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**Nonlinear ultrasound monitoring of single crack propagation in cortical bone**

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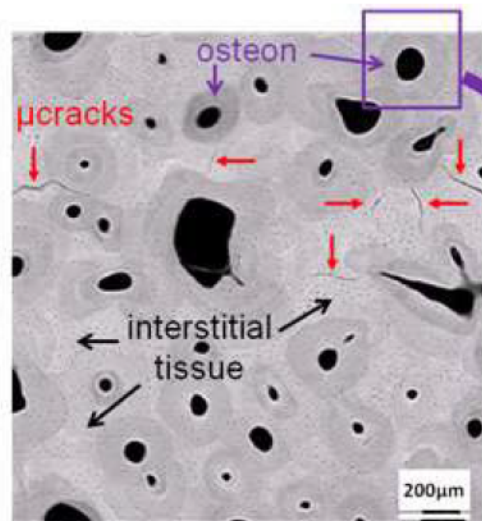
Accumulation of bone microdamage is suspected to lead to severe impairment of mechanical properties with an increase in skeletal fragility and fracture risk. The objective of the study was to evaluate the sensitivity of nonlinear resonant ultrasound spectroscopy (NRUS) measurements to the propagation in cortical bone of a single microcrack induced by 4-point bending mechanical loading. Twelve human cortical bone specimens were machined as parallelepiped beams (50\*2\*2mm) to unambiguously identify resonant modes for NRUS measurements. A central notch of 600 $\mu$ m was made to control crack initiation and propagation during four-point bending loading. The nonlinear hysteretic elastic coefficient ( $\alpha_f$ ) was derived from NRUS measurements achieved in dry and wet conditions. Each bone specimen was probed by a swept-sine around its first compression mode, applying progressively increasing drive levels. Moreover, the buried crack length was assessed by synchrotron radiation micro-computed tomography with a spatial resolution of 1.4 $\mu$ m. Despite between-sample variability,  $\alpha_f$  increased significantly in the damaged state (44.9 $\pm$ 85.4) compared to the initial value (5.5 $\pm$ 1.5) in the control undamaged state. Crack length was significantly correlated to the nonlinear elastic parameter  $\alpha_f$  ( $r^2=0.78$ ,  $p<0.001$ ). These results suggest that NRUS is sensitive to damage accumulation and can be used as a marker of bone damage.

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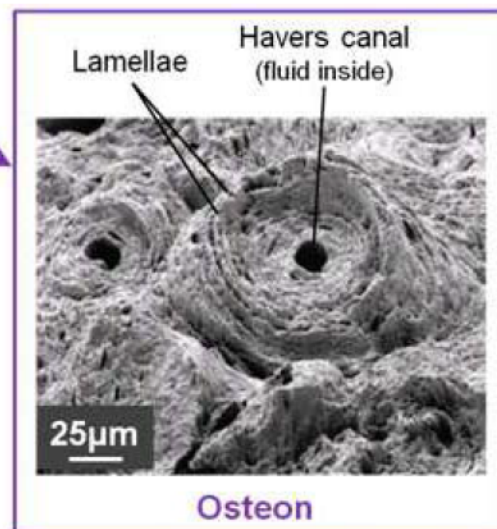
## I. Introduction

### a) *Microdamage in cortical bone*

- Two types of bone microdamage coexist: the **microcrack** (50-500 $\mu$ m) and the so-called diffuse damage. In this study, we will focus on microcracks in human cortical bone.



Histomorphometry by synchrotron radiation  $\mu$ CT (@ ESRF)



Excerpt from [1]

### b) *Why assessing bone microdamage?*

- **Context:**

**Bone microdamage** accumulation is suspected to have important consequences

1. on bone **mechanical** properties => **Increasing** of skeletal fragility and **risk of fracture**.
2. on bone **biological** properties => **initiating** bone remodeling process

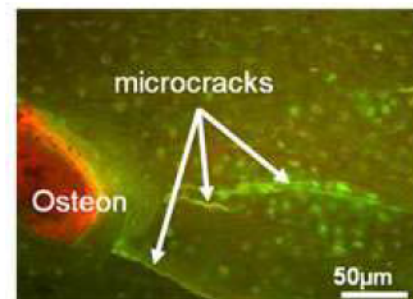
### c) *How to evaluate bone microdamage?*

- **Current growing interest in bone microdamage assessment:**

- Reference technique:

Histomorphometry by optical microscopy  
(conventional or by epifluorescence)

