Fracture Processes and Mechanisms of Crack Growth Resistance in Human Enamel

Devendra Bajaj, Saejin Park, George D. Quinn, and Dwayne Arola

Human enamel has a complex microstructure that varies with distance from the tooth’s outer surface. But contributions from the microstructure to the fracture toughness and the mechanisms of crack growth resistance have not been explored in detail. In this investigation the apparent fracture toughness of human enamel and the mechanisms of crack growth resistance were evaluated using the indentation fracture approach and an incremental crack growth technique. Indentation cracks were introduced on polished surfaces of enamel at selected distances from the occlusal surface. In addition, an incremental crack growth approach using compact tension specimens was used to quantify the crack growth resistance as a function of distance from the occlusal surface. There were significant differences in the apparent toughness estimated using the two approaches, which was attributed to the active crack length and corresponding scale of the toughening mechanisms.

INTRODUCTION

Enamel resides about the crown of all human teeth and is the most highly mineralized tissue of the body. It consists of approximately 96% mineral, 1% protein, and 3% water by weight. The inorganic portion is largely comprised of nanometer-scale carbonated hydroxyapatite rods (~25 nm thick, ~100 nm wide, and >>100 nm long) that systematically combine to form long ‘keyhole’ shaped structures (4–8 μm in diameter) known as ‘prisms.’ The prisms are surrounded by a ‘sheath’ of non-collagenous organic matrix and extend from the dentin enamel junction (DEJ) to the occlusal surface. In the “outer” enamel (closest to the tooth’s surface) the prisms extend in a nearly parallel arrangement from just beneath the occlusal surface (i.e., biting surface). However, within the “inner” enamel (closest to the DEJ) the prisms extend within groups or “bands” that follow a sinusoidal path where adjacent bands are obliquely oriented to one another (Figure 1b). This complexity in path results in a decussating structure.

Cracks are commonly observed on the outermost surface of teeth and particularly on the occlusal surface of molars (Figure 2a). These cracks are perceived to originate from the introduction of contact damage on the outer surface and propagate inwards, but could also initiate from internal defects known as ‘tufts’ and propagate outwards. This article addresses the fracture processes and toughness associated with crack extension from the outer surface of teeth inwards only. An examination of sectioned teeth under light microscope (Figure 2b) shows that cracks that form in vivo extend through the thickness of enamel over a millimeter in length or greater, and appear to have been arrested at the DEJ. Further examination of the morphology of natural cracks in enamel shows evidence of extrinsic mechanisms such as crack deflection and unbroken ligaments bridging the crack (Figure 2c). Such observations suggest that crack extension through enamel may undergo toughening, thereby emphasizing the importance of crack growth based evaluations for estimating the fracture toughness.

Investigations conducted to identify the fracture toughness of enamel have almost exclusively been performed using the indentation approach. Cracks introduced on the surface of enamel using indentations are typically tens of micrometers long (<100 μm) and fall within the definition of ‘short’ cracks. Consequently, the reported indentation fracture toughness ($K_{IC}$) for the weakest...
orientation (parallel to the prisms) in enamel is relatively low and ranges between 0.4 and 1.0 MPa·m$^{0.5}$. Though indentation evaluations of enamel are clinically relevant, they offer limited insight into the mechanisms associated with the crack extension process for long cracks (i.e., those operating over the thickness of enamel in human teeth). They may provide an incomplete/inaccurate measure of fracture toughness. In contrast, traditional methods used to evaluate fracture toughness are difficult to perform on enamel, particularly due to geometric constraints posed by the available material.

This article compares estimates for the fracture toughness of human enamel using the indentation technique and a more traditional incremental crack growth method using compact tension specimens that has recently been used to characterize R-curve behavior in enamel. The fundamental differences between each method in application to enamel are outlined and the results are presented and discussed with relation to the mechanisms active in resisting crack extension.

See the sidebar for materials and methods.

RESULTS

Typical cracks resulting from indentation of the enamel specimens are shown in Figure B. There was a notable variation in the nature of cracks that developed and extended from the indentation diagonals. Some indents exhibited four distinct cracks with one from each corner as shown for an indent in the outer enamel in Figure Ba, while others did not (e.g., Figure Bb and c). In general, the indentation diagonal lengths in the outer enamel were smaller than those in the inner enamel, thereby indicating a larger hardness. In contrast, the crack lengths were longer in the outer enamel than those observed in proximity of the DEJ.

