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Research Paper

Damage mechanisms in uniaxial compression of single enamel rods

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ABSTRACT

Enamel possesses a complex hierarchical structure, which bestows this tissue with unique mechanical properties. In this study, the mechanical behavior of single enamel rods was investigated under uniaxial compression. Numerical simulations were also performed using micromechanics models for individual enamel rods to identify the damage mechanisms contributing to the constitutive behavior. Experimental results showed that the single rods exhibited an elastic modulus ranging from 10∼31 GPa, and that they undergo post-yield strain-hardening. The primary damage mode consisted of delamination within the assembly of mineral crystals. Results from numerical simulations suggest that strain localization within individual rods is responsible for the observed delamination, which is believed to arise from the non-uniform arrangement of mineral crystals. This mechanism was independent of mineral morphology and properties. The non-uniform crystal arrangement results in friction between crystals with different inclination angles and is believed to be responsible for the post-yield strain hardening behavior.

1. Introduction

Enamel is an interesting biological composite that possesses a complex hierarchical structure. At the microscale enamel consists of stiff, highly mineralized rods that are embedded in a soft protein matrix (Ge et al., 2005; Habelitz et al., 2001). At the nanoscale the individual rods exhibit a composite assembly, with mineral crystals and proteins serving as the reinforcements and matrix, respectively (Cui and Ge, 2007; Hannig and Hannig, 2010). The mineral crystals within the individual rods are not simply arranged uniformly along the axial direction. In the head region they are aligned with the rod axis, whereas in the tail region they are arranged at an angle of approximately 60° between the rod and crystal c-axes (White et al., 2001).

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There is a gradual increase in the angle of inclination between the head and tail regions.

The hierarchical structure of enamel and relative ratio of mineral and organic elements endows this tissue with a unique combination of mechanical properties. According to indentation measures, the elastic modulus of enamel ranges from 70–120 GPa (Xu et al., 1998; Mahoney et al., 2000; Fong et al., 2000; Cuy et al., 2002). The elastic modulus of this tissue is important to its wear resistance and in facilitating the cutting and grinding of food. Resistance to fracture is another critical quality of the tooth and its tissues. Enamel undergoes an increase in resistance to fracture with crack extension (Bajaj and Arola, 2009a; 2009b; Bechtle et al., 2010; Padmanabhan et al., 2010), which plays an important role in preventing tooth fracture. Decussation of the enamel rods within the inner enamel causes crack deflection and bifurcation, both potent mechanisms that contribute to energy dissipation and the fracture toughness (Bajaj and Arola, 2009a). Additionally, crack bridging caused by uncracked ligaments in the crack wake reduces the stress intensity in the vicinity of crack tip and promotes crack closure (Bajaj and Arola, 2009b; Yahyazadehfar et al., 2013). Bechtle et al. (2010) observed that cracks propagate along the protein-rich rod sheaths, which leads to crack deflection and promotes fracture resistance as well. At the nanoscale, unfolding of protein ligaments and microcracking result in loosening of the enamel microstructure, and serves to dissipate energy during crack extension (Ang et al., 2011).

There are other aspects of the microstructure that serve important roles in promoting damage tolerance. Chai et al. (2009) reported that the crack-like defects (i.e. enamel tufts) emanating from the dentin-enamel junction contribute to stress shielding through an increase the compliance of the enamel shell. These mechanisms contribute to damage tolerance of the tooth and provide resistance to catastrophic fracture. Furthermore, using numerical simulations An et al. (2012a) showed that the non-uniform arrangement of mineral crystals within the enamel rods serves to provide the necessary stiffness that is essential for occlusion, while also enhancing energy dissipation.

An understanding of the mechanical behavior of enamel also requires knowledge of the contributing mechanisms of deformation at the level of individual enamel rods. Jeng et al. (2011) evaluated the hardness and elastic modulus of single enamel rods using nanoindentation and found that the rods exhibited region-dependent mechanical properties; the elastic modulus within the head region was larger than that of the tail region and axial-section plane. They attributed the spatial variations to the differences in crystal orientation between the head and tail regions. The complex crystal structure of the rods could be important to their damage tolerance. Yilmaz et al. (2013) performed micropillar compression experiments on single rods of bovine enamel. They found that failure of single rods occurred through localized fracture and spreading of the crystallites rather than complete collapse of the rod through buckling failure. Su et al. (2012) performed an analytical investigation concerning buckling of staggered nanocomposites of biological materials, which revealed important transformation mechanisms from local to global buckling and the role of structural hierarchy in improving buckling strength. Although that study provided new insight into the failure mechanisms of biological composites, it cannot explain the damage behavior reportedly observed in the compression failure of single enamel rods. In fact, the properties of individual enamel rods are not well established. As such, the link between this micro-structural element and the bulk behavior of enamel is not well understood.

In the present study the mechanical behavior of enamel rods was studied using a combination of experiments and numerical modeling. Micropillar compression experiments were performed on single enamel rods to determine the mechanical properties and to identify the contributing damage mechanisms. Numerical simulations were performed using micromechanics models to analyze the role of the rod architecture on the deformation behavior. Contributing damage mechanisms responsible for the failure behavior of enamel rods were revealed.

2. Materials and methods

2.1. Micropillar compression experiments

Human molars were acquired from participating dental hospitals in Shanghai, China according to approved protocols issued by the Institutional Review Board of Shanghai University. Using a programmable slicer machine (EC400, Shenyang Kejing Instrument Co., Ltd China), the molars were sectioned along a plane approximately perpendicular to the axes of enamel rods in the cuspal region. Hydration of the teeth was maintained during sectioning by frequent application of a water spray. After sectioning, the sectioned surfaces were polished using silicon carbide papers with #600~#5000 mesh. All the specimens were then stored in Hank's Balanced Salt Solution (HBSS).

A total of five cylindrical micropillar specimens were cut from five different cuspal sections using a FEI Helios nanolab 600i Dualbeam focused-ion-beam (FIB) system. The method of cutting was identical to that described by Chan et al. (2009), which leads to that the orientations of mineral crystals in the specimens are identical to those of mineral crystals in the center region of enamel rods. Processing was performed to obtain specimens with upper face diameter of approximately 3 μm, height of approximately 5 μm and taper angle of $\sim 3^\circ$ (Fig. 1(a)).

Compression testing of the enamel micropillars was carried out using a Hysitron Triboscope nanoindenter In-Situ Nanomechanical Test System (Hysitron, Minneapolis, MN, USA) with a flat punch conical indenter of 10 μm diameter. Prior to the experiments the frame compliance was determined using the direct measurement approach after Van Vliet et al. (2004). Results of this activity provided an estimated compliance of approximately 0.35 μm/N. The enamel specimens were then loaded along the axial direction with a loading rate of 0.03 μm/sec. The load and axial displacement history from the compression experiments were used to assess the engineering stress-strain behavior of the rods. Displacement of the micropillar base associated with deformation of the enamel base was treated using Sneddon's solution (Sneddon, 1965) and the mechanical response of the micropillars was estimated using the normalized stress-strain description proposed by Han et al. (2011). The stress-strain curve was determined in terms of normalized stress-strain description, which is based on Sneddon's formula (Sneddon, 1965). The normalized strain is calculated as