**University of Southern Queensland**

**Faculty of Engineering and Surveying**

# THE PRELIMINARY STUDY ON THE MECHANICAL PROPERTIES OF HEAT-TREATED BOVINE BONE USING EXPERIMENTAL AND SIMULATIONS APPROACHES

A dissertation submitted by

**Mei-ling Lau**

For the award of
**DOCTOR OF PHILOSOPHY**

2013

# ABSTRACT

A critical factor that leads to bone fracture is the deterioration of bone quality. For a severe bone fracture that incurs a loss of volume, bone is unable to recover and bone grafting may be needed. Heat-treatment of bone is proposed as one of the most reliable and simple sterilisation methods to overcome the risk of rejection and disease transfer during transplantation.

The mechanical properties of bone at the micro-structural level after heat-treatment are not well characterised. To address this, this study investigated the localised mechanical properties of micro-structural tissues with the global structural level at different pre-set temperature ranges. Bovine cortical bone was used in this study as it has similar structure and morphology to human bone.

The results of the nanoindentation test demonstrated that heat-treated cortical bones can maintain relatively high elastic modulus (E) and nanoindentation hardness (H) among values between of $90^0$C to $150^0$C as compared to those of pristine bone. A significant increase of 44% (longitudinal) and 23% (transverse) of E values were found when compared to pristine bone. Also, an increase of 43% and 38% of H values in longitudinal and transverse directions respectively were found when compared to pristine bone. Furthermore, the E and H values of interstitial lamellae in this study at various temperatures are from 18.4 to 30.5 (GPa) and 0.84 to 1.27 (GPa),

respectively. The E and H values of osteon are from 18.6 to 28.8 (GPa) and 0.83 to 1.25 (GPa), respectively.

In the current study, compressive testing was employed to measure the global stiffness (E) of the bone samples. When heated at 150ºC, the bone specimens showed an increase of 60% in stiffness (E) and an increase of 26% in yield stress. On the other hand, when heated at 90ºC, a slight increase of 11.4% in stiffness (E) and 21.5% in yield stress was recorded.

Backscattered Electron (BSE) imaging was conducted to examine the relationship between mineral content and mechanical strength within the nanoindentation regions. The data demonstrated that the non heat-treated bones obtained the highest calcium wt% amongst the three groups. As temperature increased, there was a slight decrease in calcium wt%; however, the changes were not severe in this study.

Thermal gravimetric analysis (TGA) was used to investigate the condition of organic constituents of the bovine cortical bone. The TGA results demonstrated that heat-treated bones had three stages of weight loss. The first stage was the loss of water, which started from room temperature to 160°C. The second stage included a weight loss of organic constituents starting from 200°C to 600°C. Upon reaching 600°C, the organic constituents were decomposed and mineral phase loss started taking place until 850°C.

Computational modeling – finite element analysis (FEA) was conducted to investigate the relationship between the porosity and the mechanical properties of two main components of the cortical bone. Varying the diameters of the Haversian canal and the distribution of Volkman's canals in osteonal bone models showed a significant difference. This means that the increase of the porosity apparently affected the elastic modulus of cortical bone. This validated FE model is able to simulate the bone properties with the consideration of different bone porosity and its heterogeneous mechanical properties in osteonal and interstitial bone's longitudinal and lateral directions.

Suggestions for further study of the mechanical and chemical properties of heat-treated cortical bone for clinical applications are presented.

# ASSOCIATED PUBLICATIONS

The following publications were produced during the period of candidature:

**Journal Papers:**

**Mei-Ling Lau**, Kin-Tak Lau, Yan-Dong Yao Yeo, Chi-ting Au Yeung, and Joong-Hee Lee, "Measurement of Bovine Bone Properties through Surface Indentation Technique", *Materials and Manufacturing Processes*, No. 25, 2010, pp 324–328.

**Mei-Ling Lau**, Kin-Tak Lau, Harry Ku, Debes Bahattacharyya and Yan-Dong Yao, "Measurements of Heat treatment Effects on Bovine Cortical Bones by Nanoindentation and Compression Testing", *Journal of Biomaterials and Nanobiotechnology*, No. 3, 2012, pp 105-113

**Mei-Ling Lau**, Kin-Tak Lau, Harry Ku, Francisco Cardona and Joong-Hee Lee, "Analysis of Heat-treated Bovine Cortical Bone by Thermal Gravimetric and Nanoindentation", *Composites B (Accepted)*

**Mei-Ling Lau**, Kin-Tak Lau, Harry Ku, "Mechanical Properties of Heat-treated Bovine Cortical Bones by Nanoindentation", *Bones* (*Under reviewing*)

**Mei-Ling Lau**, Kin-Tak Lau, Yan-Sheng Yin, Lan Li, May Wong, Kevin Chan, and William Chen, "A Shape Memory Alloy Energy Absorber for Backpack Design", *Materials and Manufacturing Processes*, No.25, 2010, pp 281–286.

**Conference Paper:**

**Mei-Ling Lau**, Kin-Tak Lau and Yan-Dong Yao Yeo, "Experimental Measurement of Cortical Bone Properties", *The 17th International Conference on Composites or Nano Engineering (ICCE-17), Hawaii, U.S.,* July 2009.

**Mei-Ling Lau**, Kin-Tak Lau, Yan-Dong Yao and Debes Bahattacharyya, "A Study on Bone Properties using Nanoindentation Technique", *Processing and Fabrication of Advanced Materials XIX (PFAM-19)*, New Zealand, January 2011.

