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An Inset CT Specimen for Evaluating Fracture in Small Samples of Material

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Abstract

In evaluations on the fracture behavior of hard tissues and many biomaterials, the volume of material available to study is not always sufficient to apply a standard method of practice. In the present study an inset Compact Tension (inset CT) specimen is described, which uses a small cube of material (approximately $2 \times 2 \times 2 \text{ mm}^3$) that is molded within a secondary material to form the compact tension geometry. A generalized equation describing the Mode I stress intensity was developed for the specimen using the solutions from a finite element model that was defined over permissible crack lengths, variations in specimen geometry, and a range in elastic properties of the inset and mold materials. A validation of the generalized equation was performed using estimates for the fracture toughness of a commercial dental composite via the “inset CT” specimen and the standard geometry defined by ASTM E399. Results showed that the average fracture toughness obtained from the new specimen ($1.23 \pm 0.02 \text{ MPa}\cdot\text{m}^{0.5}$) was within 2% of that from the standard. Applications of the inset CT specimen are presented for experimental evaluations on the crack growth resistance of dental enamel and root dentin, including their fracture resistance curves. Potential errors in adopting this specimen are then discussed, including the effects of debonding between the inset and molding material on the estimated stress intensity distribution. Results of the investigation show that the inset CT specimen offers a viable approach for studying the fracture behavior of small volumes of structural materials.

Keywords

crack propagation; bone; dentin; enamel; fracture toughness

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1. INTRODUCTION

The fracture toughness and fatigue crack growth resistance of materials are of critical importance in many engineering applications. They are also relevant measures of performance in the fields of medicine and dentistry. For example, the selection of materials used for replacing hard tissues of the tooth is often based on their resistance to the initiation and growth of cracks (e.g. Guazzato et al., 2004a; 2004b; Ruse, 2007; Shah et al., 2009a; 2009b). In addition, the crack growth resistance behavior of natural materials and mineralized tissues is presently inspiring the design and development of novel engineered microstructures (e.g. Deville et al., 2006; Munch et al., 2008; Barthelat, 2010; Launey et al., 2010). That has increased the need for evaluating the fracture behavior of samples prepared from small volumes of material.

The fracture behavior of engineered materials is frequently evaluated according to standard ASTM E1820 of the American Society of Testing and Materials (2006). For metals and ceramics, the fracture behaviors are conducted according to ASTM E399 (2006) and ASTM C1421 (2006), respectively. Similarly, the fatigue crack growth properties of engineered materials are generally characterized according to ASTM E647 (2006) and also frequently involve the compact tension (CT) configuration. As the existing standards were developed for evaluating engineered materials, they have adopted specimen sizes that are easy to fabricate and test. However, when the available volume of material is small, as is found in the evaluation of many natural materials, it is impossible to achieve the specimen geometry defined by the standard.

Indentation techniques have been considered to offer an alternative to the conventional approaches for evaluating the fracture toughness of brittle materials (Lawn et al., 1980; Antis et al., 1981; Fett, 2002; Fett et al., 2004). The indentation techniques have some attractive features, e.g. they are easier to apply than the standardized methods, require limited specimen preparation and often require only a short time to conduct. In addition, they require a very small volume of material. Yet, there are significant concerns regarding their use in estimating fracture toughness (Morrell 2006; Munz 2007; Quinn and Bradt, 2007; Kruzic et al., 2009). One consideration is that there are over 30 different analytical models for estimating the fracture toughness from indentation cracks (Ponton and Rawlings, 1989; Quinn and Bradt, 2007), and many of these have been developed from curve fits to data rather than from physical models for fracture (Quinn and Bradt, 2007). Even when comparing two highly brittle materials of nearly identical composition, the indentation fracture test has been shown to give highly misleading results when compared to other methods (Eilertsen et al, 2013). Some consider indentation techniques unsuitable for quantifying the fracture toughness of materials (Quinn and Bradt, 2007, Kruzic et al., 2009).

Miniaturized specimens have often been considered as an alternative for measuring the fracture properties of biomaterials and hard tissues. There are various configurations that have been adopted or developed for this purpose. For example, the use of flexural loading of beams with various dimensions has been commonly employed to measure the fracture toughness of natural materials such as nacre (e.g. Barthelat and Espinosa, 2007; Rabiei et al., 2010) and bone (e.g. Nalla et al., 2003; Ritchie et al., 2008; Koester et al., 2008). Currey

(1976) adopted a composite flexural beam configuration consisting of a portion of nacre placed in the middle of two opposing aluminum rectangular bars on the sides to measure the work to fracture. Wang et al. (1994) developed a compact sandwich specimen for evaluating the fracture toughness of bone and prosthetic materials, which has been adopted to evaluate the fracture properties of dental materials as well (e.g; Paruchuru et al., 2002; Parachuru and Jian, 2009; De Souza et al., 2011). More recently, asymmetric beams and Double Cleavage Drill Compression (DCDC) specimens were adopted for conducting mixed-mode fracture evaluations in bone (Zimmermann et al., 2009; Olvera et al., 2012).

