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Bone toughness and crack propagation: an experimental study

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Abstract

Bone is a topic of great interest for researchers, such as biologists or engineers, both interested in understanding the structure-related properties of bone and how they are affected by aging, disease and therapies. In particular, a topic of common interest between medicine and engineering is the fracture behavior of bone. Indeed, a thorough understanding of the mechanical behavior of bone is helpful to predict the fracture risk, but it can also provide the basis for the design of *de novo* biomimetic materials. In this paper, we show the initial results of an experimental study of the mechanical behavior of bovine bone, with a special focus on fracture toughness. The latter is evaluated under tensile and bending loading, by following the ASTM adopted for metals. Finally, we perform microscopic observations to better understand the fracture behavior and correlate it with the microscopic structure.

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Keywords: bone; crack; fracture toughness; biomimetic materials.

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1. Introduction

Bone is currently a topic of interest for researchers, either biologists or engineers, interested in understanding the structure-related properties of bone [1, 2] and how they are affected by aging, disease and therapies [3, 4]. In particular, a topic of common interest between medicine and engineering is the fracture behavior of bone. There is a large literature about mechanical testing of bone, under different loading, confirming a high variability of the data. Indeed, a common interest of researchers is to probe the effect of different factors on bone response. Firstly, bone structure and properties are strictly dependent on the type of tissue, on the type of animal, on the position (i.e. area of the body). Also, the effect of age, disease, and the metabolic activity directly influence the overall response of bone

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under mechanical stimuli. Indeed, bone is a living material, whose structure is continuously evolving as a result of external stimuli (e.g. mechanical). Hence, bone structure results from continuous stress-induced remodeling and, in turn, affects the mechanical response. The main interest of researchers is to understand how the structure of bone affects the mechanical behavior. Also, being bone structure hierarchically organized, this interest extends through different length scales. Therefore, a multi-scale approach is generally adopted to study the mechanisms occurring at different length scales.

One aspect, particularly investigated, is the fracture behavior of bone and the toughness amplification occurring in bone and other natural composites [5]. This fascinating feature of bone has been studied for decades and is still a hot topic for researchers. Indeed, a thorough understanding of the mechanical behavior of bone is helpful to predict the fracture risk, but it can also provide the basis for the design of *de novo* biomimetic materials. The latter is a very common trend for engineers, which aim to develop new materials with remarkable properties by mimicking smart systems existing in biological materials, such as wood, bone, nacre [6-8]. Indeed, the goal of this study is to investigate the toughening mechanisms occurring in bone and reproduce them in newly designed composite materials.

2. Materials and methods

We obtained samples from an 18-months old bovine femur, three days after slaughter. We removed the marrow and obtained test samples by wet-machining the cortical shell of the bone shaft, by means of a low-speed saw and a milling machine. We stored the samples in saline solution at 3 °C for 20 hours, then at room temperature for two hours until testing. We carried out experimental tests under mainly tensile and flexural loading. For all the tests we used an MTS ALLIANCE RT/100 universal tensile testing machine, endowed with a 100 kN load cell.

2.1. Three-point bending tests

Being bone mainly subjected to flexural loading, we investigated the response of cortical bone under this type of load by performing three-point bending tests. For this test, we followed the standard ASTM D790 [9], generally used for unreinforced plastics but also adopted for bone tissue. Tests were carried out in displacement control mode, with a 0.5 mm/min crosshead speed and a data acquisition frequency of 10 Hz. We adopted rectangular specimens (50·8·4 mm), where the length of the specimens corresponds to the main axis of the osteons.

2.2. Single edge three-point bending tests

To investigate the fracture behavior we followed the ASTM E399, the American standard generally used for metals [10]. For three-point bending fracture toughness tests we used single edge notch bending specimens, SE(B) with W = 10 mm and a through-thickness notch, which extends over half of the specimen width (see Fig. 1-a). Also in this case, the length of the specimens corresponds to the main axis of the osteons. To obtain a localized stress concentration, a fatigue pre-crack is generally used. However, bone is difficult to fatigue pre-crack because additional damage is generally created during crack propagation and the crack path is normally not straight. Hence, to create a sharp notch, we used a razor and we measured the extent of the notch (i.e. about 200 μm) by means of an optical microscope. These tests were carried out in displacement control mode, with a 0.1 mm/min crosshead speed and a data acquisition frequency of 10 Hz.

2.3. Compact tension tests

To investigate fracture toughness under a mainly tensile loading we used compact tension specimens, C(T) with W = 25.4 mm and a through-thickness notch, which extends over half of the specimen width (see Fig. 1-b). The direction of the applied load is parallel to the main axis of the osteons. As for SE(B) specimens, to create a sharp notch, we used a razor and we measured the extent of the notch (i.e. about 200 μm) by means of an optical microscope. We should stress that the dimensions of the C(T) do not follow the standard. In particular, due to the limited depth of the cortical shell, the thickness of the specimens is 40 % less than the one suggested by the standard. The tests were carried out in displacement control mode and the load applied through pin loading clevises with a 0.1 mm/min crosshead speed. In this case, to measure the crack mouth opening displacement (CMOD) we used an MTS 632.02F-20 clip gage. The data acquisition frequency was set to 10 Hz.

