Acta Biomaterialia 10 (2014) 4852–4860

Contents lists available at ScienceDirect

Acta Biomaterialia

journal homepage: www.elsevier.com/locate/actabiomat

On the mechanical properties of tooth enamel under spherical indentation

Herzl Chai *

School of Mechanical Engineering, Faculty of Engineering, Tel Aviv University, Tel Aviv, Israel

ARTICLE INFO

Article history:
Received 5 May 2014
Received in revised form 24 June 2014
Accepted 2 July 2014
Available online 14 July 2014

Keywords:
Tooth enamel
Indentation
Constitutive law
Failure stress
Crack growth

ABSTRACT

The mechanical properties of tooth enamel generally exhibit large variations, which reflect its structural and material complexity. Some key properties were evaluated under localized contact, simulating actual functioning conditions. Prominent cusps of extracted human molar teeth were polished down ~0.7 mm below the cusp tip and indented by tungsten carbide balls. The internal damage was assessed after unloading from longitudinal or transverse sections. The ultimate tensile stress (UTS) was determined using a novel bilayer specimen. The damage is characterized by penny-like radial cracks driven by hoop stresses and cylindrical cracks driven along protein-rich interrod materials by shear stresses. Shallow cone cracks typical of homogeneous materials which may cause rapid tooth wear under repeat contact are thus avoided. The mean stress vs. indentation strain curve is highly nonlinear, attributable to plastic shearing of protein between and within enamel rods. This curve is also affected by damage, especially radial cracks, the onset of which depends on ball radius. Several material properties were extracted from the tests, including shear strain at the onset of ring cracks γ_F (= 0.14), UTS (= 119 MPa), toughness K_C (= 0.94 MPa m$^{1/2}$), a crack propagation law and a constitutive response determined by trial and error with the aid of a finite-element analysis. These quantities, which are only slightly sensitive to anatomical location within the enamel region tested, facilitate a quantitative assessment of crown failure. Causes for variations in published UTS and K_C values are discussed.

1. Introduction

Tooth enamel, although nominally brittle with mechanical properties similar to glass, can survive a lifetime of repeat contact forces under harsh conditions, a feat accomplished by a unique natural design. This work is concerned with the determination of some key mechanical properties of the enamel coat, necessary information, in addition to tooth occlusal geometry and dentin support, for assessing the fracture resistance of dental crowns. The structure of enamel is well documented (e.g. [1,2]); a thorough overview of the subject is given by Ang et al. [3]. Enamel consists of aligned prisms or rods made of hydroxyapatite (HAP) nanocrystals glued by thin protein layers. The rods, ~5 μm in diameter, are connected by ≈1 μm thick interrod material which differs from rod enamel in the direction of crystalline patterns. The crystals of rods and interrods meet at sharp angles, resulting in a protein-rich space called the rod sheath. The rods are grouped into Hunter–Schreger bands, the orientations of which undulate and cross each other much like a basket weave.

Considerable effort has been devoted to the determination of mechanical properties of tooth enamel controlling tooth resilience, e.g. elastic modulus and hardness, stress–strain response, failure stress and fracture toughness. The majority of works employ nano- (e.g. [4,5]) or micro- (e.g. [6–8]) indentation, for which a small amount of material suffices. More intricate test specimens are those fabricated from the enamel coat, such as microtensile [9,10], compact tension [11] and micron-size beams [12] or micropillars [13]. Reported data generally display large variations, e.g., ultimate tensile stress (UTS) from as little as 11.4 MPa [9] to over 1000 MPa [12] and toughness K_C from 0.5 to 2.4 MPa m$^{1/2}$ [11]. Such variations may be due to a variety of factors, e.g. anatomical location, sample size, loading manner, stress risers and possibly data interpretation. It is also not clear how such data may be combined into an analytical framework capable of predicting crown failure, and what is the precise role played by the various enamel microstructural entities on tooth resilience. As a first step toward these goals a systematic, multi-phase study was conducted of the deformation and fracture of tooth enamel under localized contact simulating real-life conditions.

Polished cusps of extracted human molar teeth were indented with tungsten carbide (W/C) balls to predetermined load values,

* Tel.: +972 3 6408342; fax: 972 3 6407617.
E-mail address: herzl@eng.tau.ac.il

http://dx.doi.org/10.1016/j.actbio.2014.07.003
1742-7061/

and the unloaded samples were sectioned longitudinally or transversely to expose the internal damage. The size and distribution of induced cracks were quantified as a function of load level and ball radius. In a side effort the UTS was determined using the bilayer specimen [14], here adopted to thin layers produced from the enamel coat. An axisymmetric FEA in conjunction with a choice of material model was used to produce enamel's true stress–true strain curve from plots of mean stress vs. indentation strain data. Section 2 details the materials and methods used, while Section 3 presents the test results together with analytical expressions for enamel's constitutive response and crack initiation and growth conditions. The results are assessed against published material property data in Section 4. Major conclusions and clinical implications of the results are summarized in Section 5.

2. Materials and methods

The human molar teeth used were supplied by the School of Dental Medicine at Tel-Aviv University. The teeth, extracted from adults aged 20–30, were carefully examined under a stereo microscope to exclude any tooth having cracks, discoloration, caries or geometric abnormality. The teeth were kept in distilled water at all times prior to testing.