Regarding dental materials, Pilliar et al., (1986; 1987a; 1987b) adopted a short rod specimen for determining the fracture toughness of selected materials and later used this approach to evaluate the fracture toughness of bonded interfaces (Tam and Pilliar, 1993). Ruse et al. (1996) developed a novel notchless triangular prism (NTP) specimen for evaluating the fracture toughness of dental materials, adhesive interfaces and hard tissues (Dong and Ruse, 2003; Far and Ruse, 2003; Iwamoto and Ruse, 2003). Furthermore, different configurations of flexural beams including three-point, four-point bending, single and double notch beams (e.g. Imbeni et al., 2003; Nalla et al., 2003a; Kinney et al., 2005; Bechtle et al., 2010a) have been adopted for estimating the fracture properties of dental hard tissues.

The aforementioned techniques utilize a specimen with major dimension of 1–4 mm, which is still quite large for many potential applications. A miniature “inset” CT specimen was proposed by Zhang et al. (2007) for evaluating the crack growth resistance of hard tissues. This specimen utilizes a small piece of tissue, with major dimension of two millimeters, where the material of interest is embedded or molded within another material. The inset CT specimen presently requires the smallest volume of material that has been reported for evaluating fracture behavior using a conventional crack growth approach. However, the drawback of this specimen is that the stress intensity distribution has been defined for applications to studies on dentin and enamel only, and it has not been validated.

The primary objective of this investigation was to facilitate application of the inset CT specimen to investigations concerning the fracture behavior of materials where volume constraints are a limiting factor. A generalized equation was developed and validated for the stress intensity caused by Mode I loading of the inset CT specimen over an expanded description of the specimen geometry and a large range in elastic moduli of the inset and molding materials. Applications of the specimen to studies on the fracture resistance of hard tissues are presented and important considerations regarding the experimental process are discussed.

2. MODELING

The inset CT specimen geometry is illustrated in Figure 1(a). A detailed description of the methods used for preparing these specimens and the experimental procedures for conducting experiments with cyclic and monotonic loading will be discussed later in this manuscript. In short, the specimen design involves a volume of material of approximately $2 \times 2 \times 2 \text{ mm}^3$ that is inset within a secondary material using a molding technique. The interface between the two materials is prepared to achieve a strong adhesive bond using applicable approaches for

the materials involved. Two holes are introduced within the molding material for application of opening mode loading and a channel is fabricated on the back side of the specimen. The channel serves to reduce the required opening mode load and consequent stress across the bonded interface (achieved by the larger bonding area) while achieving the desired stress intensity (Zhang et al., 2007).

2.1 Finite Element Model

A three-dimensional finite element model was developed to quantify the stress intensity generated by opening mode loading and presence of a crack within the inset material. The model was defined according to the specimen configuration in Figure 1(a) using the commercial software (ABAQUS, version 6.7-3). The model consists of approximately 130,000 nodes and 29,000 20-noded brick elements (C3D20R) with reduced integration points (Figure 1(b)). The model was defined parametrically as a function of crack length (a) and opening mode load (P), dimensions of the specimen (W , B and B^*) and the elastic modulus and Poisson's ratio of the inset material (E_1 , ν_1) and molding material (E_2 , ν_2). Mechanical properties for both the inset and molding material were defined as homogenous and isotropic. Comments regarding these definitions will be addressed in application of the specimen to selected materials later in this manuscript. Simulations were conducted with the inset elastic modulus (E_1) ranging from 10 to 100 GPa, and with the ratio of moduli (E_1/E_2) ranging over 0.5 – 14. The model geometry was defined for a range of specimen thickness ($1.6 \text{ mm} \leq B \leq 2 \text{ mm}$) and ligament thickness ($0.6 \text{ mm} \leq B^* \leq 1 \text{ mm}$) to account for variations in the preparation and tolerances. The stress intensity distribution was evaluated for crack lengths within the inset material of $0.1 \text{ mm} \leq a \leq 1.9 \text{ mm}$, which corresponded to total crack lengths from the load-line of $1.9 \text{ mm} \leq a \leq 3.7 \text{ mm}$. The bonded interface was assumed to be infinitesimally thin and the nodes between the inset and resin composite material were tied, which assumes perfectly bonded. The effects of interfacial debonding on the stress intensity distribution were also considered in which portions of the interface were purposely treated as not bonded and the changes in stress intensity were quantified.