- ⇒ Destructive, 2D, time consuming and operator-dependent

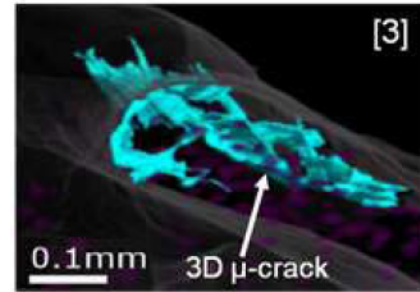
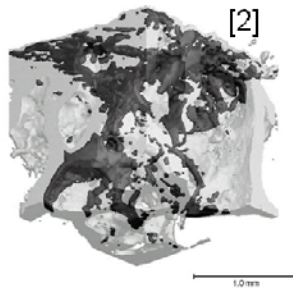


Epifluorescence microscopy (@ LIP)

- New techniques are emerging:

$\mu$ CT with contrast agent [2], Synchrotron radiation  $\mu$ CT [3]

⇒ Volumetric information but not suitable for *in-vivo* measurement



⇒ **Nonlinear acoustic techniques is a promising solution [4-6]**

#### d) *Nonlinear acoustics*

- **Advantages of nonlinear acoustics methods :**

⇒ Very sensitive to microcracks and microdamage (in composites, concrete, metals...)

⇒ Non-destructive techniques (possible transfer to the *in-vivo*)

- **Two types of elastic nonlinearity [7]:**

$$K(\epsilon) = K_0 \left[ (1 + \underbrace{\beta \epsilon + \delta \epsilon^2 + \dots}_{\text{Classical nonlinearity}}) + \underbrace{\alpha (\Delta\epsilon, \epsilon')}_{\text{Hysteretic nonlinearity}} \right]$$

Linear elastic modulus
Classical nonlinearity
Hysteretic nonlinearity

- **Classical nonlinearity (atomic origin) << hysteretic nonlinearity (cracked materials)**

⇒ In this study we investigate the nonlinear hysteretic  $\alpha$  parameter as its nonlinearity is known to be very sensitive to damage in materials.

#### e) *Preliminary results*

- **Previous *in vitro* nonlinear ultrasonic measurements in cortical bone:**

- **First results:** Nonlinear Resonant Ultrasound Spectroscopy (NRUS) method [4] was used to evaluate bone damage accumulation on human cortical bone diaphysis having undergone fatigue compression cycling.

⇒ Nonlinear elastic parameter ( $\alpha$ )  $\nearrow$  after fatigue  $\nearrow$

⇒  $\alpha$   $\nearrow$  with age (and its was observed that microcracks density also  $\nearrow$  with age)

⇒ **But critical issue:** No bone microdamage evaluation and quantification

- **Recent results:** A group of cortical bone specimens having a calibrated parallelepiped shape were taken through a progressive fatigue protocol consisting of four steps of cyclic four-point bending. Microdamage quantification by NRUS and SR- $\mu$ CT was performed on the same set of samples.
  - $\Rightarrow$  NRUS is sensitive to **non-localized** bone **micro-damage** accumulation.
  - $\Rightarrow$  Fatigue test induced **early damage** by increasing the density of **small microcracks**
  - $\Rightarrow$  For the 1<sup>st</sup> time: significant **correlation** between the variation of  $\alpha$  and the variation of **small microcracks** density.
  - $\Rightarrow$  **Linear** Young's modulus **not sensitive** to **early** bone microdamage

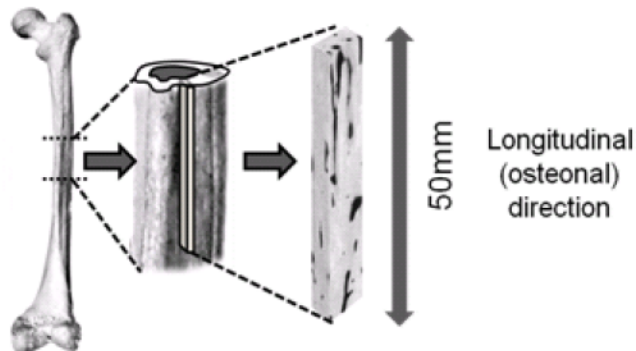
### **Aim of this study**

1. To assess NRUS sensitivity to bone **single** and **localized** crack **propagation**
2. Clarify the **relationship** between NL hysteretic parameters and crack characteristics

