EXPERIMENTAL AND NUMERICAL ANALYSIS OF THE TENSILE TEST ON CORTICAL BONE DEMINERALIZATION

Cristiane E. R. da Silva, Instituto Nacional de Tecnologia (INT), cristiane.evelise@int.gov.br
Guido R. de A. Graça, Instituto Nacional de Tecnologia (INT), guido.rezende@int.gov.br.
Jorge A. Hinostroza Medina, Instituto Nacional de Tecnologia (INT), jorge.arturo@int.gov.br
Claudio T. dos Santos, Instituto Nacional de Tecnologia (INT), claudio.santos@int.gov.br
Mauricio J. Monteiro, Instituto Nacional de Tecnologia (INT), mauricio.monteiro@int.gov.br.

Abstract. *Osteoporosis is an osteometabolic disease characterized by bone demineralization and is associated with an increased risk of fracture. Bone demineralization was chemically induced using ethylenediaminetetraacetic acid, with immersion times of 1 h, 2 h and 4 h to evaluate the variation of the ultimate tensile strength. Cortical bone tissues were evaluated from 14 fresh bovine femurs from different races and ages. These tests were performed in a universal machine with100 kN capacity and were conducted on a displacement rate of 2.5 mm/min. Due to the bones irregular geometry and specimens asymmetry, a numerical simulation was performed considering the actual specimens geometry after machining. There was no great variation in relation demineralization time and average ultimate strength values, however, in individual specimens compared to their respective controls a decrease percentage around 16% was observed. The numerical simulation faithfully reproduced the results of the stress field in the evaluated specimens.*

Keywords*: Osteoporosis. Tensile Testing. FEA. Cortical Bone.*

1. INTRODUCTION

Osteoporosis is an osteometabolic disease characterized by bone demineralization and is associated with an increased risk of fracture. These fractures most often affect the elderly population and can occur anywhere in the skeleton. The fracture strength of the bones is related to the degree of deformation of the existing elements in the multiple scales of the bone tissue, being an important indication of bone quality. Synergistic factors, such as: tissue composition; arrangement of structural features; degree of damage are responsible for bone quality (Cowin, 2010).

The life expectancy of the world's population is increasing annually. In Brazil, data from the Brazilian Institute of Geography and Statistics (IBGE, 2017) show that in 40 years the elderly population will triple and increase from 19.6 million (10% of the Brazilian population in 2010) to 66.5 million in 2050 (29.3%), impacting on profound changes in public health policies. Thus, being able to understand bone change over aging will help to diagnose osteoporosis earlier and avoid the risk of predicted fractures.

Due to the complexity of the topic, a collaborative network called PIPEDO - Interdisciplinary Project for the Study of Bone Demineralization has been formed within this project. The network is currently made up of: Laboratory of Characterization of Mechanical and Microstructural Properties (DIEMP - INT); Laboratory of Powder Technology (DIPCM-INT); Ultrasound Laboratory (Inmetro); Laboratory of Digital Image Processing (DEQM - PUC-Rio); Laboratory of Nano and Microfluidics and Microsystems (LabMEMS - COPPE-UFRJ).

In this sense, the initial results of tensile tests and numerical simulation performed in the INT will be presented, which will be used to validate the biomechanical results obtained by Quantitative Ultrasound, an important non-destructive technique with potential use in predicting fractures and bone quality in the early diagnosis of osteoporosis.

2. MATERIALS AND METHODS

Cortical bone tissues were evaluated from 14 fresh bovine femurs from different races and ages. Samples were cleaned for soft tissue removal using 30 vol. hydrogen peroxide. Afterwards, they were cut in the region of the mean diaphysis (Fig. (1)) to obtain the specimens (SP), using an abrasive disk cutting machine. We followed the nomenclature SP_X_Y, where X is the origin femur and Y is one of the four parts.

Figure 1. a) Samples cut from the mid-diaphysis b) ASTM D638 -type IV specimens.

The tensile test specimens were machined according to ASTM D638 type IV (Fig. (1b)) in the Laboratory of Nano and Microfluidics and Microsystems (LabMEMS-Coppe UFRJ) using a Minitech Mini-mill / GX model Minitech Corporation.

2.1. Bone demineralization

Bone demineralization was chemically induced using 0.1 M ethylenediaminetetraacetic acid (EDTA), pH 11. Immersion times of 1 h, 2 h and 4 h in the first 24 h were used to evaluate the slowly process.