The J-integral method was used to estimate the energy release rate according to standard methods (Anderson, 2005). In the present application, the analysis was conducted within a series of unique planes (minimum 15) that were parallel to the front surface of the specimen (and crack growth direction) and distributed essentially in an equidistant manner through the thickness. Within each plane several contours were evaluated to ensure the path-independence, and that the J-integral calculations were carried out at sufficient distance from the crack tip to satisfy the far-field value; each contour included a number of elements distributed around the crack tip. A sensitivity analysis performed on the contour integral evaluation showed that increasing the number of contours to 6, resulted in a very slight increase (less than 0.004%) in the estimated stress intensity factor (from the energy release rate).

A total of well over five hundred simulations were performed over the parametric space and the stress intensity resulting from each set of conditions was documented. The Mode I stress intensity (K_I) was estimated from the J-integral according to (Irwin, 1957)

$$K_I = \left[J \bullet E' \right]^{\frac{1}{2}} \quad (1)$$

where $E' = \frac{E}{1 - \nu^2}$, which assumes that the specimen dimensions satisfy requirements of the plane strain conditions¹ (ASTM E399). Achieving this condition requires that both the ligament thickness (B^*) and crack length (a) exceed the necessary dimensions as established by the properties of the inset material. It is important to comment that the specimen size requirements defined by ASTM E399 for the thickness and crack length are very strict; they are essential to achieve a measure of fracture toughness that is obtained under a state of plane strain and independent of specimen thickness. This dimensional constraint should be applied when adopting the inset specimen to insure that the size of the plastic zone is much smaller than the specimen thickness. If the material to be evaluated is brittle (i.e. does not undergo appreciable inelastic deformation), specimens with smaller thickness can be adopted [Anderson, 2005].

2.2 Stress Intensity Distribution

A nonlinear regression analysis was performed using commercial software (MATLAB, R2010b, Mathworks Inc., Massachusetts, USA) to derive a generalized equation for the Mode I stress intensity as a function of the specimen geometry, elastic properties and opening load. The model was constructed by fitting the solutions of the finite element model and corresponding K_I distribution over the parametric space and is given by

$$K_I = \left(\frac{P}{B^* \sqrt{W}} \right) \left[\frac{E_1 (1 - \nu_2^2)}{E_2 (1 - \nu_1^2)} \right]^\lambda \sqrt{\frac{B^* + 1}{B + 1}} (0.41 - 1.57 \alpha + 2.85 \alpha^2) \quad (2)$$

where the quantities a , B , B^* and W are defined in Figure 1(a), $\alpha = a/W$ and the term λ is given by

$$\lambda = 0.04 + 0.91 \alpha \quad (3)$$

The model defined by Eqn. 2 is valid for crack lengths within the inset material (a') of 0.1 mm < a' < 1.9 mm, specimen thickness 1.6 mm $\leq B \leq$ 2 mm, ligament thickness 0.6 mm $\leq B^* \leq$ 1 mm and W of 6.8 mm.

3. EXPERIMENTAL METHODS

3.1 Validation of the Inset CT Specimen

To validate the generalized equation for the inset CT specimen it was chosen to compare estimates of the fracture toughness for a selected material obtained using the inset CT

¹According to the ASTM E399, plane strain conditions are met when the thickness (B) and crack length (a) are $B \geq 2.5 \left[\frac{K_{Ic}}{S_y} \right]^2$, where S_y represents the yield strength and K_{Ic} is the plane strain fracture toughness. For the inset CT specimen, the relevant thickness is the ligament thickness (i.e. $B=B^*$).

specimen (Eqn. 3) and using standard sized compact tension specimens and the associated equations for the stress intensity factor defined by ASTM E399 and E1820. There were various concerns that guided the selection of the material used in this study. For instance, many biological materials of interest exhibit rising crack growth resistance (R-curve) behavior and the inset material should have a small cohesive zone, i.e. less than the inset size, so that the crack growth resistance curve reaches a plateau in less than 2 mm (size of the cube). Specific to the inset CT specimen, the inset and molding materials should have adequate bonding compatibility such that bonding would not interfere with the results. A commercial resin composite used in restorative dentistry (Filtek Z250) was chosen for this purpose. Shah et al. (2009a) evaluated the fracture resistance behavior of this material using a standard CT specimen with a width (W) of 14 mm, a thickness (B) of 2 ± 0.5 mm, and an initial notch length (a) of 3.5 mm. Results for the Filtek Z250 from that study showed a rising fracture resistance (i.e. R-curve) that reached plateau well within 2 mm of crack growth (Shah et al., 2009a; 2009b).

