen fibrils that span the DEJ. Quantitatively, by considering the criticality between the arrest and penetration of cracks normally incident to the DEJ, a lower-bound estimate of the strain-energy release rate toughness of the DEJ was found to be 115 J/m^2, i.e., much higher than that of enamel but lower than that of dentin.

Methods.

Materials. Thirteen sections of non-carious extracted human molars sterilized by gamma radiation were used in this study. Storage of samples until preparation was at 4°C in distilled water with thymol. Sections were cut using a modified water-cooled diamond saw and the samples polished with 0.25 µm diamond paste. All samples were stored fully hydrated in Hanks' balanced salt solution (HBSS) prior to testing to prevent surface demineralization[27]; samples were additionally kept moist during testing by frequently spraying with HBSS.

Characterization. Crack trajectories were examined using optical, high-resolution scanning electron (SEM) and environmental scanning electron microscopy. Some sections were etched with H_3PO_4 (35% vol) to reveal microstructural features. Although samples were kept moist at all times, during imaging with conventional SEM, dehydration due to the vacuum sometimes led to cracking either along the line of indents or at/near the DEJ. To confirm that such spurious cracking did not compromise the results, samples were also examined in the environmental SEM under a water vapor pressure of 8 torr.

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Competing interests statement

The authors declare that they have no competing financial interests.

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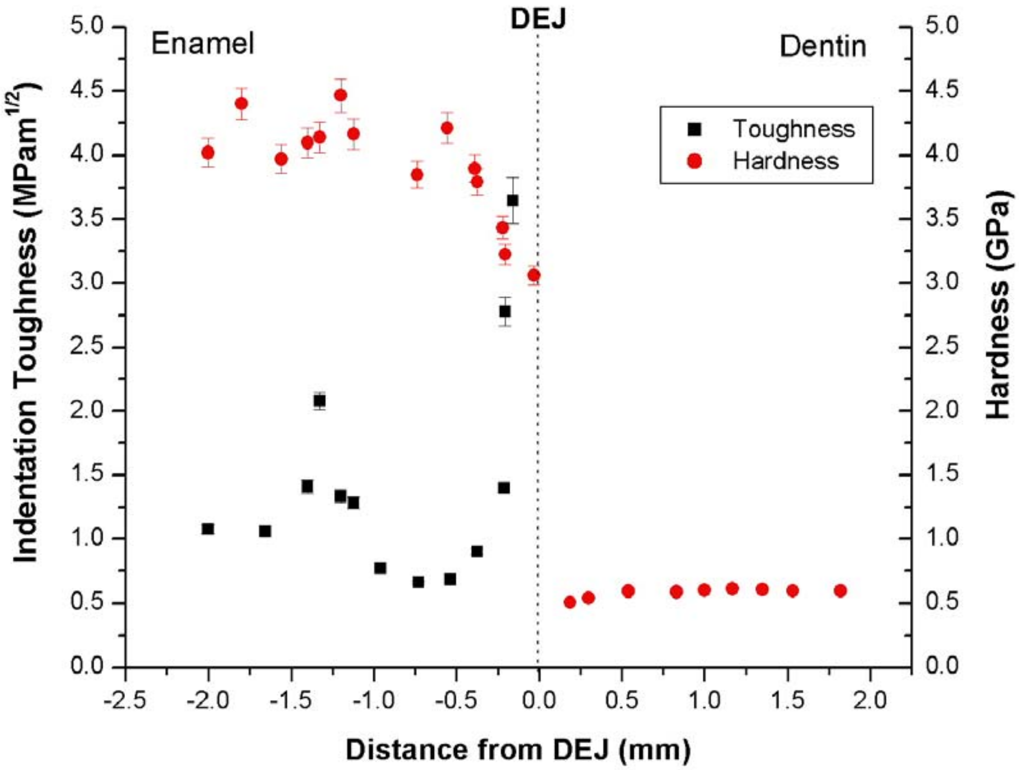
FIGURE CAPTIONS