The indentation fracture toughness distribution is plotted in terms of normalized distance from the tooth’s surface in Figure 3. Average values for $K_C$ obtained throughout the outer and the inner enamel are listed in Table I. Surprisingly, the $K_C$ did not exhibit a distinct trend with respect to distance from the outer surface. The average indentation fracture toughness that included all measurements (within both the inner and outer enamel) was $0.92\pm0.07$ MPa·m$^{0.5}$.

Results from incremental crack growth experiments using the CT specimens are shown in Figure 4. Here the critical stress intensity required to...
continue extension is plotted as a function of normalized distance from the occlusal surface. Note that these resistance curves (i.e., R-curves) exhibit two distinct regions of behavior and are defined as the outer (closest to the tooth surface) and the inner enamel (closest to the DEJ). As evident from the behavior at the onset of growth, there was essentially no increase in the critical stress intensity with crack extension in the region closest to the occlusal surface (outer enamel). However, subsequent growth within the inner enamel required a rise in the stress intensity to continue crack extension and is evident from the marked increase beyond a normalized distance of 0.5 (Figure 4). Results obtained for the inner and outer enamel of all specimens are listed in Table II. Overall, the outer enamel exhibited the lowest crack growth resistance with average $K_I$ at initiation of $0.69\pm0.12$ MPa-m$^{0.5}$. While the responses in the innermost enamel exhibited the highest crack growth resistance, the curves do not exhibit a plateau. Therefore, the apparent fracture toughness is discerned from the highest $K_I$ at the point of instability. Using this definition the fracture toughness of enamel ranges from 1.79 MPa-m$^{0.5}$ to 2.37 MPa-m$^{0.5}$ (average = $2.06\pm0.25$ MPa-m$^{0.5}$).

An optical examination of the crack morphology resulting from indentations and incremental growth was conducted to assess the contribution of toughening mechanisms to the apparent toughness. The cracks resulting from incremental growth showed features that were spatially unique (Figure 5a) along the length of extension. In the outer enamel the cracks extended over a relatively straight path as defined by the orientation of the enamel prisms. In contrast, the crack path was far more tortuous in the inner enamel, and revealed that extension was influenced...

### MATERIALS AND METHODS

Caries-free human third molars of individuals aged between 17 and 25 years were obtained from participating clinics in the state of Maryland according to an approved protocol issued by the Institutional Review Board of the University of Maryland Baltimore County. Sections of cuspal enamel (Figure Aa) were obtained using a slicer/grinder (K.O. Lee Model S3818EL) either in the form of beams (N=10, two from each tooth) or cubes (2x2x2 mm$^3$, N=5, one from each tooth) as shown in Figure Aa.

Indentation fracture specimens were prepared by mounting the beams in epoxy resin with prisms oriented nominally perpendicular to the polished surface (Figure Ab). Inset compact tension (CT) specimens were obtained by embodying the enamel cubes within a resin composite (Vit-l-escence, Ultradent Products, Inc.) with overall dimensions of 8x6x2 mm$^3$ (Figure Ac). Further details of this specimen configuration and preparation are presented elsewhere. The inset was placed in the CT specimen body such that the direction of crack growth was oriented along the axis of the enamel prisms, which is consistent with the orientation of natural cracks observed in vivo. Cracks were grown from the “outer” region (nearest the occlusal surface) towards the “inner” enamel. Additional features in the inset CT specimens included a 1 mm wide back channel to guide the direction of crack extension, a chevron notch machined using a razor blade and diamond paste (1 μm particles) to facilitate crack initiation and two counter bored holes for application of mode I loads.