Fractured pieces of the Filtek Z250 specimens tested in the study of Shah et al. (2009) were used in this study. Cubes of the material with dimensions of $2 \times 2 \times 2$ mm³ were sectioned using abrasive slicing equipment (Chevalier model SMART-H81811, Taiwan) and diamond abrasive slicing wheels (#320 mesh abrasives). The four surfaces of the cube to be bonded were sanded lightly using #150 mesh abrasive paper, and then rinsed using deionized water and dried with air. Then an adhesive resin (Peak® LC, Ultradent Products Inc., South Jordan, UT, USA) was applied to the prepared surfaces of the inset using a micro brush applicator according to the manufacturers instructions. The adhesive was cured for 10 seconds using the Ultradent Ultra-Lume ® LED 5 with the light output of 800 MW/cm². The inset was then placed precisely at the center of an aluminum mold having dimensions of 6×8 mm. A commercial resin composite (Vit-l-escence, Ultra dent Products, Inc., South Jordan, UT, USA) was introduced to fill the surrounding regions of the mold and cured for 30 seconds using the same curing light device. The exposed surfaces of each specimen were polished within the mold using #800 mesh abrasive paper to remove any surface irregularities, and then released by lightly tapping on the mold. A back channel (0.5 mm wide) and 1.0 mm depth slot were introduced within the molded specimens (according to Figure 1(a)) using the aforementioned slicing machine. The two holes were introduced using a miniature milling machine. A sharp vee-shaped notch was prepared at the sawn notch root using a razor blade and diamond paste with gentle transverse sawing motion.

The elastic modulus of resin composites is known to increase with shrinkage and age in the dry condition (Kleverlaan and Feilzer, 2005; Helvatjoglu-Antoniades et al., 2006), making it necessary to determine the elastic modulus of the Filtek Z250. Therefore, the elastic modulus of the material was evaluated in four-point flexure using beam specimens prepared from the original fractured CT specimens. Results of that evaluation showed that the elastic modulus was 14 GPa, which is nearly 1.3 times higher than that reported by the manufacturer (11 GPa). The molding material has an elastic modulus of 6 GPa. For both of the inset and molding materials similar Poisson's ratio of 0.33 was considered (Chung et al., 2004; Papadogiannis et al., 2008).

The prepared specimens were subjected to cyclic loading to generate a sharp pre-crack (~200 μm) from the razor-sharpened notch using a Bose Model ELF 3100 (Bose, Eden Prairie, MN, USA) universal testing machine. Cyclic loading was conducted using a frequency of 5 Hz and stress ratio (R) of 0.1 after methods used in prior studies (Zhang et al., 2007). Crack initiation was monitored using a digital microscope (Optem zoom 70 $\times$ 1 391940, QIOPTIQ, Luxembourg). Thereafter, quasi-static loading of the specimens was performed using a dedicated universal testing system complemented with the aforementioned microscopic imaging system. Loading was conducted with 0.5 N increments until the onset of crack extension. Crack growth was then performed incrementally using 0.5 N load increments or smaller, until the point of instability. Both the cyclic loading and incremental crack growth testing were conducted without hydration. Digital images were acquired during each loading step to identify the displacement field and crack lengths using micro Digital Image Correlation (DIC). A detailed description of the DIC process and application is given elsewhere (Zhang et al., 2007). In short, the Crack opening Displacement (COD) distributions were used to identify the crack tip from the site of zero opening displacement. A total of N=5 specimens were prepared and tested.

The bonded interface of the inset CT specimens within the finite element model was defined as infinitesimally thin and perfectly bonded. Thus, in addition to experimentally validating Eqn. 1 it was important to investigate potential contributions from the bonded interface of the inset CT specimen on the fracture behavior. Towards addressing this concern, CT specimens were prepared from the Vit-l-escence resin composite (i.e. the molding material) with and without bonded interfaces. Specifically, the inset CT specimens were prepared from the resin composite with identical inset and molding materials. The inset material was prepared as a 2 mm thick section, light cured as previously described, and then sectioned to produce the 2 $\times$ 2 $\times$ 2 mm³ cubes. Adhesive resin was applied to the intended bonding surfaces according to the manufacturers recommendations, and the insets were molded within an exterior of the same material. A total of five inset CT specimens of Vit-l-escence were prepared, and an additional five conventional CT specimens were prepared from solid Vit-l-escence without an interface. These additional specimens were prepared with geometry described in Figure 1, but were molded as a contiguous specimen. Both groups of specimens were subjected to the same experiments and identical methodology as used in evaluating the fracture behavior of the Filtek Z250.

3.2 Applications to Hard Tissues

Additional experiments were performed on human occlusal enamel and radicular dentin to provide some example applications of the inset CT specimen. Although these are both hard tissue, they possess substantially different composition and microstructure.