2.1. UTS

The UTS was determined using the bilayer test, an approach especially suitable to small-size materials [15]. A thin layer produced from the enamel coat was bonded onto a polycarbonate slab before it was indented with a W/C ball. The bending of the layer under the load induces flexural stresses, which may initiate radial cracks at the layer's subsurface. The fracture process was observed from below the sample using a video camera. The failure stress of enamel σ_F was calculated from the load P_F needed to initiate radial cracks according to

$$\sigma_F = BP_F/d^2$$
$$B \equiv (1 - 2\nu_e)/4\pi + 0.72 \log\left[E_e(1-\nu_p^2)/E_p(1-\nu_e^2)\right] \quad (1)$$

where d denotes enamel layer thickness, E and ν are Young's modulus and Poisson's ratio, respectively, and subscripts "e" and "p" stand for enamel and polycarbonate, respectively.

The enamel layers were produced as follows. After embedding the tooth in epoxy resin for support, a prominent cusp was ground down 0.7 mm below its tip using a 0.3 mm thick diamond coated disk. This was followed by polishing the surface to a mirror quality with a series of diamond pastes ending with a 1 μm particle size. Next a 1 mm thick slice was cut from this material using the diamond disk. After properly supporting the polished side of the slice on the supporting epoxy surface external to the enamel, the opposite side was polished down as before to the desired level. In this way enamel layers 0.45 mm thick and several mm in diameter free of visible damage were produced. The layers were glued to a polycarbonate slab with a two-part epoxy resin at room temperature. After the resin was cured the layer surface was indented by a 0.78 mm radius W/C ball at the rate of 0.1 mm min^{-1}. The resulting bending of the layer under the softer substrate initiated radial cracks at the layer subsurface. Substituting the crack initiation load P_F in Eq. (1) yields a failure stress σ_F. To enhance light reflection, the lower surface of the enamel layer was gold-coated before bonding.

2.2. Deformation and fracture

Prominent enamel cusps were first polished as described above, yielding a flat surface 0.7 mm below the tip of the cusp or ~1.2 mm above the dentin–enamel junction (DEJ), which was the working surface in the test. The adopted level of enamel polish reflects a compromise between a need to have a large indentation area far from a free surface and a surface not too close to the soft dentin. In this way the indentation stress field under reasonable load levels was ensured to be free of any external effects. After polishing, the roots of the teeth were cut off 2 mm below the enamel terminus, the cut surfaces placed on a flat platen and the polished surfaces indented with a W/C ball to a prescribed load value. After unloading, the samples were sectioned transversely at various depths or longitudinally through the contact center on a plane containing two opposite cusps. The cut surfaces, polished to a mirror quality as described above, were observed with optical or scanning electron microscopes. From this, contact radius, crack initiation load, crack length and other forms of damage were measured and documented. Data were produced for ball radii r = 0.2, 0.4, 0.78 and 1.55 mm. The load P varied from 5 to 920 N.

2.3. Constitutive behavior

Stress–strain curves for enamel were generated from mean stress vs. indentation strain data with the aid of an axisymmetric FEA (Ansys, Inc.). The model material used for this purpose is homogeneous and isotropic, characterized by a Ramberg–Osgood stress–strain law of the form

$$\varepsilon/\varepsilon_Y = \sigma/\sigma_Y + k(\sigma/\sigma_Y)^{n-1}, \quad (2)$$

where ε and σ are the uniaxial true strain and true stress, respectively, ε_Y (= σ_Y/E_e) and σ_Y are the corresponding quantities at first yield, k is a constant and n a hardening coefficient. Eq. (2) is a continuous function governed by three adjustable parameters which may facilitate a realistic response for a wide range of materials. The yielding of the model material is assumed to obey von Mises' flow rule with incremental plasticity and isotropic hardening. The stress–strain response of this material was implemented in the FEA using ten linear segments. Indentation was applied by W/C balls of radius r = 0.2, 0.78 or 1.55 mm. The load was transmitted to the specimen surface using a built-in contact algorithm. For simplicity a frictionless contact was assumed. The model dimensions (radius and height) are at least 100 times the contact radius, which ensures that all edge effects are eliminated. The finite-element mesh was refined until all stresses of interest converge to within 1–2%. The parameters σ_Y, k and n were determined by trial and error such that the resulting mean stress vs. indentation strain data closely matched the experimental results.

Some auxiliary ball contact tests were conducted on soda-lime glass to help establish a universal crack growth law. In all calculations Young's modulus and Poisson's ratio for enamel, W/C and polycarbonate were taken as (E_e, E_i, E_p) = (90, 600, 2.35) GPa and (ν_e, ν_i, ν_p) = (0.32, 0.22, 0.35).

3. Results

3.1. Fracture morphology

Fig. 1 shows a series of transverse sections for a sample indented with a 0.78 mm radius ball at load P = 349 N. Fig. 1a corresponds to the indented surface while b–d are 0.15, 0.24 and 0.32 mm below, respectively. The indented surface exhibits a set of ring cracks marked "C" over an annulus region limited by the contact radius as well as a set of radial cracks (R) extending outward from the contact area. As evident from Fig. 1a–c the ring cracks grow as cylindrical cracks within the material. These cracks disappear in Fig. 1d, revealing that their extent is quite limited compared to radial cracks. It is noted that cylindrical cracks are