## **II. Material & methods**

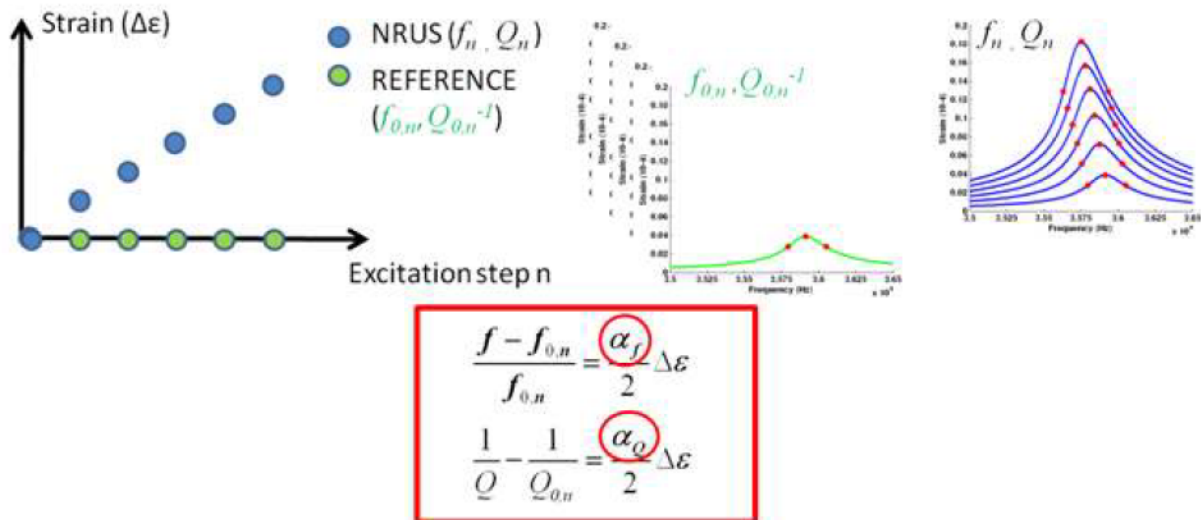
### **a) Human cortical bone samples**

- 14 parallelepiped beams ( $50 \times 2 \times 2 \text{mm}^3$ ) from 4 femoral mid-diaphysis in order to :
  - $\Rightarrow$  **Control** damage during 4-point bending test (*toughness*)
  - $\Rightarrow$  **Identify resonant modes** for NRUS measurements



### **b) Sensitive NRUS protocol:**

- **Tracking** reference frequency  $f_{0,n}$  and damping  $Q_{0,n}^{-1}$  variations during the NRUS experiment by measuring  $f_{0,n}$  and  $Q_{0,n}^{-1}$  before each increasing amplitude step. This protocol allows to correct small variations of the references due to environmental fluctuation (i.e. temperature changes) and/or material conditioning [8].

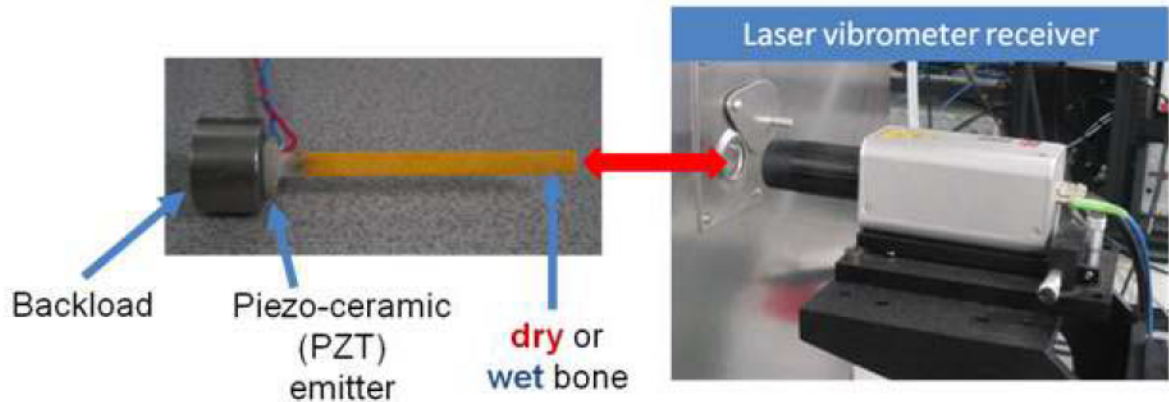


- ⇒ High sensitivity ( $\Delta f/f_0$  limit =  $10^{-5}$ ) and low variability (<20%)
- ⇒ Extraction of **2 hysteretic parameters**: elastic ( $\alpha_f$ ) and dissipative ( $\alpha_Q$ )