Figure 1. Typical profiles of (a) the Vickers hardness and (b) the indentation toughness taken normal to, and across, the DEJ from the enamel to the dentin in a human molar. Hardness indentations were made with a load range between 3 and 5 N to minimize brittle fracture damage but to still form cracks around the indents to enable toughness measurements. Lines of indents were performed on three different teeth (each from a unique patient), with three series for each tooth. The indentation toughness[28], $K_{ind,\mathrm{c}}$, was determined from the indentation load P, and the average crack lengths, c, emanating from the indent corners, according to $K_{ind,\mathrm{c}} = \chi P/c^{3/2}$, where χ is the residual indentation coefficient (taken as 0.076 for enamel[4]). Such measurements could only be made in the enamel as inelasticity in the dentin suppresses the formation of indent cracks. These profiles show that cracks in the enamel experience a region of decreasing hardness yet increasing toughness as they approach the DEJ.

Figure 2. Optical micrograph of the placement of Vickers indents in the enamel within ~20-50 μm from the (optical) DEJ in a human molar, which were used to create cracks that impact upon the DEJ. Inset shows optical micrograph with Nomaski interference contrast of one such indentation with cracks, which emanate from the intent corners, propagating into the scalloped interface.

Figure 3. The linear-elastic solutions of He and Hutchinson[22] used to determine the conditions for a normally incident crack to penetrate or deflect along an interface between two materials *1* and *2*. Whether the crack penetrates or not is a function of the (i) impingement angle, (ii) the elastic mismatch across the interface, defined by the first Dundurs' parameter $\alpha = (E_1 - E_2) / (E_1 + E_2)$, where E_1 and E_2 are the respective elastic moduli for materials *1* and *2*, and E is Young's modulus[29], and (iii) relative magnitude of the interface toughness and the toughness of material *2* on the far side of the interface, ($G_{\mathrm{c,interf}}/G_{\mathrm{c2}}$). The figure shows a plot of $G_{\mathrm{c,interf}}/G_{\mathrm{c2}}$ as a function of the modulus mismatch α. For the enamel/dentin junction where $\alpha \sim 0.53$, the absence of interface delamination leads to a criticality between penetration and arrest at the DEJ, which can be used to estimate a lower-bound for the toughness of the DEJ, $G_{\mathrm{c,interf}}$, given by 0.75 of $G_{\mathrm{c,interf}}/G_{\mathrm{c2}}$, where G_{c2} is the toughness of the dentin.

Figure 4. Scanning electron micrographs (taken using a conventional SEM) showing examples of cracks from the enamel which are normally incident on the DEJ and are arrested after propagating less than ~10 µm beyond the interface into the mantle dentin. Behind the arrested crack tip, numerous uncracked-ligament "bridges" can be seen; these are regions of uncracked material that oppose the opening of the crack and sustain load that would otherwise be used for crack growth. Such bridging, which is a form of crack-tip shielding[30] and is prominent toughening mechanism in dentin and bone[24,25], acts to reduce the effective driving force for crack extension, thereby arresting the crack. Cracking can also be seen near, and nominally parallel, to the DEJ. However, by comparing these images with corresponding images in the environmental SEM (at 8 torr water pressure), such "delamination" cracking was found to be an artefact caused by dehydration *in vacuo* in the conventional SEM.



