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Effect of hydration and crack orientation on crack-tip strain, crack opening displacement and crack-tip shielding in elephant dentin

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ABSTRACT

Objectives. To quantify the extent of crack-tip plasticity, crack opening displacement (COD) and crack bridging for crack growth perpendicular (HAH) and parallel (RAR) to the tubules in elephant dentin under both hydrated and dry conditions to better understand their influence on intrinsic and extrinsic toughening during crack growth.

Methods. Compact tension test-pieces were prepared from a tusk of African elephant ivory. Crack-tip strain mapping and COD measurements by digital image correlation (DIC) technique were made under incremental loading and unloading of cracks for hydrated and dry dentin of different orientations.

Results. For the RAR test-piece the plastic zones were significantly larger in the hydrated condition compared to when dry. By contrast, the plastic strains in the HAH test-piece were negligible in both wet and dry conditions. In the RAR condition the crack front was broken up into overlapping longitudinal 'fingers' with crack bridging regions in between, the ligaments extending 400 μm behind the crack front in the dry case. This could only be seen in 3D by X-ray CT. Extrinsic shielding reduces the crack-tip stresses by 52% and 40% for hydrated and dry RAR test-pieces respectively. No significant bridging was found in the HAH case.

Significance. For crack growth parallel to the tubules, collagen plasticity determines the intrinsic toughening, whereas microcracking from the tubules governs extrinsic shielding via ligament bridging, which is maintained further behind the crack in the hydrated case. For cracks grown perpendicular to the tubules, neither toughening mechanisms are significant.

1. Introduction

The crack-resistance of dentin, as the major constituent of teeth and tusk, is a subject of considerable biomechanical interest. The resistance of a material to crack propagation (its fracture toughness) is related to intrinsic and extrinsic toughening mechanisms. Extrinsic toughening mechanisms operate primarily behind the crack tip by introducing crack-tip shielding, which reduces the local stress intensity actually

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experienced at the crack tip. Intrinsic toughening mechanisms operate ahead of the crack tip and contribute to the material's inherent resistance to crack growth [1].

This paper aims to characterize and compare the extent of crack-tip plasticity (intrinsic toughening) and bridging (extrinsic toughing) of elephant dentin under hydrated and dry conditions to reveal the importance of the microstructure in inhibiting crack propagation. From a practical viewpoint, elephant dentin (tusk) has the advantage over human dentin that tusks are very much larger than human teeth making the preparation of nominally identical compact tension test-pieces of different orientations from the same region easier than for human teeth [2] where sample to sample variations would increase the scatter. Ivory tusk is made up of a peripheral component, the cementum, continuous with, but structurally different from, enamel and a main core comprising dentin (see Fig. 1a). The cementum layer is softer than the dentin inside [3]. The dentinal tubules, are the predominant feature in the main dentin, running approximately radially from the central pulp to the periphery of the dentin (see Fig. 1b). These tubules are embedded in a mineralized collagen matrix, which consists of Type I collagen fibrils and Mg-containing hydroxyapatite [3–8]: the highly mineralized hydroxyapatite (HAP) improves the stiffness, while collagen provides toughness [9]. While the main constituents of elephant and human dentin are similar, there are some important differences. Firstly, while human dentin has tubules of approximately circular cross-section, the tubules in elephant dentin are elliptical with the major axis parallel to the length of the tusk (see Fig. 1c and f). This ellipticity is often exaggerated further in micrographs because the tubules also have a periodic wavy trajectory as they emanate radially from the central pulp cavity of the tusk to the cementum layer [10]. Moreover, elephant dentin has no highly mineralized peritubular cuff. Another critical difference is that in human dentin the collagen fibrils are arranged in a planar random mat, perpendicular to the long axis of the tubules [11]. As such they form a mesh and cross-link around the tubules to provide its desirable mechanical properties [8,12]. By contrast recent work by Alberic [13] and Lu [14] suggest that for elephant dentin the collagen fibrils are approximately aligned ($+/-10^{\circ}$) to the semi major axis of the tubules, which coincides with the axial direction of the tusk (see Fig. 1e and f).