**Indentation Fracture**

Surfaces of the indentation specimens containing the exposed enamel were polished using silicon carbide abrasive paper with successively smaller particle sizes, and then finished using diamond particle suspensions (Buehler) of sizes 9, 3, and 0.04 μm. Indents (N=5) were made on the polished surfaces of enamel using a Leitz Miniload II Microhardness Tester with 3 N indentation load. The magnitude of loading was chosen according to results of a previous study. Cracks extending from the indentation were measured using a Leica model DMRM compound optical microscope (Leica Microsystems Inc.). The crack length was estimated from the average of all four cracks emanating from the diagonals (Figure Ba). Some of the indentations exhibited a complex crack network (Figure Bb, c) rather than a single crack extending from each of the four indentation corners. There is no published standard for defining the crack length from indentations with these features and/or rejecting indentations with such cracks. Here the indents exhibiting complex crack patterns (as in Figure Bb and c) were excluded from the indentation fracture toughness measurements. After the crack length measurements the surface of each specimen was polished to remove approximately 200 μm of material. Then an indentation was performed and the crack lengths were measured. This process was repeated until reaching the DEJ. Previous observations of cracks in enamel showed that they exhibited a Palmqvist configuration and, therefore, the crack lengths were measured from the indentation corners to the crack tip. For Palmqvist cracks where the ratio of crack length (c) to indent diagonal (L) is small (0.125≤c/L≤1.25) the indentation fracture toughness ($K_{Ic}$) can be estimated for each indentation according to Equation 1, where $E$ and $HV$ are the elastic modulus and the transition point hardness, respectively, and $P$, $L$, and $c$ are the indentation load (kg), average diagonal length (m), and crack length (m), respectively. (All equations are shown in the table.)

![Figure A. Schematic diagram of specimen configurations.](image) (a) A schematic of a molar sectioned bucco-lingually depicting the microindentation beam section and the inset for the CT specimen. The arrow indicates the direction of crack growth in the inset of the CT specimen. (b) Microindentation specimen. The cuspal beams were mounted in cold-cure epoxy such that the occlusal surface faced outward and the enamel prisms were oriented perpendicular to the potting surface. (c) The inset CT specimen. The inset tissue is approximately 2x2x2 mm$^3$. The tissue is embodied within a dental composite resin.
by a number of extrinsic toughening mechanisms. Evidence of crack bridging was apparent by the presence of unbroken ligaments of tissue as highlighted in Figure 5a; the ligaments were tens of micrometers in thickness and consisted of bundles of prisms. Crack bifurcation and crack deflections were also evident and continuously evolved within the inner enamel over the path of extension. These growth characteristics were triggered by the complex meshing of prisms within the decussated region. Such deflections promote energy dissipation by reduction of the local stress intensity at the crack tip, which in turn required higher driving forces to continue crack propagation.

A closer examination of the cracks resulting from indentations revealed that extrinsic toughening mechanisms such as crack-curving (Figure 5b) and crack bridging (Figure 5c) were also active, but on a much smaller scale than in incremental crack growth achieved using the CT specimens. Crack-curving was evident from the periodic waviness in the crack path as it extended about individual prism boundaries (Figure 5b). In fact, regardless of the measurement approach (indentation or incremental fracture) the crack primarily traversed through the protein rich regions between adjacent teeth. Bridging by unbroken ligaments of prisms was also apparent in the indentation cracks, but the size of the ligaments was limited to a few micrometers in thickness, substantially less than that in cracks resulting from incremental crack growth.

**DISCUSSION**

The apparent fracture toughness of enamel determined using the indentation approach (0.92 MPa·m$^{0.5}$) was less than half that determined for the innermost enamel using the incremental crack growth measurements achieved measures of the indentation behavior of enamel showed that the transition point hardness (where hardness becomes independent of load) was achieved for indentation loads of 2 N and greater.\textsuperscript{7} Note that the values of $K_{C(IF)}$ reported in the literature for enamel and other materials have been alternatively described as the indentation fracture resistance and the fracture toughness. While the former is most technically correct, here the values will be regarded as the indentation fracture toughness for convenience and to aid in the comparison with the incremental crack growth measures.

