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## Research paper

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

Siang Fung Ang, Anja Schulz, Rodrigo Pacher Fernandes, Gerold A. Schneider\*

Institute of Advanced Ceramics, Hamburg University of Technology, Denickestr. 15, 21073, Hamburg, Germany

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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:  $K_{I0, Ir} = 0.5\text{--}1.6 \text{ MPa m}^{0.5}$  (based on Irwin's 'near-field' solution) and  $K_{I0, cz} = 0.8\text{--}1.5 \text{ MPa m}^{0.5}$  (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  $0.02\text{--}0.15 \text{ MPa m}^{0.5}$ . The crack-closure stress at the crack tip was computed as  $163\text{--}770 \text{ MPa}$  with a cohesive zone length and width  $1.6\text{--}10.1 \mu\text{m}$  and  $24\text{--}44 \text{ nm}$  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  $40 \mu\text{m}$  deep and  $8 \mu\text{m}$  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  $1.5 \text{ MPa m}^{0.5}$  up to  $2.5 \text{ MPa m}^{0.5}$  at  $1.5 \text{ mm}$  crack extension in human enamel (Bajaj and Arola, 2009a) and up to  $4.4 \text{ MPa m}^{0.5}$  at  $500 \mu\text{m}$  crack extension in bovine enamel (Bechtle et al., 2010a). The reported toughening mechanisms are crack bridging of tissue ligaments of  $\sim 10 \mu\text{m}$  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  $\sim 90\%$  of apatite crystallites,  $\sim 8\%$  of water and  $\sim 2\%$  of organic matrix by volume (Healy, 1998). In some studies, bovine enamel was

\* Corresponding author. Tel.: +49 40 42878 3037; fax: +49 40 42878 2647.

E-mail address: [g.schneider@tuhh.de](mailto:g.schneider@tuhh.de) (G.A. Schneider).