2.2. Tensile Testing

Tensile tests were performed in order to obtain the ultimate tensile strength of evaluated bones. These tests were performed in a universal machine INSTRON model 3382 with 100 kN capacity and were conducted on a displacement rate of 2.5 mm/min. Figure (2a) shows the assembly and execution of the test. 07 SPs were tested before the demineralization and 03 SPs in each demineralization step. Nevertheless, it was used the Outlier testing for analyzing of outliers. Therefore, the SP_04_1_2h was disqualified for the study. The manufacture of the custom clamping claws to the geometry of the SPs was also developed in this project and a previous analysis of the fixation and stress concentration in the assembled SP was performed using numerical simulation (Fig. (2b)).

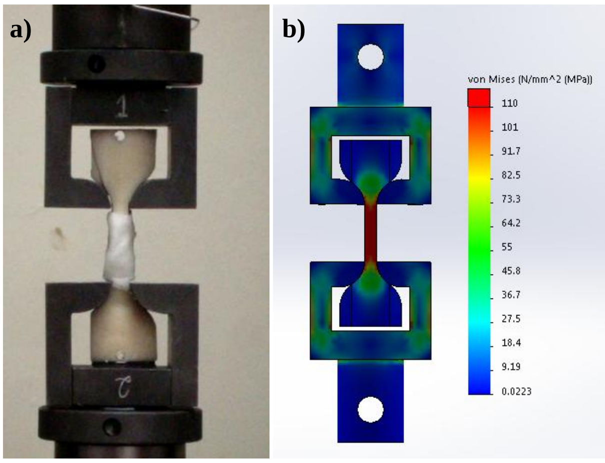

Figure 2. a) Tensile testing scheme, b) Assembly and simulation, before testing.

2.3. Numerical Simulation

Due to the bones irregular geometry and SPs asymmetry, the stresses field is not homogeneous and the fracture in the useful region of SPs is not guaranteed. Therefore, a numerical simulation was necessary considering the actual geometry of the SPs after machining. The 3D Range Vision optical scanner was used, this device is based on a stereo vision approach with structured light projection. The optical unit is composed of two digital cameras (1800 pixels) and a white light projector (1920 x 1080 pixels). The resolution achieved was 0.0012 mm. Figure (3) shows this procedure, with the SP on the rotating base on the left and the scanner on the right. After the scanning, the obtained image should be treated. The images were independent and had to be added manually and remove noise through ScanMerge 3D software from Range Vision itself. Then the model were extracted in STL format and imported into Solidworks® software. The discretization of the geometry is performed using 3D tetrahedral elements with size of 1 mm. The boundary conditions applied were fixation of the lower surface of the body and loading of 5kN on the upper surface, attempting to reproduce the conditions of a tensile testing (Fig. (5)). The mechanical properties used were based in FENG and JASIUK, 2010, which is an isotropic linear elastic material modeling. The values of Elastic Modulus (E) and shear modulus (G), as well as the Poisson coefficient (υ) are given in Tab. (1).

Table 1. Mechanical properties of cortical bone.

Model	Elastic Modulus, E GPa Longitudinal	Shear Modulus G, GPa Longitudinal	Poisson's Coefficient, υ
Isotropic Cortical Bone	17.4	5.0	0.20

Figure 3. Specimens scanning setup.

