Journal of Biomechanics 47 (2014) 1060–1066

Contents lists available at ScienceDirect

Journal of Biomechanics

journal homepage: www.elsevier.com/locate/jbiomech
www.JBiomech.com

Size dependent elastic modulus and mechanical resilience of dental enamel

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ARTICLE INFO

Article history:
Accepted 22 December 2013

Keywords:
Tooth enamel
Young's modulus
Nanoindentation
Deformation
Finite element analysis

ABSTRACT

Human tooth enamel exhibits a unique microstructure able to sustain repeated mechanical loading during dental function. Although notable advances have been made towards understanding the mechanical characteristics of enamel, challenges remain in the testing and interpretation of its mechanical properties. For example, enamel was often tested under dry conditions, significantly different from its native environment. In addition, constant load, rather than indentation depth, has been used when mapping the mechanical properties of enamel. In this work, tooth specimens are prepared under hydrated conditions and their stiffnesses are measured by depth control across the thickness of enamel. Crystal arrangement is postulated, among other factors, to be responsible for the size dependent indentation modulus of enamel. Supported by a simple structure model, effective crystal orientation angle is calculated and found to facilitate shear sliding in enamel under mechanical contact. In doing so, the stress build-up is eased and structural integrity is maintained.

1. Introduction

Being the hardest tissue in the human body (Fig. 1(a)), dental enamel is built to last (Lawn et al., 2009). The remarkable resilience of enamel stems from its unique microstructure that comprises highly organised hydroxyapatite (HAP) platelets (Fig. 1b, c); Nanci, 2008; Xie et al., 2009a, 2008). Electron and atomic force microscopy investigations also reveal that a thin protein layer is present between the HAP crystallites in mature enamel (Habelitz et al., 2001; Warshawsky, 1989) and this, though small in quantity, plays an important role in regulating the mechanical properties of enamel to uniquely suit its functions (Gao et al., 2003; Lawn et al., 2010). Young's modulus is commonly used to quantify a material's stiffness. Enamel has a hybrid laminate structure at nanoscale (He and Swain, 2007; Xie et al., 2009b), which means its modulus is determined not only by the volume fraction of its constituents but also by the spatial arrangement of the mineral crystals (Xie et al., 2009a).

Young's modulus of enamel is routinely measured using the depth-sensing indentation (DSI, also called nanoindentation) method (Oliver and Pharr, 1992, 2004). Values reported in the literature vary by over 100% (Cuy et al., 2002). Such a wide range results from multiple factors, such as variations in location, testing conditions (i.e., dry or wet) or age (Cuy et al., 2002; Habelitz et al., 2001; Lewis and Nyman, 2008; Park et al., 2008; Staines et al., 1981). Moreover, the Young's modulus of enamel was found to decrease with increasing indentation depth (Zhou and Hsiung, 2007). Given the dependence of Young's modulus of enamel on indentation depth, it is important to conduct measurements under depth control when interrogating the stiffness over the thickness of enamel. Unfortunately, most nanoindentation tests have been performed under load control (i.e., at constant load), making it difficult to compare the modulus values obtained from different regions, where indentation depths may differ (He et al., 2006; Xie et al., 2007b; Zhou and Hsiung, 2007). Moreover, although the sample preparation and testing conditions are known to influence the measured mechanical properties of enamel, (Guidoni et al.,

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http://dx.doi.org/10.1016/j.jbiomech.2013.12.030