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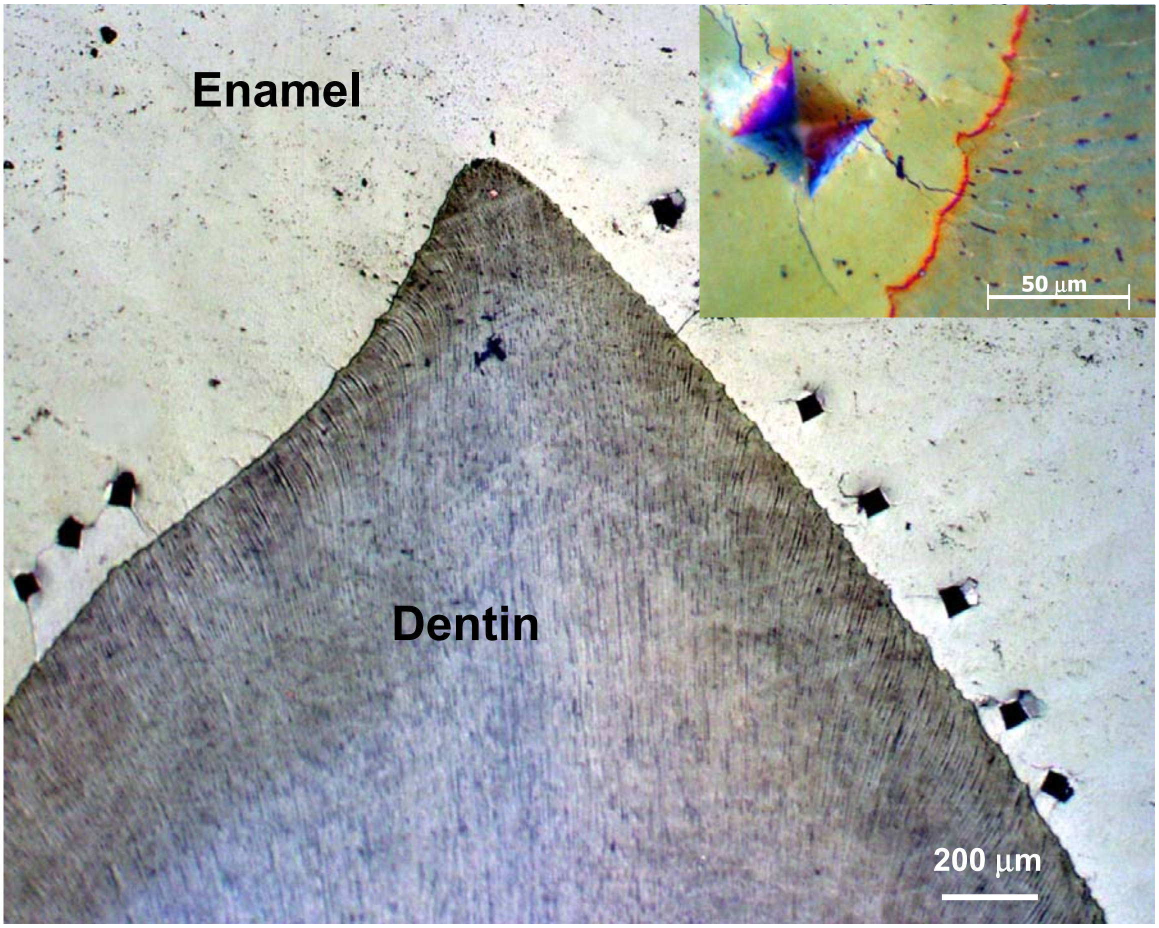