• **Experimental setup:**

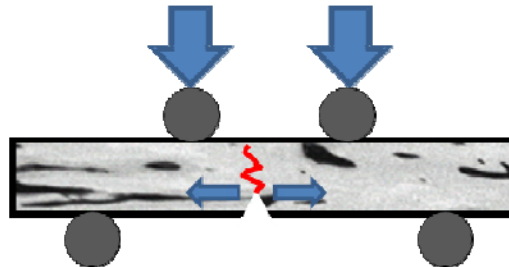
- **Emitter:** the piezo-ceramic applies progressively increasing drive levels ( $\varepsilon = 10^{-6} - 10^{-4}$ ) around the **1<sup>st</sup> compression mode** ( $\approx 15\text{kHz}$ ).
- **Receiver:** Resonant response measured by a laser vibrometer.

Note that the specimens were measured in **DRY** and **WET** conditions after toughness test.



### c) Toughness test

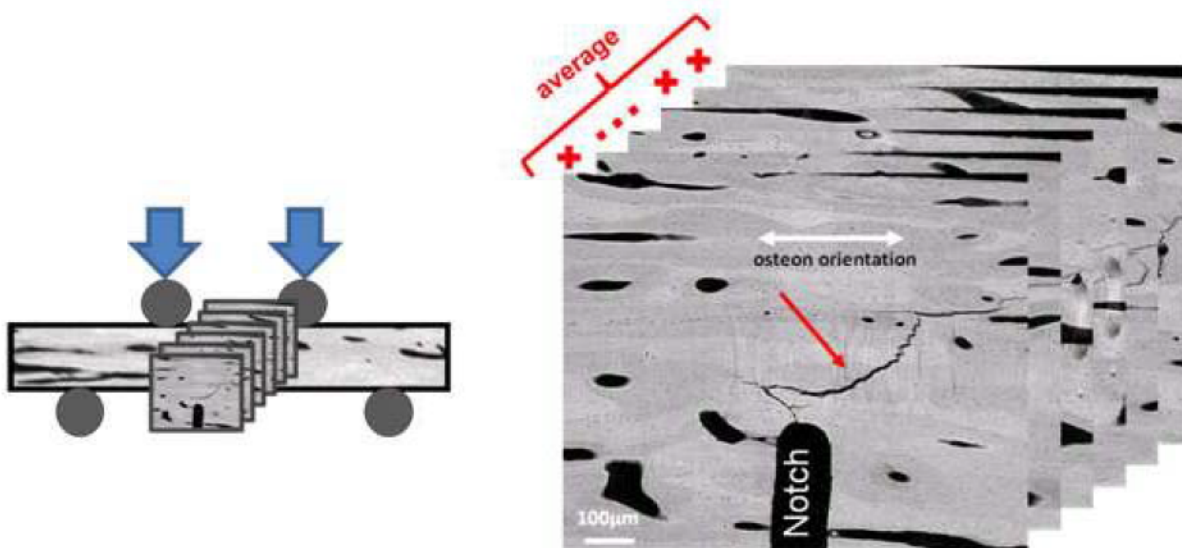
- The toughness test consists on a **single crack** initiation and propagation. The procedure is the following :
  1. Notch in the sample
  2. Quasi-static loading:  $\mu$ crack initiation and growing
  3. Stop loading before the rupture



⇒ The toughness test was stopped at different loading stage for each specimen in order to get samples with different crack lengths (Cr.Le)