**Incremental Crack Growth**

The inset CT specimens were subjected to fatigue loading to initiation precracks (~0.3 mm long) from the prepared notch for an initial crack length of approximately 2 mm from the load line (Figure Ac). The precracked samples were then subjected to quasi-static loading in 1 N increment until the onset of crack extension. Stable crack growth was achieved using 0.5 N load increments, followed by a dwell at each load and then followed by partial unloading and reloading. Digital images were acquired during each stage to identify the displacement field and crack lengths using micro Digital Image Correlation (DIC). In short, the crack opening displacement (COD) distributions were used to precisely identify the crack tip from the location of zero opening displacement. A detailed description of the DIC process is given elsewhere.\textsuperscript{11} The opening mode stress intensity ($K_I$) distribution with crack extension was calculated according to Equation 2$^4$ where $P$ is the opening load (N), $\alpha$ is the ratio of a to W, B (mm) is the specimen thickness, and $B^*$ (mm) is the specimen thickness adjacent to the back channel as illustrated in Figure Ac.

Results obtained from both indentation and crack growth measurements were normalized by dividing the distance of the measured indent (or the crack tip position from the occlusal surface) by the total thickness of enamel from the occlusal surface to the DEJ. The normalization allowed the property distributions to be described over a distance from 0 (near the tooth’s surface) to 1 (near the DEJ), regardless of differences in the enamel thickness between teeth. Statistical differences were evaluated using the Students t-test and significance was identified by $p<0.05$.

---

**Equations**

\begin{align}
K_{C(IF)} &= 0.008225 \left( \frac{E}{HV} \right)^{2/5} \left( \frac{2P}{L} \right)^{1/5} \left( \frac{1}{c} \right)^{1/5} \tag{1}
\end{align}

\begin{align}
K_I &= \frac{P}{B\sqrt{W}} \left[ \frac{B^2 + 1}{B + 1} \left( 1.69 - 8.01\alpha + 12.53\alpha^2 \right) \right] \text{[MPa·m$^{1/2}$]} \tag{2}
\end{align}

\begin{align}
\Delta K_{C(IF)} = K_g \cdot \Delta a \tag{3}
\end{align}

---

![Figure B. Crack morphology about selected Vickers indents in the enamel specimens. (a) A typical indentation and crack pattern with well-defined cracks at each of the indentation corners. This indent was obtained in the outer enamel. (b) Indents near the DEJ resulting in microcracks but no distinct cracks from the indentation corners. (c) A complex crack network observed for an indent placed near the intersection of the outer and inner enamel. Note that the indentation patterns in (b) and (c) were not included in estimating the $K_{C(IF)}$.](image-url)
evident about the indentations closest to the DEJ (Figure Bb), which prevented estimates of the toughness within 50 μm of the DEJ. Imbeni et al.\textsuperscript{16} placed indents within 20 to 50 μm of the DEJ but only reported toughness values for those indents farther than 200 μm, indicating similar difficulties. The drastic differences in crack patterns observed with distance from the occlusal surface (Figure B) signify that there are spatial variations in the mechanisms of toughening, and the relative contributions of these mechanisms to the estimated toughness. Nevertheless, those mechanistic aspects of the response are not reflected in the indentation fracture toughness distribution (Figure 3). Despite complications in measuring indentation crack lengths, the $K_{c(IF)}$ measurements listed in Table I are consistent with previous reported values (0.4–1.0 MPa-m$^{0.5}$) for human enamel.\textsuperscript{4,5}

One of the primary differences between the indentation and incremental crack growth responses is the extent of extrinsic toughening contributing to the measurements. Both measurement approaches exhibited influence from crack deflection and crack bifurcation (Figures B and 5), as well as contributions by unbroken ligaments of tissue. Posterior bridging of the cracks was most extensive during incremental crack growth, both in terms of the ligaments size and the scale of bridging zone operating behind the crack tip (up to a millimeter in length). Cracks extending from the indentations also underwent bridging (e.g., Figure 5c). However, the physical size of the bridging elements was limited to the scale of the microstructure. Also, the bridging zone (50 μm) was limited to approximately ten times the order of the microstructure. Thus, the $K_{cub}$ measurements for enamel are influenced by contributions from a posterior process zone, but they are restricted to those that develop over a comparatively short crack length.

