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Research paper

Sub-10-micrometer toughening and crack tip toughness of dental enamel

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ABSTRACT

In previous studies, enamel showed indications to occlude small cracks in-vivo and exhibited *R*-curve behaviors for bigger cracks ex-vivo. This study quantifies the crack tip's toughness (K_{I0}, K_{III0}), the crack's closure stress and the cohesive zone size at the crack tip of enamel and investigates the toughening mechanisms near the crack tip down to the length scale of a single enamel crystallite. The crack-opening-displacement (COD) profile of cracks induced by Vickers indents on mature bovine enamel was studied using atomic force microscopy (AFM). The mode I crack tip toughness K_{I0} of cracks along enamel rod boundaries and across enamel rods exhibit a similar range of values: $K_{I0,Ir}$ = 0.5–1.6 MPa $m^{0.5}$ (based on Irwin's 'near-field' solution) and $K_{I0,cz}$ = 0.8–1.5 MPa $m^{0.5}$ (based on the cohesive zone solution of the Dugdale–Muskhelishvili (DM) crack model). The mode III crack tip toughness $K_{III0,Ir}$ was computed as 0.02–0.15 MPa $m^{0.5}$. The crack-closure stress at the crack tip was computed as 163–770 MPa with a cohesive zone length and width 1.6–10.1 μm and 24–44 nm utilizing the cohesive zone solution. Toughening elements were observed under AFM and SEM: crack bridging due to protein ligament and hydroxyapatite fibres (micro- and nanometer scale) as well as microcracks were identified.

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1. Introduction

Enamel is the outermost layer of teeth. Throughout our lifetime, enamel remains intact despite millions of mastication loadings in the form of compression, shear and torsion. These result in a distribution of cracks in enamel (Bodecker, 1953; Chai et al., 2009). Despite this, enamel could be considered as a damage-tolerant material against crack propagation due to the following reasons. Small cracks in enamel of 40 μm deep and 8 μm wide were observed to be occluded by mineral deposition, proposed as a key phenomenon to repair tiny enamel cracks in vivo (Hayashi, 1994). Studies of ex-vivo cracks over larger distances have shown that enamel exhibits *R*-curve behavior; the stress intensity increased from values between 0.5 and 1.5 MPa $m^{0.5}$ up to 2.5 MPa $m^{0.5}$ at 1.5 mm crack extension in human enamel (Bajaj and Arola, 2009a) and up to 4.4 MPa $m^{0.5}$ at 500 μm crack extension in bovine enamel (Bechtle et al., 2010a). The reported toughening mechanisms are crack bridging of tissue ligaments of ~10 μm wide or bigger, microcracking, possible bridging by protein ligaments and crack deflection promoted by enamel rod decussations mainly existing in the inner enamel (Bajaj and Arola, 2009b; Bajaj et al., 2008).

The teeth of all mammals appear to be very similar on a histochemical basis (Oesterle et al., 1998). As a reference, the composition of human enamel varies about ~90% of apatite crystallites, ~8% of water and ~2% of organic matrix by volume (Healy, 1998). In some studies, bovine enamel was

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used to substitute human enamel (Nishimura et al., 2008; Ruse et al., 1990) due to their structural similarity (Sanches et al., 2009) that can be described by three hierarchical structural levels. The elementary construction unit consists of ~50 nm diameter apatite fibers (Sanches et al., 2009; Glimcher et al., 1965) that are probably surrounded by a 1–2 nm thin organic layer, observed earlier in human enamel (Frazier, 1968). These nanofibers are closely-packed to form the first level hierarchical structure, 3.7–8.8 μm rods (Sanches et al., 2009) that spans from the dentino-enamel junction (DEJ) to near the tooth's surface (Gray et al., 1995). 'Interrod substance' was observed in between rods where the crystallite orientations are significantly different from those in the center of the rods (Bajaj and Arola, 2009a; Bechtle et al., 2010b; Glimcher et al., 1965). The less dense 'interrod' regions are accumulated with proteins and water, thus appear as distinct protein-rich structures (Maas and Dumont, 1999). Bundles of these rods including the interrod regions create the second hierarchical layer. Groups of enamel rods have same local orientations but different from the adjacent groups of enamel rods, they form bands called Hunter-Schreger Bands as the third level of hierarchical structures (Ten Cate, 2003).