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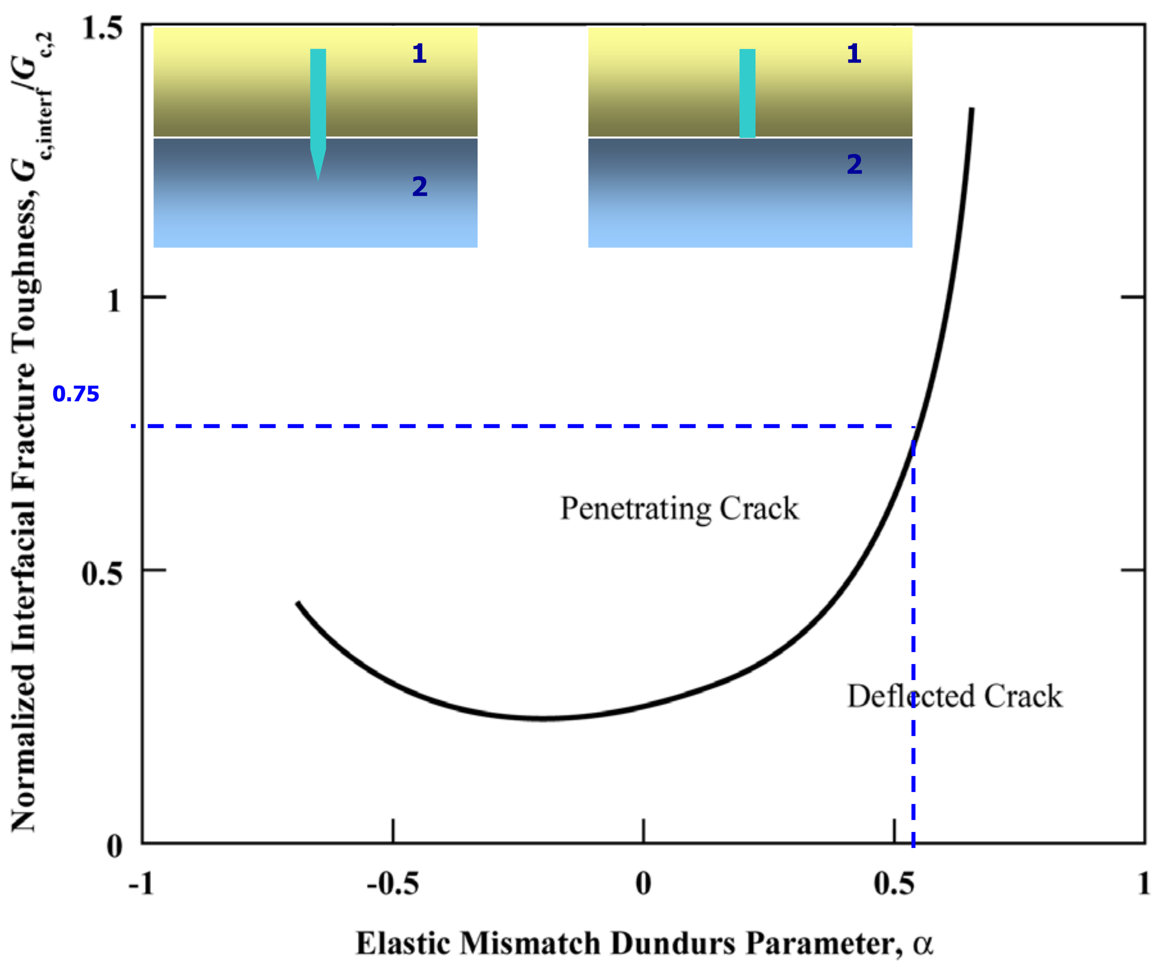