A first-order estimate of the contribution from extrinsic mechanisms to the indentation toughness measurements can be obtained using the crack growth toughness from the incremental crack growth technique. The rise in indentation toughness with crack growth is estimated by Equation 3, where $K_g$ is the average crack growth toughness (2.62±1.39 MPa-m$^{0.5}$/mm) as derived from the incremental crack growth approach\textsuperscript{8} and $\Delta$ is the average indentation crack length. The apparent contribution of such mechanisms for typical indentation crack lengths (~50 μm; Figure Ba) is approximately 0.13±0.07 MPa-m$^{0.5}$ and represents just over 15% of the average indentation toughness. Interestingly, this value is close to a direct estimate of the apparent contribution from these toughening mechanisms, which is obtained by subtracting the initiation

### Table I. Results from the Indentation Fracture Resistance Measurements for the Outer and Inner Enamel Obtained using the Average Crack Length from All Four Indentation Corners

<table>
<thead>
<tr>
<th>Age</th>
<th>Outer Enamel</th>
<th>Inner Enamel</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>1.02</td>
<td>0.88</td>
</tr>
<tr>
<td>20</td>
<td>0.95</td>
<td>0.85</td>
</tr>
<tr>
<td>21</td>
<td>0.87</td>
<td>0.80</td>
</tr>
<tr>
<td>21</td>
<td>0.98</td>
<td>0.96</td>
</tr>
<tr>
<td>25</td>
<td>0.97</td>
<td>0.87</td>
</tr>
<tr>
<td>Avg = 21±3</td>
<td>0.96±0.06</td>
<td>0.87±0.06</td>
</tr>
</tbody>
</table>
toughness obtained using the CT specimens (0.69±0.12 MPa-m^1/2) from the average indentation fracture toughness for the inner enamel (0.87±0.06 MPa-m^1/2); the difference is ~0.18±0.18 MPa-m^1/2, indicating approximately 20% growth in crack growth resistance attributable to the extrinsic mechanisms. These contributions are substantially less than those developed over the “long” crack extension achieved with the CT specimens. In that approach the crack undergoes more than 300% increase in resistance to extension from initiation to fracture. Therefore, when applied to enamel the indentation-based method appears to quantify the “initiation” behavior at best (i.e., at the onset of extension), but is unable to account for the substantial contributions of extrinsic toughening mechanisms to the fracture toughness.

There is some concern with the measurements of fracture toughness obtained from the CT specimens as well. The R-curves (Figure 4) did not reach a plateau, indicating that a steady-state resistance was not obtained. For a material with rising R-curve behavior the plateau toughness is sometimes regarded as a measure of the “true” fracture toughness. But that may be impossible to achieve with the extent of tissue available from the human tooth; cracks were grown across nearly the entire enamel thickness in sections with the largest available dimensions. Also, in light of nature’s design to protect the structural integrity of the tooth it can be argued that in vivo cracks extending from the outer surface inwards should exhibit a continuous rise in toughness to protect the underlying less mineralized dentin. Therefore, although not the “true” fracture toughness, the toughness estimated using the CT specimens should be most representative of the degree of crack growth resistance operating in vivo for crack extension in enamel. There may be concern that the new specimen geometry caused the unique crack growth resistance curves for enamel. However, a validation of the specimen geometry has been performed and distinguished that the inset CT specimen can provide reliable measures of the crack growth resistance of materials over the range of permissible crack lengths.

While the indentation fracture approach does not characterize the fracture toughness of enamel, it does provide some useful information pertinent to contact induced cracking that may be clinically relevant and is not achieved from the incremental crack growth responses. It should be borne in mind that indentation cracks arise from sharp contacts, whereas clinically induced cracks are more likely to occur from blunt contacts. Apart from surface cracking, subsurface damage develops in the form of inelastic deformation (Figure 6) caused by inter-rod shearing within the protein rich region between the prisms and between the apatite crystallites. Inelastic deformation is particularly dominant in the regions of higher protein content near the DEJ. There the indentation energy is exhausted in the development of “microcracks” or separation of the individual prisms about their boundaries as evident in Figure Bb. Contact cracks resulting from the sharp indentations had a Palmqvist configuration. That may have some physical significance. According to early work on ceramic materials the indentation stress field can be divided into elastic and residual components; the reversible strain energy associated with the elastic field enhances median crack extension during the loading cycle and the irreversible plastic field primarily provides the driving force for radial crack extension in the unloading stage. In indentation of enamel there is no median crack extension’ suggesting that either the reversible elastic field is small or is suppressed by the microstructural damage in enamel. In support of the later, the inelastic damage in the compressive-shear field would suppress any subsurface tensile stress.