Enamel is the outermost hard tissue of the human tooth and serves as a protective enclosure for the underlying tissues. It varies in thickness about the tooth with a maximum of near 2.5 mm on the occlusal surface. Tooth enamel is nearly 96% mineral by weight, with the remainder consisting of 1% organic material and 3% water (Ten Cate, 2008). The mineralized material consists of carbonated hydroxyapatite crystals that systematically combine to form long 'keyhole' shaped rods (4–8 μm in diameter) (Robinson, 1995). The

micro-scale enamel rods are arranged in a columnar fashion, extending from the dentin–enamel junction (DEJ) to the occlusal surface. Inset CT specimens were prepared using the procedures described in Section 3.1 with human enamel serving as the inset material. Briefly, sections of cuspal enamel were sectioned from about the occlusal surface of human 3rd molars with the 2×2×2 mm³ geometry. The enamel surfaces intended for bonding were etched for 10 sec using a 37% phosphoric acid gel, after which the aforementioned adhesive resin was applied and light cured. The enamel cubes were then molded within the Vit-l-escence resin composite such that the direction of the crack growth is oriented along the axis of the enamel prisms (Figure 2(a)). Additional features were then introduced to achieve the inset CT specimen geometry of Figure 1(a).

Inset CT specimens were also prepared from the dentin of human teeth, a hard tissue consisting of approximately 70% mineral, 20% organic and 10% water by weight. The microstructure of dentin is dominated by a network of tubules that extend radially from the pulp outward to the DEJ and cementum (Ten Cate, 2008). For the present study 2×2×2 mm³ sections of dentin were obtained from the radicular portion of human teeth (Figure 2(b)). Etching of the bonding surfaces was performed for 10 sec, followed by application of the adhesive resin, and then placement of the inset dentin in the mold. The sections were arranged to achieve crack propagation from the pulp outward to the cementum.

The dentin and enamel specimens were loaded cyclically to initiate a crack (~0.25 mm) from the razor-sharpened notch, followed by incremental monotonic loading. The specimen hydration was maintained during both the cyclic loading and quasi-static loading phases of testing. Cyclic loading was conducted with the samples submerged in Hanks Balanced Salt Solution (HBSS). Hydration of the samples during incremental loading was maintained using a saturated cotton swab “cradle” that was nestled beneath the specimen and retained moisture from an eyedropper of HBSS. A total of five samples of each material were tested to failure and the load and crack length measurements were used in constructing the fracture resistance curves.

RESULTS

Fracture resistance curves obtained for the Filtek Z250 using the inset CT configuration are shown in Figure 3(a). The responses obtained from experiments performed using the standard CT geometry as reported in Shah et al (2009a) are also shown in this figure for comparison. The Filtek Z250 exhibited rising R-curve behavior, and the responses obtained from the standard and the inset CT specimen geometries are consistent. The fracture resistance obtained from both configurations exhibits an initiation toughness of approximately 0.8 MPa•m^{0.5}, undergoes toughening with crack extension and reaches a plateau after approximately 1 mm of crack growth. From an assessment of the steady state responses at plateau, the average fracture toughness obtained using the inset CT specimens was 1.23 ± 0.02 MPa•m^{0.5}. That value is less than 2% greater than from the steady state toughness (1.21±0.07 MPa•m^{0.5}) reported by Shah et al (2009a). According to a statistical comparison of the results, there is no significant difference (p= 0.72) between the two estimates of fracture toughness.

The fracture resistance curves for the Vit-l-escence resin composite evaluated using the inset CT specimen and specimens without bonded interface are shown in Figure 3(b). In contrast to Filtek Z250, the Vit-l-escence showed a relatively flat R-curve response, with limited evidence of toughening. The inset CT specimens of Vit-l-escence resin composite prepared with and without bonded interface have nearly identical fracture resistance curves. The average fracture toughness obtained using the inset CT with and without bonded interfaces was $0.73 \pm 0.03 \text{ MPa} \cdot \text{m}^{0.5}$ and $0.74 \pm 0.03 \text{ MPa} \cdot \text{m}^{0.5}$, respectively. There was no significant difference ($p=0.26$) between the estimated toughness of the two groups of specimens (Figure 3b) and no evidence that the interface contributed to the fracture resistance behavior. The aforementioned values are consistent with that ($0.69 \pm 0.13 \text{ MPa} \cdot \text{m}^{0.5}$) reported in a previous evaluation by Soappman et al. (2007) using a similar CT specimen geometry.

The fracture resistance curves obtained for crack growth in human enamel are plotted in Figure 4(a). Some of the results in this figure were presented previously in the investigation reported by Bajaj and Arola, (2009b). As evident from the distribution in data, human enamel exhibits an interesting fracture behavior. Although at the initiation of crack growth the apparent toughness is relatively low, ranging between 0.50 to $0.90 \text{ MPa} \cdot \text{m}^{0.5}$, the crack growth resistance increases by nearly a factor of two over less than 2 mm of crack extension. This increase occurs as the crack propagates from the outer enamel towards the inner tooth enamel nearest the DEJ, where the enamel microstructure changes as a result of decussation. Consistent with the fracture resistance curves reported for enamel in previous studies (Bajaj and Arola, 2009b; Bechtle et al., 2010a; Bechtle et al., 2010b), they do not achieve a plateau prior to fracture of the specimen. Using the maximum value at failure to define a measure of criticality or “toughness”, the average for the enamel specimens was $2.07 \pm 0.24 \text{ MPa} \cdot \text{m}^{0.5}$.

