CHARACTERIZATION OF CORTICAL BONE MICRODAMAGE BY NONLINEAR RESONANT ULTRASOUND SPECTROSCOPY

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SUMMARY
The objective of the study is to evaluate the resonant ultrasound spectroscopy technique for measuring micro-damage accumulation in cortical bone induced by four-point bending fatigue. Results show that nonlinear ultrasonic coefficients are sensitive to micro-damage accumulation.

INTRODUCTION
Accumulation of bone microdamage is suspected to lead to severe impairment of mechanical properties such as bone toughness and stiffness [1, 2] with an increase in skeletal fragility and fracture risk [1]. The ability to characterize non-invasively microdamage accumulation in cortical bone would therefore be important to improve fracture risk prediction. Conventional techniques used to characterize damage accumulation are inherently invasive and destructive [3]. A preliminary in vitro study by our group suggested that accumulation of damage induced by mechanical fatigue were reflected by hysteretic nonlinear elastic properties measured by resonant ultrasound spectroscopy (NRUS) [4] and by nonlinear wave modulation spectroscopy (NWMS) [5]. The overall objective of the present study was to evaluate NRUS for measuring micro-damage accumulation in cortical bone using both mechanical experiments and 3-D high-resolution synchrotron micro-computed tomography (µCT). In the present paper, we report the results of NRUS and mechanical tests achieved on human cortical bone specimens. The specimens were prepared following a specific protocol designed to control damage localization during four-point bending fatigue cycling and to unambiguously identify resonant modes for NRUS measurement. Results from 3-D µCT measurements will follow in a subsequent report.

METHODS
Specimen preparation and measuring protocol
Sixteen human cortical bone samples were prepared from the femoral mid-diaphysis of four female donors (age = 88.5±9.8). They were wet machined (Isomet 4000, Buehler GmbH, Düsseldorf, Germany) as parallelepiped beams (50×4×2mm), defatted and stored in a freezer at -20°C before experiments. The procedure for the NRUS and mechanical studies began with the initial NRUS measurements for all samples to determine the initial nonlinearity of the material. The samples were then taken through a damage step, consisting of cyclic four-point bending as described below, during which mechanical parameters were determined. After each cycling session, a nonlinear elasticity experiment was conducted. Four damage steps were achieved. After each damage step, three specimens were removed for future 3-D µCT investigations of micro-damage.

NRUS measurements
The principles of NRUS measurements have been extensively described elsewhere [6]. Briefly, a piezoceramic emitter was bonded at one end of the specimen for NRUS measurements. Each sample was probed by a swept-sine encompassing the first resonant modes of the cortical beam (assumed to be pure compression modes under symmetric loading conditions). The peak resonance frequency \( f \) and energy loss \( Q^{-1} \) were measured as a function of strain applying increasing voltage drive level. The dynamic strain amplitude \( \varepsilon \) was calculated from the longitudinal particle displacement measured by a laser vibrometer LSV 1MHz (SIOS, Germany). When hysteric nonlinearities appear for strains above approximately 10\(^{-5}\) [7], two nonlinear parameters \( a_q \) (elastic) and \( a_o \) (energy loss) related to the frequency shift \( \Delta f \) and the change of energy loss as a function of strain, respectively can be extracted:

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\frac{f - f_0}{f_0} = \frac{\Delta f}{f_0} = a_q \Delta \varepsilon \quad (1) \quad \frac{1}{Q} \frac{1}{Q_0} = \frac{a_q}{2} \Delta \varepsilon \quad (2)
\]

where \( f \) and \( Q \) are the resonance frequency and Q-factor at increased strain level, \( f_0 \) and \( Q_0 \), their corresponding value at the lowest drive amplitude [7]. During the NRUS measurements, specimens were placed into a climate chamber to control for temperature (37°C ±0.1°C) and for relative humidity (15%-±5%).

