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Relating crack-tip deformation to mineralization and fracture resistance in human femur cortical bone

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ABSTRACT

The risk of bone fracture increases with age because of a variety of factors that include, among others, decreasing bone quantity and quality. Despite recent advances, the roles of bone microstructure and trace mineralization in the fracture process are not well understood. In this study, we utilize a combination of in-situ fracture toughness testing, digital strain mapping, and X-ray photoelectron spectroscopy techniques to characterize the near-tip strain field, fracture toughness, and chemical elements on the fracture surface of bone specimens from donors of two ages (48-year-old and 78-year-old females). We show that age-related embrittlement of bone fracture is associated with higher near-tip strains by lamellar shear and crack deflection at lamellar interfaces in the young bone and their absence in the old bone. The different near-tip deformation behaviors may be associated with the presence of Si and Zn in the young bone but more Ca and P and the lack of Si and Zn in the old bone.

Introduction

Humans experience an age-related increase in the incidence of skeletal fractures that may be due to a variety of factors including reduced bone mineral density, impaired balance and reflexes, changes in bone geometry, porosity, architecture, and mineral and organic phases, as well as damage accumulation [1–5]. It is becoming increasingly evident that bone mass alone cannot account for changes in observed fracture risk and that measures of bone mass should be supplemented with measures of bone quality [6]. However, the mechanisms through which various measures of bone quality act to control bone mechanical properties and ultimately fracture risk are not well understood. Of particular interest is bone fracture toughness, which is a measure of the ability of bone tissue to resist initiation and propagation to failure of a crack. As a result, a mechanistic understanding of the bone quality and other factors that control crack initiation and growth process in bone tissue is important in developing a methodology for fracture prediction, particularly considering bone material and microstructural modifications resulting from pharmacological treatment, aging and disease processes.

Recent work has shown that cortical bone exhibits a resistance-curve fracture behavior that varies with age [7]. The development of microdamage and linear microcracks also changes with age and the mode of loading (tensile versus compression) [8]. Bridging of crack surfaces by uncracked ligaments occurs at several length scales, including one of a nanoscaled non-fibrous organic glue material found between mineralized collagen fibrils [9]. Despite these recent advances, the role of the bone microstructure, which is comprised of Haversian, lamellar and interstitial bone tissue, in affecting the local deformation and the fracture process is still poorly understood. In particular, it remains unclear how bone mineralization affects the crack growth process and the fracture resistance in bone. The aim of this article is to report recent results of fracture-resistance curves, crack-tip strain measurements, and the corresponding mineral content for cortical bone of two age groups that shed new light on the role of mineralization in the crack growth process and in understanding of the risk of bone fracture with aging. It should be noted that while this study is focused on cortical bone, cancellous bone is also prominently involved in many clinical fractures.

Materials and methods

Cortical bone from human femurs of two ages (48-year-old female and 78-year-old female) were studied. Throughout this paper, specimens from the 48-year-old donor will be designated as "young", while those from the 78-year-old donor will be designated as "old". Compact-tension specimens ($n=4$ for young bone and $n=5$ for old bone) were machined from the mid diaphysis. Briefly, two cuts approximately 10 mm apart were made transverse to the

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long axis of the femur approximately 25 mm below the lesser trochanter using a band saw while keeping the specimen wet under constant irrigation creating rings of cortical bone. The rings were then cut into quarters using the band saw. Using a mini-milling machine (Sherline, model 5400, Vista CA), specimens of dimensions of about 8.2 mm height (anatomical circumferential direction), 6.0 mm width (anatomical radial direction), and 1.96 mm thickness (anatomical longitudinal direction) were machined under constant irrigation to minimize damage due to specimen heating. The specimens were notched with a 0.5 mm thick slitting saw attached to the mini-milling machine and precracked using a razor blade [10], resulting in a crack length of about 1.44–2.19 mm. The compact-tension specimens were orientated such that the cracks propagated in the radial (R) direction normal to the loading direction in the circumferential (C) direction of the bone, which is often referred to as the C–R orientation [11]. All specimens were polished using an increasingly finer grit paper up to with a final polishing step using a 0.05 μm diamond slurry. Fracture toughness tests were conducted at ambient temperature under displacement-controlled conditions at a displacement rate of 0.001 mm/s using a custom-designed mechanical loading stage operated under a digital microscope (Keyence VHX-100, Osaka, Japan). The specimens were fully hydrated in physiological buffered saline solution prior to testing. During testing, the near-tip region of the compact-tension specimens was imaged using the digital microscope at each load step to document the cracking process, the amount of crack extension (Δa) and the crack path. After testing, the stress intensity factor (K) was computed using the standard K solutions for compact-tension specimens described in ASTM E399 [12].