Results obtained from the experiments with radicular dentin using the inset CT specimen are shown in Figure 4(b). To the author’s knowledge, these are the first R-curves reported for radicular dentin in the literature. Consistent with the enamel, there is a rise in the crack growth resistance with extension in all the responses obtained. It is important to note that the crack is extending from the inner dentin outward for all samples. Some reach a plateau prior to reaching instability, while others do not. The fracture resistance curves exhibit an initial resistance at the onset of crack growth ranging from 1.3 to $2.0 \text{ MPa} \cdot \text{m}^{0.5}$. Following initiation, there is an increase in the crack growth resistance of between 1 and $1.5 \text{ MPa} \cdot \text{m}^{0.5}$ over approximately 1 mm of extension. Fracture occurred at maximum value of stress intensity ranging from 2.3 to $3.5 \text{ MPa} \cdot \text{m}^{0.5}$.

DISCUSSION

A generalized equation was developed for the inset CT specimen that describes the stress intensity within the inset material for Mode I loading. The equation is defined over a selected range in specimen geometry, and accommodates a range of inset and molding materials in terms of their elastic behavior. A comparison of estimates for the fracture toughness of a commercial resin composite determined experimentally using the inset CT specimen and standard CT geometry showed that the values obtained from the two approaches were within 2%. Furthermore, the fracture resistance curves obtained from the two specimens were in close agreement. These findings establish that the inset CT specimen

can be used with confidence to quantify the fracture resistance of materials, and that it provides a viable option for investigations where the volume of available material is too small to satisfy existing standards.

In addition to evaluating the fracture behavior of selected resin composites using the inset CT specimen, fracture resistance curves obtained using the inset CT were presented for human enamel and radicular dentin. The inset CT approach has been adopted and used successfully in previously reported evaluations on the fracture behavior of human enamel (e.g. Bajaj et al., 2008; Bajaj and Arola, 2009a; Bajaj and Arola, 2009b; Yahyazadehfar et al., 2013a). Collectively these studies have distinguished that the fracture resistance of tooth enamel is both anisotropic and location dependent. Though estimates of the fracture toughness for root dentin have been reported, there have been no prior reports of fracture resistance curves for radicular dentin. In a study involving healthy human root dentin, Kinney et al., (2005) reported an average fracture toughness of $1.79 \pm 0.06 \text{ MPa}\cdot\text{m}^{0.5}$ via flexural loading of beams. Similarly, Wang (2005) compared the mechanical behavior of coronal and radicular dentin from bovine incisors and estimated that the fracture toughness of the root tissue was nearly $5 \text{ MPa}\cdot\text{m}^{0.5}$. The resistance curves in Figure 4(b) exhibited a fracture resistance reaching as high as $3.5 \text{ MPa}\cdot\text{m}^{0.5}$, which far exceeds that reported for cracks oriented perpendicular to tubule direction of coronal dentin (Nazari et al., 2009), and is comparable or greater than the fracture resistance of coronal dentin in plane with tubules (Ivancik et al., 2012). Clearly additional investigation would be required to understand the discrepancies between reported values, and other important aspects of endodontic therapy on the fracture resistance of this tissue. One issue to consider in future investigations is that the measured fracture toughness values and R-curves for crack bridging toughened materials are sample geometry dependent parameters rather than true material properties. Instead, it is thought that the bridging stress distribution, which is a cohesive zone law describing the relation of stress and crack opening displacement behind the crack tip, is a material property (Greene et al., 2012). Furthermore, the procedures for determining the bridging stress distribution from R-curves using fracture mechanics weight functions have been well documented in other publications (Fett et al., 1995; Fünfschilling et al. 2009).

There are two important concerns regarding studies on the fracture behavior of hard tissues that warrant discussion. Equation 1 is defined using a single value for the elastic modulus. Most biological materials are anisotropic. Nevertheless the strongest degree of anisotropy is in the microstructure, and not the elastic modulus. For example, previous work for both dentin and enamel show that the elastic properties are largely isotropic [Kinney et al., 1996; Braly et al., 2007]. Thus, Equation 2 was applied to study the fracture resistance of dentin and enamel using their “elastic modulus” with no limitations. For materials with a large degree of elastic anisotropy, the inset CT can be used, but Equation 2 must be used with caution. In these applications a new estimate for the stress intensity distribution should be developed according to the methodology discussed in Section 2. The second point addresses the contributions of inelastic deformation. Many natural materials exhibit undergo near-tip inelastic deformation that contributes appreciably to their fracture resistance. As such, LEFM is not able to accurately estimate the fracture toughness of these materials. There are also potential effects of various extrinsic toughening mechanisms contributing to the fracture resistance, including crack deflection, bridging ligaments, etc. Two comments are offered.