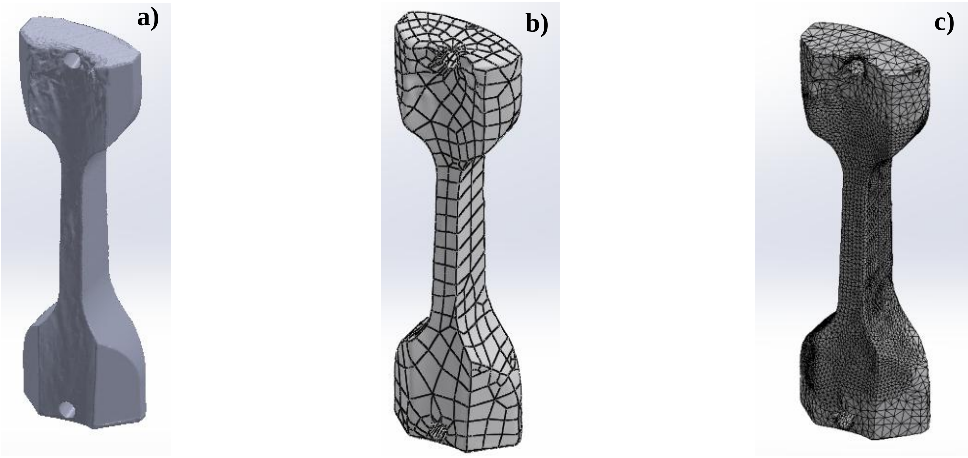

Figure 4. Trimetric view of SP 10_4. a) STL b) CAD Model c) FEM Mesh

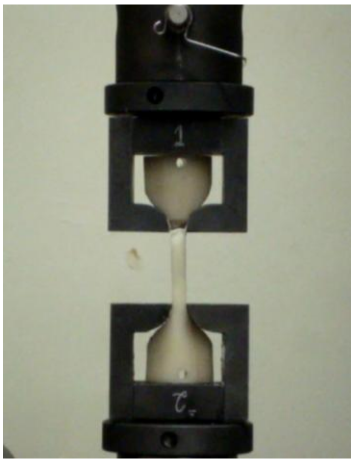

Figure 5. Tensile testing fracture.

2.4. Uncertainty Estimation

The ultimate strength in a specimen is a function of the area (S) and applied load (F). Using the Eq. (1), (2), (3) the measurements uncertainty (u_c) was calculated (Gabauer, 2000).

$$u_c(\sigma) = \sqrt{c_F^2\, u_F^2 + c_S^2\, u_S^2} \quad (1)$$

$$c_F = \frac{1}{S} \quad (2)$$

$$c_S = -\frac{F}{S^2} \quad (3)$$

The load uncertainty value u_F depends on the instrument used in the test. This uncertainty value was obtained from certified of calibration number 17062701DM. Thus, u_F = 0.0017 * F (range of load = 10 kN).

The instrument used to measure the dimensions of the cross-section area of the specimens was a caliper with a resolution of one-hundredth of a millimeter, i.e. R = 0.01mm / √12 = 2.8868 x10^{-3} mm (rectangular distribution). The Type A uncertainties were estimated as the standard deviation from 5 repeated measurements, divided by / √5. The measurements expanded uncertainty estimation was based on Guide to the Expression of Uncertainty in Measurement (GUM) (Inmetro, 2008).

3. RESULTS

3.1 Tensile Testing

The presented results will be important for correlations with the other techniques evaluated, mainly the biomechanical obtained results by quantitative ultrasound. In Tab. (2), are shown ultimate tensile strength and their respective expanded uncertainty (U) (MPa) and average ultimate tensile strength (MPa) before and after demineralization. The results of the specimens without demineralization had their ultimate tensile strength ranging from 83.3 MPa to 139.8 MPa, and measurements expanded uncertainty among 1.6 to 4.3 MPa. These results point to a variation that may be due to the different origin of the animals, anisotropy of the biological material and even the irregular thickness of the specimen (Feng *et al*, 2010). Besides, the average ultimate tensile strength after demineralization show almost none variation compared to the intact bones, it can be observed that after 2 and 4 h of demineralization there was a drop of ~ 16 % and 19 %, respectively compared to their respective controls. Throughout this project will be carried out tensile tests in 52 SPs, which increase the database and, consequently, will allow a better analysis of the relationship between the degree of bone demineralization and the mechanical properties.

Table 2. Ultimate tensile strength results, before and after demineralization.

Condition test	Demineralization Time [h]	Bone	Ultimate tensile strength and Respective Expanded Uncertainty (U) [MPa]	Average ultimate tensile strength and Standard Deviation [MPa]	Ultimate tensile strength loss after demineralization compared to the control [%]
Intact Bone	0	SP_8_4	102.9 ± 2.2	110 ± 18	Not applicable
		SP_13_3	123.2 ± 2.4		
		SP_11_3	101.8 ± 3.1		
		SP_07_2	139.8 ± 3.7		
		SP_10_4	100.3 ± 3.1		
		SP_12_3	116.7 ± 4.3		
		SP_14_3	83.3 ± 1.6		
Demineralized Bone	1	SP_14_1	53.6 ± 1.0	76 ± 20	-36
		SP_14_2	84.7 ± 2.1		2
		SP_14_4	90.2 ± 1.8		8
	2	SP_11_1	85.9 ± 2.6	101 ± 22	-16
		SP_7_1	116.4 ± 3.7		-17
	4	SP_8_1	82.9 ± 1.7	106 ± 24	-19
		SP_12_4	130.8 ± 2.7		12
		SP_10_2	103.3 ± 2.4		3

3.1. Numerical simulation

Figure (6) shows a simulation of SPs after scanning, in the case of SP 10-04. The results of the equivalent stress distribution (von Mises) show a concentration around 127 MPa close to the contact area between the SP and the claw and in the region of thinnest lower SP thickness. This condition was repeated by inverting the loading and fixation conditions. As expected, the fracture was located at the thickness, as shown in Fig (6) c, and it was confirmed in laboratory tests (Fig. (5)).