The use of linear-elastic fracture mechanics for a porous material such as bone tissue can be justified on the basis of a recent analysis [13] that showed that the stress intensity factor parameter remains valid for an elastic material with voids. The stress intensity factor for a porous material is only slightly increased by the presence of porosity. The weak dependence is described by a coupling parameter that is a function of elastic properties of the unvoided materials and material constants used to describe the porosity field [13]. Unfortunately, the analysis [13] did not provide a simple relation between the stress intensity factor and the porosity level. Although the K solution for porous materials is still not fully developed, the recent analysis [13] supports the notion that applying the traditional stress intensity factor solution as described in the ASTM procedure [12] to porous materials is likely to be reasonably accurate.

Loaded and unloaded digital micrographs of the same crack-tip region were analyzed to obtain the near-tip displacement and strain fields using a digital strain mapping technique [14]. A t-test assuming unequal variance (one-tail and two-tail) was used to compare the crack-initiation toughness, K_o, at the onset of crack growth, and the slope of the linear K vs. Δa curves for the two ages. Specimen porosity was computed from images taken at 100× total magnification using an image based methodology as described by Wang and Ni [15]. The porosity measurements included the Haversian canals, Volkmann canals, and lacunae. Osteon density was determined by counting the number of osteons per unit area of measure [16]. Using micrographs of the crack path meandering through the bone microstructure, the number of osteons exhibiting crack deflection and that of crack penetration were determined for individual specimens. The results were utilized to compute the relative frequencies of crack deflection by osteons observed in both young and old bone.

To gain insight on the mechanisms potentially related to bone quality responsible for preventing or hindering interlamellar shear in the old bone, the chemical elements present on the fracture surfaces of the young and old bone tissue were analyzed using X-ray photoelectron spectroscopy (XPS). X-ray photoelectrons spectroscopy, also known as electron spectroscopy for chemical analysis (ESCA) is a quantitative spectroscopic technique that measures the elemental composition, empirical formula, chemical state, and electronic state of the elements that exist within a material [17,18]. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and the number of electrons that escape from the top 1 to 10 nm of the material being analyzed [17,18]. XPS analyses were performed on the fracture surfaces of the bone specimen at six different locations ($n=6$) and at six different depths by varying the sputtering times. The six locations were randomly selected from the fracture surfaces of a single compact-tension specimen for each of the two ages. Since the X-ray beam sampled a circular area of 100 μm in diameter, each location was about 100 μm in diameter, and corresponded to the spot size of the X-ray beam. The spacing between two locations was about 1 mm apart. Optical microscopy was used to ensure the sampling area was not over a Haversian canal. The microstructural features (such as lamellar layers, interfaces, or sub-microned pores) within the 100 μm sampled area, unfortunately, could not be revealed using optical microscopy. XPS analysis of the surface was conducted with a PHI 5000 VersaProbe (Physical Electronics, Chanhassen, MN) equipped with an Al K-α X-ray radiation source. The X-ray takeoff angle was 45° and the spot size was about 100 μm. The survey scans were taken with an energy resolution of 1.5 eV using a source with power and current of 125 kW and 10 μA, respectively. Charge neutralization using a dual beam of low energy Ar^+ ions and low energy electrons was used during data acquisition. Depth profiling was performed by sputtering using an Ar^+ ion beam (5 kV beam energy, 3 μA beam current, 1 mm × 1 mm raster size) for 30 s in between each survey scan. Attempts were made to determine the sputtering depth as a function of time. For depth profiling, it was necessary to sputter for a known time and then measure the depth of the trench left behind. However, the effort was unsuccessful because the fracture surfaces were not flat and contained pores, so it was not possible to locate

Table 1
Comparison of crack-initiation toughness (K_o) at the onset of crack growth and the slope of linear K vs Δa curve for young (48-year-old) and old (78-year-old) bone.

	48-year-old female					78-year-old female				
	K_o, MPa $(m)^{1/2}$	Slope, MPa $(m)^{1/2}$/mm	Porosity (%)	Osteon density (#/mm^2)	Crack-deflection osteons$^+$ (%)	K_o, MPa $(m)^{1/2}$	Slope, MPa $(m)^{1/2}$/mm	Porosity (%)	Osteon density (#/mm^2)	Crack-deflection osteons$^+$ (%)
	1.07	1.85	15.23	4.64	50	0.34	0.026	18.51	4.85	0
	2.24	2.38	7.10	1.69	54	0.29	0	31.41	4.43	0
	2.05	1.76	12.98	3.80	76	0.18	0	21.63	4.32	0
	1.07	1.86	5.74	4.43	86	0.35	0.086	15.72	3.69	32
						0.4	0.041	11.39	4.43	–
Mean	1.61*	1.96*	10.26*	3.64	0.654*	0.31*	0.031*	19.73*	4.35	0.08*
(S.D.)	0.625	0.283	4.6	1.35	0.164	0.083	0.036	7.53	0.42	0.167

The specimen porosity is also given.
* This signifies a statistically significant difference ($p<0.05$) between the means of the old and the young.
$^+$ Number of osteons exhibiting crack deflection normalized by total number of osteons along the crack path.