An evaluation of the fracture resistance using EPFM may be warranted and studies on bone and dentin accounting for plasticity have been reported [e.g. Yan et al., 2007; 2008]. Alternatively, a cohesive zone law may be utilized in the evaluation of fracture behavior to provide more detailed information on the contribution of various toughening mechanisms to the overall fracture resistance. In this method, the crack opening displacement (COD) or load-opening displacement from experiments is used as the benchmark solution for finite element simulations involving a cohesive zone. Detailed description of the hybrid methods and examples of their application to various biomaterials are discussed elsewhere (e.g. Yang et al., 2006; Barthelat and Espinosa, 2007; Yan et al., 2008; Ivancik and Arola, 2013). Nevertheless, the inset CT specimen can serve as a viable method supporting further studies that are aimed at evaluating fracture behavior using an EPFM analysis or to develop a cohesive zone law.

An important consideration in adopting the inset CT specimen is selecting a mold material for a specific inset material. The primary requirement is the ability to develop adequate bond strength between the molding and inset materials. For mineralized tissues including bone, dentin and enamel, dental adhesive resins are a natural choice and are relatively simple to apply. The second major consideration is the influence of the molding material elastic modulus (E_2) on the stress intensity that develops within the inset material. The influence of elastic modulus ratio of the inset and molding material (E_1/E_2) on the stress intensity distribution is shown in Figure 5 over permissible crack lengths. It is important to note that the stress intensity in this figure is normalized by the opening load magnitude, and defined over the range in permissible crack lengths in the inset (a'). As evident in this figure, small values of the elastic modulus ratio ($E_1/E_2 \approx 1$) maintain a uniform magnitude of stress intensity over the crack extension, with increase in K_I of less than 1.5 times over the permissible range in crack length. With an increase in modulus ratio to values of 10 or higher, the magnitude of stress intensity increases by a factor of at least 4. That can increase the chance for unstable crack growth, especially for larger degrees of crack extension or materials with only minimal toughening. Therefore, in application of the inset CT specimen for analyzing the fracture behavior of selected materials, it is beneficial to choose a molding material with elastic modulus that is similar in magnitude to the inset material.

Achieving adequate bond strength between the inset and molding materials is an important prerequisite for using the inset CT specimen. Those materials with low fracture toughness are better candidates for this approach, as the required opening mode load to achieve the critical stress intensity is lower. That in turn decreases the magnitude of interfacial stress and chance for debonding. Adhesive joints in bonding between hard tissues and resin composites are very sensitive to fatigue degradation (Mutluay et al. 2013; Yahyazadehfard et al., 2013b). That raises the value of increasing the notch length (a') within the inset, and rigorously sharpening the notch to help achieve crack initiation with the minimum period of cyclic loading. Despite these precautions, debonding can occur, especially in situations where strong bonds are not easily achieved. As such, it is worthwhile to comment on the effects of debonding in using the inset CT.

A displacement field map obtained from DIC for opening-model loading of a Filtek Z250 specimen that was prepared with blunt notch geometry is shown in Figure 6(a). The

specimen was subjected to larger than normal loads to promote interface debonding. Indeed, after a period of cyclic loading the displacement field reveals a discontinuity in the field between the inset and molding materials, which occurs due to debonding. The influence of debonding on the local stress intensity was evaluated using the finite element model and its importance is expressed for two different conditions in Figures 6(b) and 6(c). Debonding of up to 50% of the leading edge (from the notch face to midway between A and B) results in a reduction of the stress intensity by 12% with respect to that estimated using Eqn. 2. For debonding along the entire leading edge (from A to B), the local stress intensity is reduced by up to 20% as shown in Figure 6(b). Further extension of debonding to the peripheral edge (C to D) and occupying up to 50% of the length reduces the local stress intensity by up to 70% as shown in Figure 6(c). Interface debonding with use of the inset CT is not problematic. Nevertheless, as evident in Figure 6 debonding between the inset and molding materials can result in substantial errors in evaluations of the fracture resistance using the inset CT specimen. That is a recognized limitation of this approach, and provides motivation to seek methods and materials for adhesive bonding that are robust. It is advisable that DIC be used for crack length measurements as defects along the interface can be more easily identified through the displacement field maps. If DIC is not available, then the fracture resistance evaluated farther from the opening load load-line is less susceptible to errors as evident in Figures 6(b) and 6(c).