In recent years, much work has been done on the fracture mechanics of human, bovine and elephant dentin [15–18]. Crack growth is highly anisotropic. For most kinds of dentin (e.g. human, bovine and elephant dentin), the fracture toughness K_c is 55–65% higher for cracks propagating parallel to the long axis of the dentinal tubules compared to crack propagation perpendicular to the tubules [2,17,19,20] and the fatigue life in human dentin is two orders of magnitude longer for cracks growing parallel to the tubules as opposed to perpendicular to them [4,21]. Moreover, it has been found that fracture toughness increases with the hydration level [22] and tends to fall with the age of the donor [23,24]. Several toughening mechanisms in dentin, particularly for the case where a crack propagates parallel to the tubules, have been proposed such as crack blunting [2,24], crack-tip bridging [2,19,24,25], and microcracking [19,25].

The resistance-curve (R-curve) is a means of evaluating the build-up of crack retarding effects during subcritical crack propagation (i.e. before unstable fracture occurs). Rising R-curve behavior is particularly important in biomaterials, e.g. teeth, bones and tendons [26–31]. Normally an R-curve is expressed in terms of the crack-driving force (the stress intensity K) as a function of the crack extension, Δa, to quantify the increasing fracture resistance with crack length. Previous investigations have revealed that dentin exhibits much higher fracture toughness and crack growth resistance (expressed via an R-curve) when hydrated rather than dry along with enhanced crack-tip blunting [23,32,33]. Hydration has been reported to decrease the elastic Young's modulus and hardness by approximately 35% and 30% compared with the dry dentin [34–36]. The hydrated tissues exhibit viscoelasticity, demonstrating a good ability to recover the elastic energy stored in the region surrounding the deformed area [37].

Our objective is to quantify the mechanisms of toughening as a function of sample orientation and hydration specifically for elephant dentin. We consider the plastic zone ahead of the crack (intrinsic component) and the extent of crack-tip shielding in the crack wake expressed as the fraction of the applied stress intensity transmitted to the crack tip (extrinsic toughening). Here digital image correlation (DIC) is adopted for full-field crack-tip strain field mapping. It requires no, or very little, sample preparation and can be applied to awkward biological materials. Previously it has been used to quantify volume shrinkage and load transfer in dentin [38,39] and bones [40,41]. Here, it is used for the first time to quantify the elastic and plastic (inelastic) crack-tip strains accumulated as the load is increased for test-pieces cracked in different orientations under both wet and dry condition. The obtained plastic strain sizes are then compared with predictions made using the measured elastic stress fields with different crack-tip yield criteria to establish the crack-tip shielding.

2. Materials and experimental methods

2.1. Test-piece preparation

The mature African elephant tusk used in this study originated from Zaire and was impounded at London Heathrow airport and made available by the UK Customs House, ethically and legally, solely for the purpose of scientific research. The hierarchical microstructure of the elephant dentin is illustrated in Fig. 1. Compact tension test-pieces ($10 \times 8 \times 2$ mm) conforming to plane strain conditions were prepared based on ASTM E1820 [42]. The test-pieces were extracted from the interior of the elephant ivory sample as shown in (Fig. 1a). In order to investigate the anisotropic properties, two orientations of compact tension test (CT)-pieces were excised: (1) HAH, in which crack plane is in the hoop-axial plane and grows in the hoop direction (Fig. 1f), and (2) RAR, in which the crack plane is in the radial-axial plane and grows in the radial direction (Fig. 1e). The 3D alignment of the dentin tubules was observed using an Xradia Versa XRM-500 laboratory X-ray microscope (Fig. 1b). In reality these tubules are not straight but oscillate over a larger length scale. SEM images show the elliptical cross sections of the tubules (Fig. 1c) and the surrounding collagen fibrils are