2.4. Microscopic observations

We used an optical microscope (LEITZ WETZLAR GmbH type 307-148-002) to observe the microstructure of cortical bone before testing and the fracture surfaces after testing. The surfaces of untested samples were accurately prepared before testing by polishing with silicon carbide papers of increasing grit number (600, 800, 1200, 2500), stirring in distilled water and final polishing with diamond abrasive paper.

Fig.1. (a) SE(B) specimen; (b) C(T) speciemen.

3. Results and discussion

The results obtained from the experimental tests showed a good repeatability and a good agreement with the literature [2, 11, 12]. In particular, from three-point bending tests we obtained a flexural stiffness of 10.7 ± 1.4 GPa and a bending strength of 217 ± 16 MPa. The bending strength in presence of a notch is 132 ± 14 MPa, reduced by 40 % with respect to the unnotched specimens.

Regarding the fracture toughness tests, after post processing the results we could confirm that all the fracture toughness tests were performed in plain-strain conditions, though the smaller thickness of C(T) samples compared to the standard requirements. Firsly, we followed the LEFM to determine the fracture toughness and we found $K_C = 5.6 \pm 0.1$ MPa√m for SE(B) and $K_C = 5.8 \pm 0.6$ MPa√m for C(T) specimens. However, during the tests we observed a rather plastic behavior, which required the adoption of nonlinear fracture mechanics. Hence, by following the standard ASTM E-1820 [13] we determined the J integral, which is a measurement of fracture toughness in non-linear elastic singularity. In particular, it describes the fracture toughness at crack initiation. For SE(B) specimens $J_c = 3.2$ kJ/m^2, whereas for the C(T) ones $J_c = 4.7 \pm 2.2$ kJ/m^2. These results confirms previous findings. Also, by observing the fracture behavior, we noticed a strong dependence on water content. Indeed, bone is a highly hygroscopic material, whose water content affects the ability of deforming and dissipating energy under mechanical loading, leading to a more ductile material. For instance, the fracture toughness results from our experiments appeared to be lower than the fracture toughness measurements of previous studies performed in water [12]. In particular, the plastic contribution (*i.e.* J_{pl}/J_{tot}) increases of 28% when performing tests in water. Indeed, the amount of absorbed plastic energy is about 80 % for tests performed in wet conditions [12], compared to 57 %, corresponding to our tests. A further decrease of the mechanical properties can be observed when testing dry specimens [14]. In this study we mainly focused on ductile initial cracking. However, as extensively shown in previous studies, the fracture toughness –intended as resistance to crack growth – increases with crack length, meaning that the larger the crack the tougher becomes bone [2, 15, 16], in contrast to brittle materials, where toughness remains constant regardless the crack extent.

In the above described tests we investigated the transversal fracture toughness, where transversal is referred to a direction orthogonal to the osteon main axis. As expected, the fracture mode is different from traditional materials such as steel: for SE(B) specimens the surface of fracture is rough, sign of absorption of a large amount of energy before fracture; fro C(T) specimens instead, the crack tends to deviate of 90° and propagate vertically, probably along the weak osteon-osteon interface.

In view of mimicking bone structure in new biomimetic materials, we also investigated the internal mechanisms involved in the fracture process and affecting the crack path. The microscopic observations confirmed the toughening mechanisms of crack deviation and splitting, largely discussed in the literature and predicted by our numerical analyses. Microscopic images of untested bone samples show holes, delaminations between different circumferential lamellae, and cracks, tens of microns long (see Fig. 2b). The cracks never show a linear path, but instead a clear interaction with the microstructure, confirming a crucial role played by osteons, which represent the characteristic cylindrical feature of the microstructure of bone, and probably by the cement lines, which are the external layer of each osteon [11]. Indeed, as shown by the microscopic image in Fig. 2b, the osteons are sites of cracks: microcracks originates from osteons, porosities and canals, which act as stress concentrations zones.

This preliminary study confirms the possibility of adapting testing benchmarks generally used for structural materials to perform experimental measurements of bone mechanical properties. The microscopic analyses confirm previous findings about characteristics features of the microstructure of cortical bone.

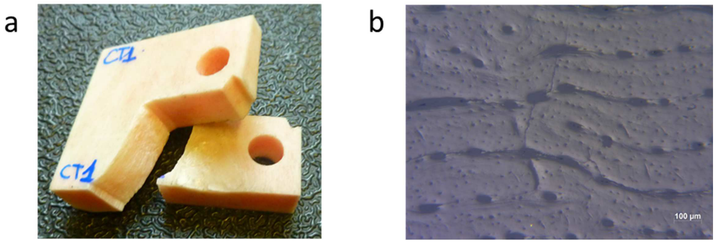

Fig. 2. (a) Fracture of a C(T) specimen; (b) Microstructure of cortical bone (Optical Microscope,50X magnitude).

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