<table>
<thead>
<tr>
<th>Age (yrs)</th>
<th>Outer Enamel (K_I)</th>
<th>Inner Enamel (K_C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>17</td>
<td>0.65</td>
<td>2.37</td>
</tr>
<tr>
<td>18</td>
<td>0.52</td>
<td>2.27</td>
</tr>
<tr>
<td>18</td>
<td>0.66</td>
<td>1.79</td>
</tr>
<tr>
<td>20</td>
<td>0.77</td>
<td>1.95</td>
</tr>
<tr>
<td>22</td>
<td>0.83</td>
<td>1.91</td>
</tr>
<tr>
<td>Avg ±2</td>
<td>0.69±0.12</td>
<td>2.06±0.25</td>
</tr>
</tbody>
</table>

Table II. Results from the Crack Growth Resistance Measurements Obtained for Outer and Inner Enamel using Incremental Crack Extension

Figure 5. Crack morphology resulting from incremental crack growth and the indentations. (a) Incremental crack extension in the outer enamel occurred along a straight path followed by growth in the inner enamel that occurred over a tortuous path. Crack growth in the inner enamel was accompanied by a number of mechanisms including crack bridging, crack bifurcation and crack deflection. (b) Indentation cracks also exhibited extrinsic mechanisms such as crack curving along the interprismatic boundaries, and (c) crack bridging at smaller length scales. (b) and (c) are magnified views of the corners of the indent shown in Figure Ba.
that may result in formation of median cracks. As such, the residual indentation on the surface is left with a Palmqvist (surface) crack and a residual subsurface damaged zone (Figure 6). This interpretation appears appropriate for low indentation loads such as the ones used in this study. Lee et al.22 have recently shown that for higher loads (>100 N) enamel undergoes development of median cracks beneath the indentation and within the inelastic zone, similar to that observed in ceramic materials.23 However, it is important to note that these so called “median” cracks in enamel could be cracks generated from “axial splitting” due to compliance mismatch between the indenter and the enamel a phenomenon observed in compression testing of ceramics.24 Regardless, such contact induced cracks could then potentially undergo incremental extension under the forces of mastication through the thickness of enamel.

From a mechanistic perspective it seems that both the indentation approach and incremental crack growth based evaluations hold value for evaluating fracture of enamel and in studying the contribution of fracture processes to the toughness. Since the indentation technique does not quantify the fracture toughness and is relatively insensitive to spatial variations in contributions of extrinsic mechanisms of toughening, it cannot reliably be used to quantitatively evaluate the toughness. However, it does appear to have some qualitative value in the interpretation of scale dependent mechanisms of the crack growth resistance. Results presented here demonstrate that the incremental crack growth technique captures the nature of toughening over the length of extension occurring in teeth in vivo and uncovers the important contributions from microstructural variations on the fracture behavior of the inner and the outer enamel.

CONCLUSION

This study evaluated both the indentation fracture approach and an incremental crack growth technique using CT specimens. Despite marked spatial variations in the microstructure of enamel, the estimated toughness from indentations was independent of distance from the tooth’s surface. However, the more traditional technique using CT specimens distinguished that there are spatial differences in the toughness of enamel and that this tissue undergoes a significant rise in toughness with crack extension. Overall the average fracture toughness (Kc = 2.06 MPa-m0.5) measured in the innermost enamel was more than twice that estimated from indentations (Kc(IMP) = 0.92 MPa-m0.5). The largest rise in crack growth resistance occurred in the inner enamel (with resistance increasing in proximity of the DEJ), and was attributed to a concert of extrinsic mechanisms of toughening and the sustained contribution of unbroken ligamens of tissue operating over relatively large crack lengths.

ACKNOWLEDGEMENTS

The authors acknowledge support from the National Institutes of Health (NIDCR DE016904) and (NIDCR DE017983) and the National Science Foundation (BES 0521467). The authors would also like to thank Ultradent Products, Inc. for supplying the Vit-l-escence resin composite and Dr. Judith Porter of the University of Maryland, Baltimore for help with bonding practice.

References