It is well-known from ceramic materials that toughness measurements with Single-Edge-Notch-Beam (SENB) or Compact-Tension (CT) specimens are usually not able to measure the crack tip's toughness, K_{I0}, in particular when steep crack resistance curves prevail (Fett et al., 2008; Fünfschilling et al., 2009; Özcoban et al., 2010). In brittle materials the most common used method to measure K_{I0} is to measure with high spatial resolution using an atomic force (AFM) or scanning electron microscope (SEM) the crack opening displacement (COD) in the crack tip region (Kounga Nijwa et al., 2003; Rödel et al., 1990; Meschke et al., 1997, 2000). With this information Irwin's crack tip solution (Irwin, 1958; Lawn, 1993) is applied to evaluate K_{I0}.

In enamel a crack tip toughness study based on COD measurements is missing which is the objective of this investigation. AFM is used as a high spatial resolution tool for COD measurement to calculate K_{I0} (Irwin, 1958; Lawn, 1993). The information is also used to apply a cohesive zone models developed by Goodier and Field (Goodier and Field, 1963; Hahn, 1976; Hahn and Rosenfeld, 1965) for the Dugdale–Muskhelishvili crack model (Dugdale, 1960; Muskhelishvili, 1953) to compute the closure stresses at the crack tip, the cohesive zone width and length, as well as the corresponding crack tip's toughness. In addition to AFM, SEM is also used to investigate toughening mechanisms around and behind the crack tip.

In a number of other studies (Bajaj and Arola, 2009a,b; Bechtle et al., 2010a; Bajaj et al., 2008) enamel's toughening mechanisms are already reported but not with a special focus on the different lengths scales. As enamel is hierarchically structured the objective is to identify the specific toughening mechanisms on the first and second hierarchical level as defined above using AFM and SEM.

2. Experiments

(A) *Specimen preparation:* Mature bovine incisors were used due to their larger amount of enamel compared to human teeth. The bovine teeth are stored in Hanks' Balanced Salt Solution (HBSS) before sample preparation. The rectangular prisms of $\sim 3 * 2$ mm^2 were first cut out from the labial side of mature bovine incisors (Fig. 1(a)) by using a saw (Brüder Mannesmann 92571, Germany) under water irrigation. The cows were sacrificed between 3–4 years old. The specimen's surface was first ground with an abrasive paper of grit 4000. It was further polished with diamond suspensions of 1 μm, 0.25 μm and finally 0.05 μm (Bühler, Germany). The polished tooth was then glued face-up on a steel plate with wax for subsequent investigation on the same day. A total of 15 specimens were investigated (13 specimens under AFM and 2 specimens under SEM).

(B) *Vickers indents and AFM measurements:* The freshly polished enamel surfaces were introduced with Vickers indents of 400 g for 10 s (3212, Zwick GmbH, Germany). An example is shown in Fig. 1(b). Without the introduction of the Vickers indent, no cracks were observed on the specimens following the same polishing procedures and examined under AFM with the same experimental conditions. The weight of 400 g was selected because the induced crack lengths were long enough that the crack lengths to indentation size ratio was bigger than 3. Yet, they should not be too long; due to the fact that scanning of high resolution pictures with AFM is extremely time consuming, excessive crack lengths would lower the chance to find the crack tip within one day after sample preparation and therefore are undesirable. 3–5 indents were made on the polished surface of each specimen; they were at least 500 μm apart. Only one specimen was investigated within a day. No samples are re-used overnight because polished samples, if left immersed in HBSS solution for rehydration overnight, were covered with a layer of salt deposition that precludes nanoscale observation of the sample surface under AFM. Only one crack (from one specimen) could be investigated in one day. The indents were first observed under a light microscope (Aristome, Ernst Leitz Wetzlar, Germany). Then, the crack profiles were examined under AFM (Dimension 3100, Veeco, USA) in tapping mode. Cracks with insufficient crack lengths were discarded. We calculated the total crack lengths by summing up the lengths under the optical microscope attached with the AFM system and the AFM topography scans. All the measurements were carried out in an air environment.

As the darker contrast in the AFM topography images belongs to lower topography levels, the crack can be identified as a dark contrast due to the deeper position of the AFM tip. A X-Y sample stage was used to trace the cracks away from the Vickers indents as far as the crack tips. Super sharp tips with tip apexes < 10 nm (SuperSharpSilicon, Nanosensors, Switzerland) were used together with a slow scanning frequency of 0.2 Hz to acquire high resolution images (an example is shown in Fig. 1(c)). The height profiles across the crack at the desired positions are later obtained from the topographical images to measure the COD between two opposing crack walls ($2u$) as well as the height difference ($2u_z$) (Fig. 1(e)), both as a function of the distance from the crack tip (X). The COD measurement was stopped when the crack could no longer be differentiated from the surface roughness, this position is taken as the zero position for X (Fig. 1(f)). The resolution of the AFM measurement depends on the

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