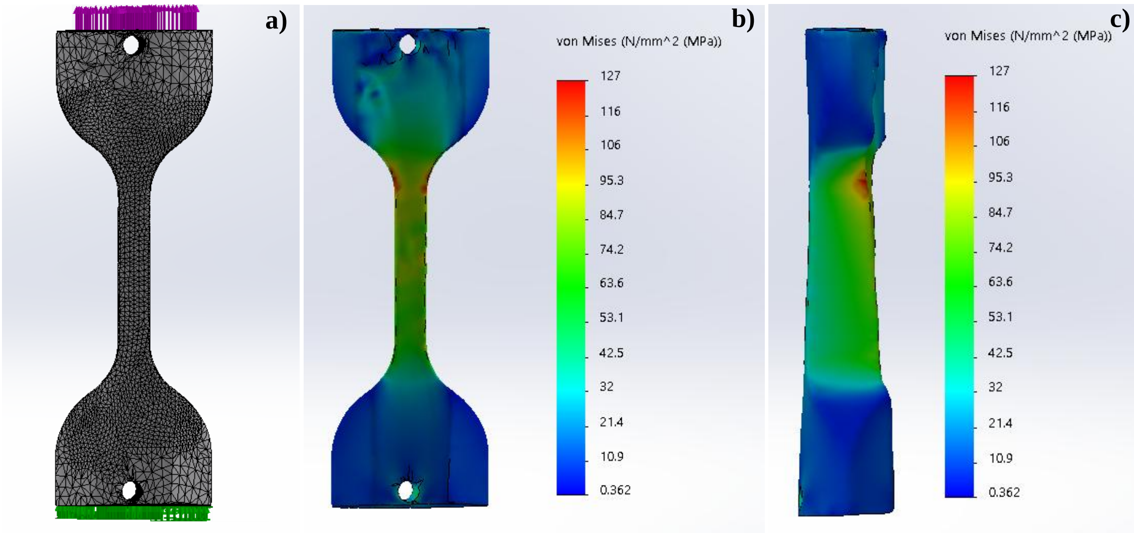

Figure 6. a) Discretization and boundary conditions, b) stress distribution, frontal and left lateral view (c) Detail of the SP irregular thickness.

4. CONCLUSION

There was no great variation in relation demineralization time and average ultimate strength values, however, in individual SPs compared to their respective controls a decrease percentage around 16% was observed. The numerical simulation faithfully reproduced the results of the stress field in the evaluated SPs. This tool will be used in subsequent analyzes to optimize the geometry of the SP and thus ensure the fault in the central region, which enables to validate the calculation of the elastic modulus. In addition, it will be evaluated geometric models obtained from computerized micro-tomography in order to consider the internal defects of the SP and thus be able to accurately predict the actual behavior of these specimens.

5. REFERENCES

ASTM. AMERICAN SOCIETY FOR TESTING AND MATERIALS. ASTM D638: Standard Test Method for Tensile Properties of Plastics. AMERICAN SOCIETY FOR TESTING AND MATERIALS,

COWIN, S. & Telega, **J. Bone Mechanics Handbook**, 2nd Edition. -. Appl. Mech. Rev. 56, B61, 2003.

GABAUER, W. The Determination of Uncertainties in Tensile Testing; Voest-Alpine Stahl Linz GmbH: Linz, Austria, 2000.

INMETRO, I. N. TECNOLOGIA, Q. E. GUM 2008 - Guia para a expressão de incerteza de medição. [S.l.]: [s.n.], 2008.

IBGE. “Perfil dos Idosos Responsáveis pelos Domicílios”. Disponível: https://ww2.ibge.gov.br/home/presidencia/noticias/25072002pidoso.shtm#sub_economia, Setembro/2017. Acesso em: 18 out. 2017.

FENG, L., JASIUK, I., “Effect of specimen geometry on tensile strength of cortical bone”. **J. Biomed. Mater. Res.** 95A. 580-587. 2010.

6. ACKNOWLEDGES

The authors acknowledge the support of MCTIC/CNPq, Robson Oliveira Centeno, Wellington Gilbert Fernandes and Rafael de Abreu Vinhosa from DIEMP-INT and thanks LabMEMS-Coppe UFRJ for provision of the specimen, Jorge Lopes for the scanning support and Unesa for demineralization bone.

7. RESPONSIBILITY FOR INFORMATION

The authors are solely responsible for the information included in this work.