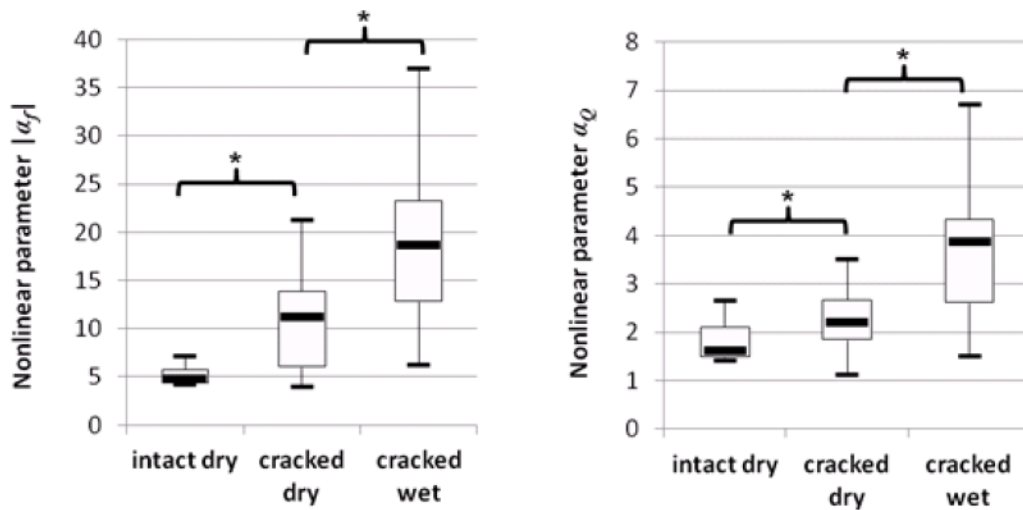
### d) Histomorphometry

- Histomorphometry was performed for the first time using **synchrotron radiation  $\mu$ CT (SR- $\mu$ CT)** [3]:
    - One region of interest (ROI) of  $2.15 \times 1.75 \times 1.75 \text{mm}^3$  encompassing the notch area
    - Six 2-D longitudinal SR- $\mu$ CT cross-sections were extracted from the ROI volume (resolution=  $1.4 \mu\text{m}$ ; surface =  $4 \text{mm}^2$ )
- ⇒ **Evaluation of the average single crack length: Cr.Le [ $\mu\text{m}$ ]**



### III. Results

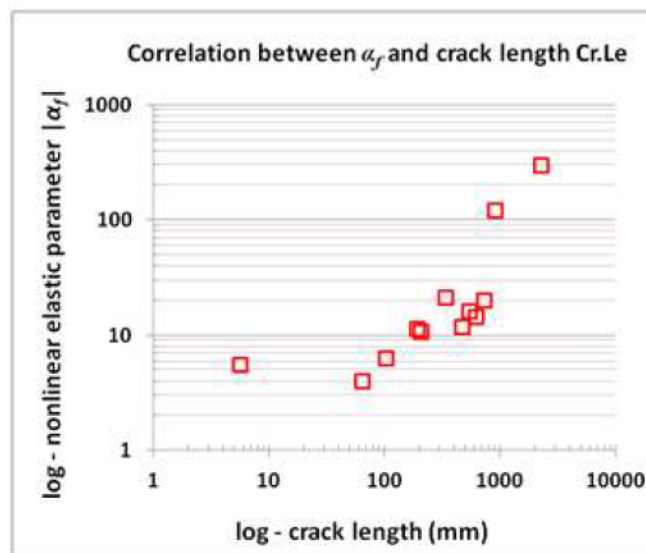
#### a) Nonlinear ultrasonic parameters



- ⇒ Both nonlinear parameters increase significantly ( $\alpha_f$  :  $p < 0.006$ ;  $\alpha_Q$  :  $p = 0.02$ ) after crack propagation.
- ⇒ Both nonlinear parameters are significantly higher when bone specimens are wet ( $\alpha_f$  :  $p < 0.002$ ;  $\alpha_Q$   $p < 0.001$ )

**Hypothesis:** the variation of the nonlinear hysteretic behavior could be due to capillarity forces [9] and/or bone organic phase (collagen) properties modification.

#### b) Histomorphometry



- ⇒ Crack length Cr.Le ranges between  $6\mu\text{m}$  to  $2277\mu\text{m}$
- ⇒ **Significant correlation** ( $r^2=0.8$ ;  $p < 0.01$ ; Spearman test) **between Cr.Le and  $\alpha_f$**  (measured in dry condition)



#### IV. Discussion and Conclusion

- Our results evidence:
  - Nonlinear elastic  $\alpha_f$  and dissipative  $\alpha_D$  parameters are **sensitive** to **single crack** propagation. This is in agreement with other studies conducted on steel [10, 11] or concrete [12].
  - Hysteretic elasticity  $\nearrow$  with bone **hydration**. This was also observed in rocks [13].
  - Similar trends for the first 3 compression modes (*data not shown*). This confirms the robustness of the results which are independent on the compression modes.
- For the 1<sup>st</sup> time:
  - Successful **concurrent assessment** of an ultrasonic nonlinear parameter ( $\alpha_f$ ) and bone crack length (**Cr.Le**).
- Next step:
  - Take into account other single crack parameters (volume, **thickness...**)
    - ⇒ Better correlation with nonlinear parameters?

#### V. Acknowledgement

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