Biological materials possess unique mechanical properties. The collective advances in understanding of their microstructures and mechanisms of crack growth toughening are being used towards developing bio-inspired materials with exceptional performance (e.g. Deville et al., 2006; Munch et al., 2008; Launey et al., 2010). Finding answers from natural materials to support future developments in engineering materials will require novel methods of testing and a multi-scale approach (Barthelat, 2007). The inset CT specimen described herein offers one alternative to measure the fracture behavior of these materials. But despite its attractive features, there are limitations as well. The specimen was designed with a posterior back channel to increase the bonded surface area per unit crack length with respect to the fracture area. That simply reduces the chance for debonding of the interface. The holes used in application of the opening loads are counter-bored to center the opening load over the thickness of the remaining ligament, and that effectively eliminates the other two modes of fracture (Bajaj et al, 2006). However, a stress concentration develops about the channel, which results in a non-constant opening mode stress and stress intensity across the ligament thickness. Thus, the description provided by Eqn. 2 is the average stress intensity across the thickness. One could use symmetric channels on the two sides of the specimen, but that reduces visibility of the crack when using optical approaches. In addition, the current inset CT specimen geometry uses a “specimen” with major dimensions of 2 mm. This specimen configuration has potential for use on a wide range of biological materials, and with inset sizes smaller than $2 \times 2 \times 2 \text{ mm}^3$. To support this effort, work is presently underway towards development of a global equation for the inset CT specimen accommodating a range of inset sizes. Perhaps an additional drawback of the inset CT specimen is its fabrication; the process of making these specimens requires an investment of time. Nevertheless, there are limited alternatives when working at this scale. It is hoped that

this description of its application and supporting information will serve the materials community and enable future studies that would otherwise be impossible.

SUMMARY

An “inset CT” specimen was described for evaluating the fracture resistance of small volumes of material and a new generalized equation was developed to describe the Mode I stress intensity distribution that results over a wide range of inset and molding materials. A validation of the specimen geometry and equation was performed by comparing the fracture toughness estimated using the inset CT specimen to an evaluation performed using standard CT samples and stress intensity equations. Both approaches showed similar R-curve behavior and provided measures of fracture toughness within 2% agreement. Experimental applications to the dentin and enamel of human teeth show that the specimen can be used to for evaluating the fracture resistance of hard tissues as well. The choice of molding material is an important one, and there are distinct advantages to choosing one with similar elastic properties to that of the inset, and that can achieve strong and durable bonds. Overall, the inset CT specimen will enable characterizations of crack growth resistance under both static and cyclic loading, for materials that could otherwise not be examined due to the volume of material available.

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Figure 1.

The inset CT specimen. a) Schematic diagram of the specimen geometry and definition of the primary variables. The nominal dimension of the inset is cube is 2 mm. All the dimensions are in millimeters. b) The finite element model of the inset specimen. The quantities E_1, ν_1, E_2, ν_2 represent the elastic modulus and Poisson's ratio of inset and molding materials, respectively.

Figure 2.

Preparation of inset CT specimens from hard tissues for experimental evaluation of the fracture resistance behavior. a) an enamel section obtained from a 3rd molar and placement of the enamel within the resin composite. The direction of crack growth achieved is from the occlusal surface to the DEJ. b) schematic of a cube of root dentin obtained for preparation of an inset CT specimen. Note that the cube is embedded within the resin composite (as shown in (a)) to achieve crack growth from the pulp canal outward as shown by the arrow.

Figure 3.

Crack growth resistance behavior of the “calibration” materials obtained from the experiments. a) crack growth resistance behavior of the Filtek Z250 commercial resin composite using the inset CT specimen (n=7) and ASTM 399 [from Shah et al., 2009]. b) a comparison of the crack growth resistance of Vit-I-escence commercial resin composite using inset CT specimens (n=5) and CT specimens (n=5).

Figure 4.

Representative crack growth resistance curves within hard tissues of the tooth using the Inset CT specimen a) Crack growth resistance response of the human enamel for the cracks growing from occlusal surface to the DEJ [Bajaj and Arola, 2009b]. b) Crack growth resistance response for the human root dentin for the cracks growing from pulpal dentin to the cementum (n=5).

Figure 5.

Numerical results for the generalized inset CT specimen. The global stress intensity described as a function of the elastic modulus ratio (E_1/E_2) of the inset (E_1) and the molding materials (E_2).

a)

b)

c)

Figure 6.

Influence of debonding on the local driving force for fracture. The local stress intensity at the crack tip is normalized by the apparent global stress intensity achieved for the perfectly bonded condition. The bold portion of the interface is the region between the inset and molding material that remains bonded. a) displacement field map obtained from DIC for a

Filtek Z250 Inset CT specimen with interfacial debonding. The processed image illustrating the amount of the debonding that developed under cyclic loading used during the crack initiation phase of specimen preparation. b) Normalized stress intensity factor for debonding between A and B, equivalent to 100% of the edge length. c) Normalized stress intensity factor for complete debonding between A and B, and 50% of the edge length between B and C.