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Research Paper

Quantification of nonlinear elasticity for the evaluation of submillimeter crack length in cortical bone

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ABSTRACT

The objective of this study was to investigate the sensitivity of the nonlinear elastic properties of cortical bone to the presence of a single submillimetric crack. Nonlinear elasticity was measured by nonlinear resonant ultrasound spectroscopy (NRUS) in 14 human cortical bone specimens. The specimens were parallelepiped beams ($50 \times 2 \times 2$ mm^3). A central notch of 500 μm was made to control crack initiation and propagation during four-point bending. The nonlinear hysteretic elastic and dissipative parameters α_f and α_Q, and Young's modulus E_{us} were measured in dry condition for undamaged (control) specimens and in dry and wet conditions for damaged specimens. The length of the crack was assessed using synchrotron radiation micro-computed tomography (SR-μCT) with a voxel size of 1.4 μm. The initial values of α_f, measured on the intact specimens, were remarkably similar for all the specimens ($\alpha_f = -5.5 \pm 1.5$). After crack propagation, the nonlinear elastic coefficient α_f increased significantly ($p < 0.006$), with values ranging from -4.0 to –296.7. Conversely, no significant variation was observed for α_Q and E_{us}. A more pronounced nonlinear elastic behavior was observed in hydrated specimens compared to dry specimens ($p < 0.001$) after propagation of a single submillimetric crack. The nonlinear

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elastic parameter α_f was found to be significantly correlated to the crack length both in dry ($R=0.79$, $p<0.01$) and wet ($R=0.84$, $p<0.005$) conditions. Altogether these results show that nonlinear elasticity assessed by NRUS is sensitive to a single submillimetric crack induced mechanically and suggest that the humidity must be strictly controlled during measurements.

1. Introduction

This study focuses on bone microdamage and particularly on the microcracks that are thought to play an important role in bone strength (Burr et al., 1998; Diab and Vashishth, 2005; Hernandez et al., 2014; Yeni and Fyhrie, 2002), toughening mechanisms (Diab and Vashishth, 2005; Fletcher et al., 2014; Vashishth, 2004; Yeni and Fyhrie, 2002) and remodeling (Cardoso et al., 2009; Verborgt et al., 2000). The link between microcracks and bone fracture is not fully elucidated yet (Burr, 2011; Chapurlat and Delmas, 2009; Gupta and Zioupos, 2008), though microdamage is suspected to be at the origin of some spontaneous fractures (Shane et al., 2010).

The quantitative assessment of damage characteristics remains challenging. Histomorphometry is the gold standard, although providing only 2-D damage characterization (Lee et al., 2003; O'Brien et al., 2007, 2002). New techniques are emerging, such as synchrotron radiation micro-computed tomography (SR-μCT) (Haupert et al., 2014; Larrue et al., 2011; Voide et al., 2009). However, none of these invasive techniques can be applied in vivo, nor is able to assess damage in a large bone volume. Consequently there is a clear need for alternative techniques to assess bone microdamage.

Nonlinear acoustics methods have been introduced recently for the investigation of bone microdamage. They include nonlinear resonant ultrasound spectroscopy (NRUS) (Haupert et al., 2014; Muller et al., 2008), nonlinear wave modulation spectroscopy (NWMS) (Ulrich et al., 2007) and dynamic acousto-elastic testing (DAET) (Moreschi et al., 2011; Renaud et al., 2008). These approaches, directly inspired from nonlinear elastic wave spectroscopy (NEWS) methods developed in the field of nondestructive testing of materials, have been reported to be sensitive to fatigue-induced damage in materials such as concrete (Antonaci et al., 2010; Chen et al., 2010; Van den Abeele and De Visscher, 2000), metals (Cantrell, 2006; Cantrell and Yost, 2001; Frouin et al., 1999; Kim et al., 2006, 2004; Nagy, 1998; Sagar et al., 2011; Zagrai et al., 2008) or composites (Aymerich and Staszewski, 2010; Bentahar and El Guerjouma, 2009; Meo et al., 2008; Van den Abeele et al., 2009, 2001b). However, so far, few studies have attempted to quantify microdamage, using nonlinear acoustic methods (Moreschi et al., 2011; Van den Abeele et al., 2009, 2001a). Haupert et al. (2014) have investigated the relationship between the nonlinear elastic behavior measured by NRUS and fatigue-induced microdamage of cortical bone specimens, taking advantage of micrometer resolution imaging by SR-μCT. With this approach, the study evidenced a significant correlation between the variation of bone microcrack density and the variation of nonlinear elasticity. These findings not only revealed the sensitivity of NRUS to early bone microdamage, but also established the first quantitative relationship between the variation of nonlinear elasticity of bone and variation of a microcrack characteristic.

A complementary vision to the fatigue test is to produce damage locally by initiating and propagating a single crack in a controlled manner. In this study, calibrated specimens of human cortical bone undergoing a toughness test were measured by NRUS and then imaged by SR-μCT. Our main objective was to investigate whether the nonlinear elastic behavior or the elastic modulus are changed after propagation of a localized single crack and to specifically assess the relationship between the nonlinear elastic behavior and the length and the orientation of the crack. This was achieved by comparing NRUS measurements on native and cracked specimens in dry condition. An ancillary objective was to investigate the influence of hydration on the nonlinear response. To this goal, NRUS testing was performed on cracked specimens in dry and wet conditions.

2. Methods

2.1. *Specimen preparation*

Fourteen human cortical bone specimens were prepared from the femoral mid-diaphysis of four donors (age$=79.4\pm3.9$). Ethical approval for the collection of samples was granted by the Human Ethics Committee of the Département Universitaire d'Anatomie de Rockfeller (Lyon, France). The tissue donors or their legal guardians provided informed written consent to give their tissue for investigation, in accord with legal clauses stated in the French Code of Public Health. Parallelepiped beams-shaped specimens ($50\times2\times2$ mm^3) were wet machined (Isomet 4000, Buehler GmbH, Düsseldorf, Germany) oriented along the proximal to distal direction of the femur. Then they were notched by a diamond wire saw (ESCIL W3000, Chassieu, France) in their middle to form a notch of roughly a quarter of the specimen width (i.e. 500 μm). The notch orientation was such that the nominal crack-growth direction for subsequent toughness testing was transverse to the long axis of the specimen, in a direction perpendicular to the osteon alignment. All specimens were defatted for 12 h in a chemical bath of