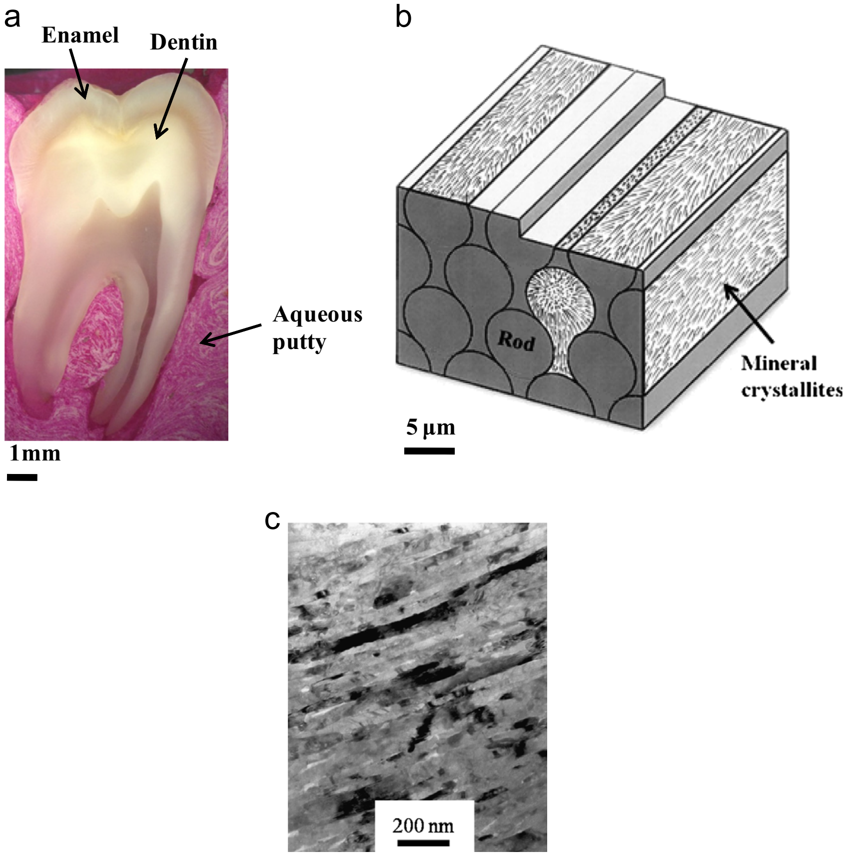

Fig. 1. Tooth structure viewed at different length scales. Optical image of (a) tooth macrostructure consisting of enamel and dentin. (b) Schematic illustration of enamel microstructure formed by tightly packed rods (or prisms) that are aligned perpendicular to the enamel–dentin junction. Inside the rods the mineral crystallites are bonded together by proteins (Habelitz et al., 2001; Warshawsky, 1989) (modified from (Nanci, 2008)). (c) Transmission electron micrograph of small enamel section (refer to 'box' in (b)) prepared parallel to the *c*-axis of rods.

2006; Staines et al., 1981) the majority of mechanical tests were carried out under dry condition or on 'wet' specimens that had previously been dried during preparation (Cuy et al., 2002; Xu et al., 1998).

In this study, we conducted an in-depth investigation of the elastic behaviour of enamel under the influence of mechanical loads. Enamel samples were prepared and tested in a hydrated state. Young's modulus of enamel was determined using a line scan obtained from the occlusal surface to the enamel–dentin junction (EDJ) at various depths. A mechanistic model was used to link crystal orientation angle with the depth dependent elastic behaviour of enamel. Furthermore, the implications of crystal orientation in promoting shear deformation and dissipating indentation energy were examined and clarified.

2. Experimental

2.1. Sample collection and preparation

Ethics approval was given by the Ethics Review Committee of Edith Cowan University prior to the collection of teeth. Nine third molars extracted from patients aged 20–30 years were used in this study. The teeth were intact without any decay or other disease and were extracted for clinical reasons and on the treating dentists' advice because of crowding in the jaw and impaction of the teeth preventing them from eruption. Informed consents were obtained from patients involved. Upon extraction, teeth were stored at 4 °C in Hanks' Balanced Salt Solution (HBSS) (Sigma-Aldrich Co., St. Louis, USA) with the addition of 0.02% thymol crystals to prevent demineralisation and inhibit bacterial growth (Habelitz et al., 2002; White et al., 1994).

Struers resin and hardener were mixed to form 30 mm diameter by 10 mm high cylindrical blocks inside Struers plastic moulding cups. A ~15 mm diameter hole was then drilled through the centreline of the block, which was later filled from one end with aquatic putty (Selleys Knead-It Aqua, Selleys, Australia). The tooth was inserted root first into the cylinder and pressed into the putty, leaving only the occlusal surface exposed. A precision saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) was used to section the partially embedded tooth at a distance of 5 mm from its edge in a direction parallel to mesial–distal plane. HBSS was used as a coolant during sectioning. The cut surface was then ground and polished with series of Struers silicon carbide papers up to 4000 grit. The final polishing of the specimens was done on a soft polishing cloth soaked in water (LaboPol 25, Struers, Copenhagen, Denmark). The *c*-axis of prisms was organised roughly parallel to the polished surface. Once prepared, the specimen was stored in HBSS that contains 0.02% thymol crystals.

2.2. Nanoindentation testing

A depth-sensing indentation system (Ultra-Micro Indentation System, UMIS-2000, CSIRO, Australia) equipped with a Berkovich indenter was used to measure the Young's modulus of enamel (Oliver and Pharr, 1992, 2004). The indenter tip was calibrated by conducting single-cycling indentation tests on fused silica (a standard material having a known modulus of 72 GPa) at different loads. To conduct measurements in a wet condition, a specially designed stainless steel holder was used, and the specimen was clamped onto the bottom using a slide-screw assembly. A stainless steel plate of 1 mm in thickness was placed on the specimen to distribute the clamping force evenly. The specimen was submerged in HBSS before testing. The polished surface of enamel was indented from the occlusal surface to the enamel–dentin junction (EDJ). Each row was generated along a line parallel to the surface and contains 5 indents with an interval of 50 µm. The spacing between rows is also 50 µm. Load–partial unload tests were run in a closed-loop under load control to determine the elastic modulus of enamel at different depths. A maximum load of 400 mN was applied in eight increments. The loading rate was set to 2.5 mN/s, which represents the static response of the material. Following each increment were 10 decrements, from which the average Young's modulus and standard errors were calculated with the load applied roughly perpendicular to the prism direction (Oliver and Pharr, 1992, 2004) and plotted as a function of indentation depth. Recent nanoindentation studies have shown that prism orientation has little impact on the hardness and modulus of enamel. (Braly et al., 2007;