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

A dissertation submitted by

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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°C to 150°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 150°C, 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 90°C, 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:**


**Conference Paper:**


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

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Signature of Candidate

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Date

ENDORSEMENT

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Signature of Principle Supervisor

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Signature of Associate Supervisor

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Date

27/02/2013
Date
ACKNOWLEDGEMENTS

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<th>Description</th>
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<tr>
<td>2-D</td>
<td>Two-dimensional</td>
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<tr>
<td>3-D</td>
<td>Three-dimensional</td>
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<tr>
<td>AFM</td>
<td>Atomic force microscopy</td>
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<tr>
<td>ANOVA</td>
<td>Analysis of variance</td>
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<tr>
<td>BMU</td>
<td>Basic multicellular unit</td>
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<tr>
<td>BSE</td>
<td>Backsattered electron</td>
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<tr>
<td>Ca</td>
<td>Calcium</td>
</tr>
<tr>
<td>Co-Cr</td>
<td>Cobalt-chromium alloy</td>
</tr>
<tr>
<td>CT</td>
<td>Computed tomography</td>
</tr>
<tr>
<td>$d$</td>
<td>Gauge diameter</td>
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<tr>
<td>DOF</td>
<td>Degree of Freedom</td>
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<tr>
<td>E</td>
<td>Young’s modulus or modulus of elasticity</td>
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<tr>
<td>EDX or EDS</td>
<td>Energy dispersive X-ray spectroscopy</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite element analysis</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite element modeling</td>
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<tr>
<td>GH</td>
<td>Growth hormone</td>
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<tr>
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<td>Hardness</td>
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<td>IOF</td>
<td>International Osteoporosis Foundation</td>
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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:**

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<tr>
<td>A</td>
<td>Area</td>
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<td>Ac</td>
<td>Projected contact area</td>
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<tr>
<td>Er</td>
<td>Reduce modulus</td>
</tr>
<tr>
<td>Ei</td>
<td>Elastic modulus of indentor</td>
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<td>Contact stiffness</td>
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**Unit:**

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<tr>
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<td>Centimeter</td>
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<td>cm³/min</td>
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<td>GPa</td>
<td>Giga pascal</td>
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<tr>
<td>kN</td>
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<tr>
<td>MPa</td>
<td>Mega pascal</td>
</tr>
<tr>
<td>Nm</td>
<td>Nanometer</td>
</tr>
<tr>
<td>wt%</td>
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