**Mei-Ling Lau**, Kin-Tak Lau, Harry Ku, Debes Bahattacharyya and Joong-Hee Lee "Assessing Heat-treatment Effects on Bovine Cortical Bones by Nanoindentation", *Processing and Fabrication of Advanced Materials XX (PFAM-20)*, December 2011.

# CERTIFICATION OF DISSERTATION

I certify that the ideas, designs and experimental work, results, analyses and conclusions set out in this dissertation are entirely my own effort, except where otherwise indicated and acknowledged.

I further certify that the work is original and has not been previously submitted for assessment in any other course or institution, except where specifically stated.

Sindy, Mei Ling Lau

0061014581

Signature of Candidate

Date

## ENDORSEMENT

Signature of Principle Supervisor

Signature of Associate Supervisor

Date

27/02/2013

Date

# ACKNOWLEDGEMENTS

I would like to express my most sincere appreciation to my PhD project supervisors Dr. Harry Ku and Prof. Alan Kin-Tak Lau for all their support, encouragement, valuable inputs and guidance provided at every stage of this thesis. I would also like to extend my gratitude toward the entire departmental and technical staffs, including Dr. Francisco Cardona and Mohan Trada for their assistance and support in using facilities and materials for conducting the experimental work. Special thanks to Annmaree Jackson for her contribution to edit the thesis and to all people who have been actively involved in carrying out this thesis and those who have given me this opportunity to do this thesis.

In addition, I wish to express my deepest appreciation to my family and sisters Kit Hing, Mei Lan and Mei To for their constant support throughout the study. I would like to extend my appreciation to my friends for their encouragement. Special thankful and grateful to Ahmed Naji for his supported and encouraged me all these days.

A particular appreciation is also extended to the University of Southern Queensland and the Centre of Excellence in Engineered Fibre Composites for financial support.

# TABLE OF CONTENTS

# LIST OF FIGURES

# LIST OF TABLES

# LIST OF ABBREVIATIONS

| | |
|---|---|
| 2-D | Two-dimensional |
| 3-D | Three-dimensional |
| AFM | Atomic force microscopy |
| ANOVA | Analysis of variance |
| BMU | Basic multicellular unit |
| BSE | Backsattered electron |
| Ca | Cacium |
| Co-Cr | Cobalt-chromium alloy |
| CT | Computed tomography |
| *d* | Gauge diameter |
| DOF | Degree of Freedom |
| E | Young's modulus or modulus of elasticity |
| EDX or EDS | Energy dispersive X-ray spectroscopy |
| FEA | Finite element analysis |
| FEM | Finite element modeling |
| GH | Growth hormone |
| H | Hardness |
| HA | Hydroxyaptite |
| HIV | Human immunodeficiency virus |
| IOF | International Osteoporosis Foundation |
| L | Longitudinal |

| | |
|---|---|
| MR | Magnetic resonance |
| NI | Nodal interpolation |
| OI | Osteogenesis imperfect |
| P | Phosphorous |
| PBS | Phosphate-buffered Saline |
| PCL | Polycaprolactone |
| PE | Polyethylene |
| PGA | Poly (glycolic acid) |
| PLA | Poly (lactic acid) |
| PLGA | Poly (lactic-*co*-glycolic acid) |
| PLLA | Poly- L-lactic acid |
| PMMA | Polymethyl methacrylate |
| pQCT | Peripheral quantitative computed tomography |
| PTH | Parathyroid hormone |
| QCT | Quantitatively computed tomography |
| R-curve | Propagation toughness |
| RT | Room temperature |
| SD | Standard deviation |
| SEM | Scanning electron microscope |
| SHPB | Split Hopkinson Pressure Bar |
| T | Transverse |
| Ti | Titanium alloy |
| TCP | Tricalcium phosphate |
| TEM | Transmission electron microscopy |

| | |
|---|---|
| TFB | Treated femur bone |
| TRB | Treated rib bone |
| TGA | Thermal gravimetric analysis |
| UFB | Untreated femur bone |
| URB | Untreated rib bone |
| USA | United States of America |
| XRD | X-ray diffraction |

**Equation parameters:**

| | |
|---|---|
| A | Area |
| Ac | Projected contact area |
| Er | Reduce modulus |
| Ei | Elastic modulus of indentor |
| Es | Elastic modulus of specimen |
| νi | Poisson’s ratio of indentor |
| νs | Poisson’s ratio of specimen |
| Pmax | maximum indentation force |
| $\sigma$ | Compressive stress |
| $\varepsilon$ | Compressive strain |
| P | Applied force |
| S | Contact stiffness |
| $\xi$, $\eta$, $\zeta$ | Global degree of freedom |
| $u$,$v$,$w$ | Local degree of freedom |

**Unit:**

| | |
|---|---|
| % | Percent |
| $^{o}$ | Degree |
| $^{o}C$ | Degree Celsius |
| cm | Centimeter |
| $cm^3$/min | Cubic centimeter per minute |
| GPa | Giga pascal |
| kN | Kilo-newton |
| mg/dl | Milligrams per deciliter |
| mm | Millimeter |
| $mm^3$ | Cubic millimeter |
| mN | Millinewton |
| µm | Micron-meter |
| MPa | Mega pascal |
| Nm | Nanometer |
| wt% | Weight percent |