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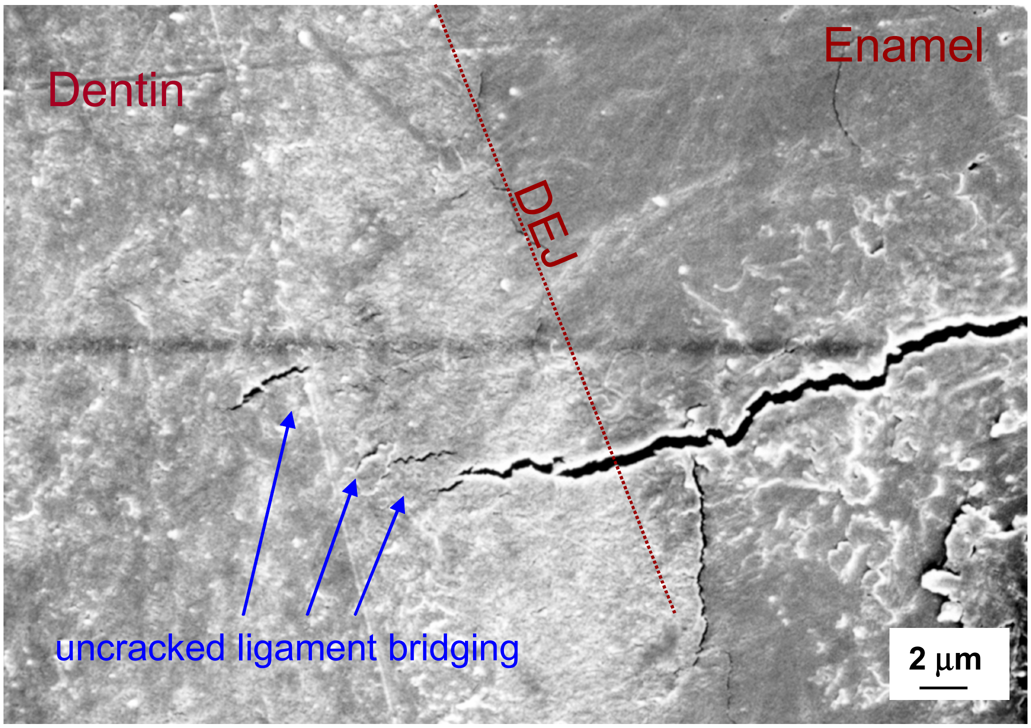
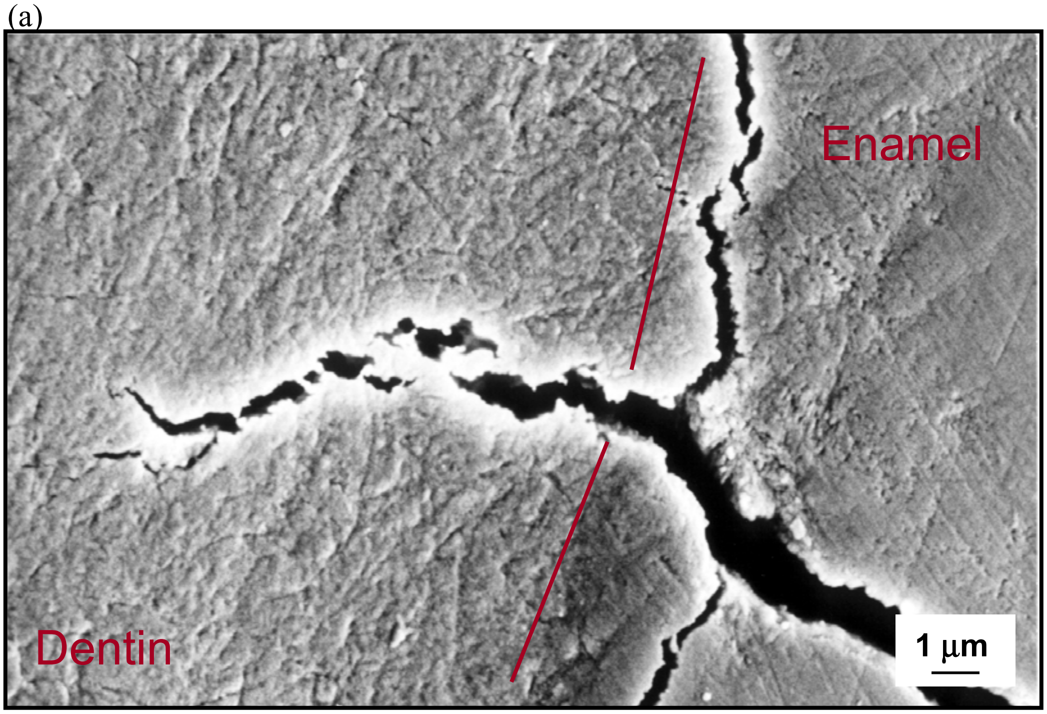


(a)



(b)

Figure 4. **Figure 4.** Scanning electron micrographs (taken using a conventional SEM) showing **e**xamples of cracks from the enamel which are normally incident on the DEJ and

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