Biomechanical testing
The piezoceramic emitter attached to the specimen for NRUS measurements was removed before each mechanical testing. All specimens were progressively damaged by cyclic four-point bending at 2Hz in a saline solution at 37°C (±1°C) using a hydraulic testing machine (INSTRON, 8802, High Wycombe, England) with a 1kN loading cell (accuracy 0.5%) and the internal displacement transducer (accuracy 1%). In this configuration, damage is expected to occur specifically in the mid region of the sample [5], while the ends remaining intact may be used as control. Initial Young’s modulus was determined during pre-cycling after 20 cycles (\( E_{0\text{pre}} \)) with a displacement range of 0.15 mm. From the pre-cycling, the load (\( F_{\text{max}} \)) corresponding to 5000µε at the mid-span was computed for all specimens [8]. The four-point bending
fatigue was then applied between -10N and –Fmax. During the cycling session, load and displacement curves were recorded to extract the following parameters: secant modulus (E_{sec}), loading modulus (E_{load}), and linear elastic beam theory (LEBT) modulus (E_{LEBT}) [9]. E_{LEBT} has been shown to decrease as bone micro-damage accumulates [8-10]. The progressive damage was performed in four steps, each step are defined by a multi-criteria: decrease of the E_{LEBT} or pre-determined number of cycles or E_{LEBT} vs cycles curve evolution. This multi-criteria definition of each step was chosen to avoid specimen failure before the end of the fourth step.

RESULTS AND DISCUSSION

The measurement precision of NRUS (coefficient of variation), assessed by repeating three times the measurements with intermediate debonding and repositioning, was found to be 7.5% and 6.6% for α_f and α_q, respectively. Despite substantial between-sample variability, the nonlinear ultrasonic properties increased in all specimens with the progression of fatigue cycling. Mean values (±standard error) of both α_f and α_q (normalized by their initial values) represented as a function of damage step in figure 1 indicate a relative 50% and 20% increase on average of α_f and α_q respectively at the end of fatigue cycling. Note that the number of specimens included in each cycling session decreased as a function of the damage step, because 3 specimens were removed for μCT experiments after each session. The highest mean values recorded at the end of the second cycling session are attributed to three specimens that showed a dramatic increase of nonlinear properties (more than 200%) compared to other specimens. Because these three specimens were suspected to be seriously damaged, they were removed from further mechanical testing for μCT imaging. Altogether the results evidence a similar evolution of both α_f and α_q and a higher sensitivity of α_q during fatigue cycling.

The number of cycles required to achieve the desired E_{LEBT} reduction was found to vary between the specimens (e.g., 1279±993 cycles for the first cycling session) despite homogeneous initial biomechanical properties (E_{sec}=15.1±2.9GPa, apparent density=1792±155g.mm⁻³). The mean value (±standard error) of E_{LEBT} (normalized by the initial value) is represented as a function of damage step in figure 1. Both elastic moduli (E_{sec} and E_{load}) show a limited variation during the fatigue cycling with a final value comparable to the initial one. As expect the E_{LEBT} presents an important decrease along the fatigue experiment. The mean decrease reaches 43% at the end of the fourth step. Altogether our results evidence:

- A progressive decrease of E_{LEBT} during fatigue cycling, strongly suggestive of micro-damage accumulation, in agreement with previous works [8,10].
- A similar evolution of α_f and α_q, both increasing during fatigue cycling. The magnitude of the variation is higher for α_f compared to α_q. These data suggest that α_q, a parameter reflecting nonlinear dissipative mechanisms, does not add valuable information to the measurement of nonlinear elasticity reflected by α_f.

- The absence of variation of linear elastic properties. As reported previously the elastic properties are almost constant during fatigue cycling [9].

![Figure 1: Normalized nonlinear elastic and dissipative coefficient and normalized mechanical modulus (linear elastic beam theory) as a function of damage step](image_url)

CONCLUSION

Of note is the good response of the non linear ultrasonic parameters, and particularly of α_f during fatigue cycling, in line with the evolution of E_{LEBT}. E_{LEBT} has been shown to be an indicator of fatigue micro-damage accumulation [8-10]. Thus, the results of this study suggest that monitoring of fatigue microdamage in cortical bone can be achieved non invasively using nonlinear elastic wave spectroscopy techniques. Further validation should come from μCT investigations of the specimens enrolled in this study.

ACKNOWLEDGEMENTS

This research was supported by the Agence Nationale pour la Recherche (ANR), France (Grant BONUS_07BLAN0197).

REFERENCES