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Hierarchical microcrack model for materials exemplified at enamel

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ABSTRACT

Objective. This article investigates the mechanical properties of a material with hierarchically arranged microcracks.

Methods. Hierarchically structured biomaterials such as enamel exhibit superior mechanical properties as being stiff and damage tolerant at the same time. The common mechanical explanation for this behavior is based on the hierarchically structured arrangement of hard minerals and soft organics and their cooperative deformation mechanisms. In situ mechanical experiments with mm-sized bovine enamel bending bars an scanning electron microscope reveal that enamel is able to withstand mechanical loading even if it contains microcracks on different lengths scales. To clarify this issue an analytical hierarchical microcrack model of non-interacting cracks is presented.

Results and Significance. The model predicts a decrease of the elastic modulus and the fracture strength with increasing levels of hierarchy. The fracture strain on the other hand may decrease or increase with the number of hierarchical levels, depending on the microcrack density. This simple hierarchical microcrack model is able to explain already published experiments with focused ion beam prepared μm-sized enamel cantilevers on different hierarchical levels. In addition it is shown that microcracking during loading in hierarchical materials may lead to substantial pseudoplastic behavior.

1. Introduction

Many biomaterials such as enamel, bone or nacre consist of hard minerals surrounded by soft proteins, which are arranged in a hierarchical manner. Dental enamel, for instance, consists of nanometer-scaled hydroxyapatite (HAP) fibers arranged into rod-like structures. These rods are organized into groups with different orientations crossing each other to form the so-called decussation pattern. This kind of structure – from the HAP nanofiber to the decussation pattern level (as shown in Fig. 1) – is an example of a hierarchical structured biomaterial. In this example, one can define a single HAP nanofiber as the hierarchical level 0 and the bundled HAP nanofibers as the hierarchical level 1. Enamel rods can be defined as the hierarchical level 2, and the decussation pattern as the hierarchical level 3 [1,2].

To overcome the material availability problem with human teeth, bovine teeth – that are thicker and less curvy – are quite frequently used in dental studies, due to their similar morphologies. Both species exhibit three levels of hierarchy: nanofibers, rods and Hunter–Schreger Bands. Moreover, the studies of Yahyazadehfar et al. [3] and Bechtle et al. [4] on human and bovine enamel respectively, revealed that in both species similar toughening mechanisms prevail.

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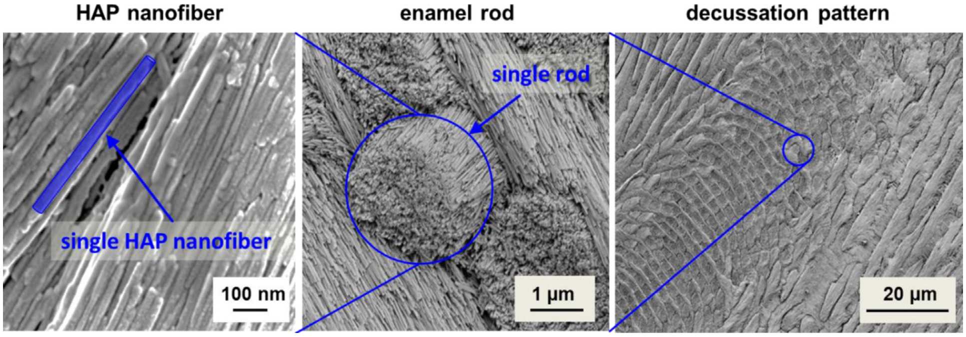

Fig. 1 – Hierarchical structure of bovine enamel: from a single HAP nanofiber to the decussation pattern.

Enamel is a highly mineralized tissue (up to ~85 vol%) and thus the hardest material in the human body. From a mechanical point of view, the most significant aspect is that the hierarchical structure enables enamel being stiff and damage tolerant at the same time. Toughening mechanisms such as crack bridging, microcracking or crack deflection operate during crack growth and are supposed to be even more efficient than in non-hierarchical materials [3–5]. Experiments with µm-sized cantilevers of different hierarchical levels have shown that bovine enamel shows a transition from a stiff and high-strength material to a damage-tolerant and low-strength material on the hierarchical level 3 (decussation pattern) [1]. This result matches very well with the hierarchical mechanics model developed by Gao and co-workers based on the suggestion from Jäger and Fratzl [6]. The model uses a tension-shear-chain model to explain the mechanical behavior of composite structures under tension. It bases on the assumption that the hard particles carry the tensile load, whereas the soft elements transfer the tensile load via shear to the hard particles. A comprehensive explanation of this model can be found elsewhere [7–10].

In this study, in situ bending experiments were performed with mm-sized bovine enamel specimens to get more insight into the role of hierarchy, the interaction of the hierarchical levels and the corresponding deformation mechanisms. While loading the enamel specimens, microcracks on different length scales could be observed on the sample surface in the scanning electron microscope (SEM). Despite of these microcracks, enamel could withstand mechanical loading and deformed macroscopically in a linearly elastic manner until catastrophic failure occurred. However, it is unclear if the microcracks were already in enamel before or if they were generated during the experimental procedure. It is possible that the microcracks are introduced via sample preparation, during evacuating the chamber of the SEM or while loading. Nevertheless, the experiments lead to the question: what is the influence of microcracks on the mechanical behavior of a hierarchically structured biomaterial. It is well known that enamel contains microcracks in different length scales [5] and that enamel continues its function through millions of chewing cycles even if it contains inherently crack-like defects [11].

In this work, we propose a hierarchical microcrack model for hierarchically structured materials. The analytical model shows the influence of microcracks on different hierarchical levels on the elastic modulus, the fracture strength and the fracture strain.

2. Experiments

2.1. Sample preparation

Bovine permanent mandibular incisors were used in this study due to their larger size compared to human enamel. They were extracted at a local slaughterhouse (Lippeck & Richter GmbH, Hamburg). Roots were cut off, the pulp interior was removed and the teeth were disinfected within a 0.1 wt% thymol solution for 24 h. Teeth were then rinsed and further stored in Hank's Balanced Salt Solution (HBSS, Invitrogen, USA) to avoid any dehydration and correlated changes in mechanical properties in the sample preparation process. Slices of around 2 mm thickness, as shown in Fig. 2a, were cut out of the middle of bovine incisors using a Buehler Isomet 4000 precision saw. These slices were further sectioned from the labial side into bar-like shape by the saw. The gross bars are grinded down using the profile forms with differently sized gaps for the tooth bar getting smaller with each profile form (shown in Fig. 2b) so that the final required rectangular shape ($\sim 1 \times 1 \times 10\,\text{mm}^3$) can be obtained. The last grinding step is done with 1200 grit SiC paper and only the side, which is monitored in the SEM, was further polished with 1 µm and 0.25 µm diamond suspension. The polished surface layer was then subsequently etched for 1 s using 36% hydrochloric acid to make the structural features visible and was sputter-coated with a thin gold layer (some nanometers).

2.2. Observation of microcracks during in situ bending experiments in the SEM

The bovine enamel bars were loaded with a 3-point bending device (Microtest 200, Gatan GmbH, Germany) with a span distance of 7 mm, where the sample surface was observed simultaneously by means of the Zeiss Supra 55VP scanning electron microscope (3 kV, 10^{-6} mbar). Before performing the test, the specimen was firstly pre-loaded with a force of ~1N to ensure that the specimen did not move while evacuating the chamber of the SEM. The experiments were performed