# Effect of Age on the Fracture Behavior of Bovine Bone: an Analytical and Experimental evaluation

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① Fracture

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# DECLARATION

I, **Mr. Waseem Ur Rahman**, Reg. No. **17-FET/MSME/S14** student of MS mechanical engineering in Session 2014-2016, hereby declare that the matter printed in the thesis titled **"Effect of Age on the Fracture Behavior of Bovine Bone: an Analytical and Experimental evaluation"** is my own work and has not been printed, published and submitted as research work, thesis or publication in any form in any University, Research Institution etc. in Pakistan or abroad.

Waseem Ur Rahman:____________________

Dated: 22/09/2016

# Certificate of Approval

This is to certify that the work contained in this thesis entitled, **"Effect of Age on the Fracture Behavior of Bovine Bone: an Analytical and Experimental evaluation"** was carried out by **Waseem Ur Rahman Registration# 17-FET/MSME/S14,** it is fully adequate in scope and quality, for the degree of MS ( Mechanical Engineering ).

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# DEDICATION

I dedicate all my efforts to the **Holy Prophet Mohammad (S.A.W.),** who has been sent as a mercy to all mankind, my parents who have always supported me morally and financially, and have always prayed to Almighty ALLAH for my success, to my entire family members and to my respected teachers. I also dedicate it to my parents, brothers and sisters for their support and prayers.

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Praise to Almighty ALLAH, the most Benevolent, the Omnipotent and Merciful, the Creator of the Universe, who enabled me to accomplish this declaration. Blessing on MOHAMMAD (S.A.W), the seal of the prophets and his pious progeny. I am grateful to International Islamic University, Islamabad for providing me a chance to enhance my skills and knowledge through this thesis and I wish to express my deepest gratitude to my worthy and honorable supervisor **Dr. Rafiullah Khan** for his kind supervision with keen interest and valuable guidance. His professional understanding and profound knowledge and kind attitude helped me in compiling this dissertation. His constructive criticism and directional guidance always motivated me to work hard.

I am especially grateful to Dr. Altaf Ahmad and Mr. Muzamil Hussain for a helpful discussion during my research work. Also, I am especially grateful to Mr. Noor Rahman for guiding me in bone fracture research And in the end, I would like to pay my special thanks to my friend Mr. Habibullah who helped me in preparing my thesis.

I pray ALLAH to help those who helped and guided me from getting down to the conclusion of my dissertation

# ABSTRACT

Bone is a natural composite anisotropic material it composed of mainly three materials, organic, inorganic and water. Bone has developed to provide structural support to organisms, protection of internal organs, and transmission of forces and therefore its mechanical properties are of great physiological relevance.

The mechanical properties of the bones do not remain constant with age; rather, they change throughout in life. Literature review shows that mechanical properties of cortical and femur bones change with the passage of time. The literature further reveals that the age effect on hip bone is less investigated.

The objective of this thesis was to investigate age effects on fracture toughness, young modulus and ultimate strength of bovine's hip bone. The above properties were experimentally investigated using samples of hipbone from various categories of male bovine. American society for testing and materials ASTM E-399 were used to investigate fracture toughness while tensile strength and young modulus were investigated using ASTM D-3039. The strain fields at the crack tip of compact tension (CT) specimens were examined using digital image correlation technique. The image files obtained during the DIC analysis were processed using free software NCORR.

Results show that the properties increased from the youngest age to adulthood after which the properties decreased. The mechanical properties in transverse direction were higher as compared to longitudinal direction.

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# List of Abbreviations

ASTM: American Society for Testing and Materials
BMD: Bone Mineral Density
CT SCAN: Computerized Tomography Scan
CT: Compact Tension
DIC: Digital Image Correlation
FEM: Finite Element Modeling
GUI: Graphical User Interface
HA: Hydroxyapatite
LCT: Longitudinal Compact Tension
MRI: Magnetic Resonance Imaging
TCT: Transverse Compact Tension
UTM: Universal Testing Machine

# Chapter 1

# INTRODUCTION

## 1.1 Introduction

Bone is a complex natural hierarchical composite material with an anisotropic structure which consists organic material (cells, lipids), inorganic material (mineral crystal) and water [1]. Bone primarily contains approximately 70% mineral, mainly hydroxyapatite (HA), and 8% water by weight, 22% proteins (90% type I collagen) [2, 3]. In type-I collagen, include approximately 90% of organic matrix, imparts toughness and ductility to bone. The collagen fibers are composed of tropocollagen triple helical molecules that self-assemble into larger fibrils, a few hundred nanometers in diameter, and show the characteristic 67 nm D-periodicity as shown in figure 1.1. The key functions of bone include protection of internal organ [4, 5], mineral storage, support to the body [6], positions for muscle attachments and shock absorption.

The bone can be divided structurally into two types, cortical bone (compact bone) and trabecular bone, also known as spongy or cancellous. The cortical bone shows significant inelastic deformation, from outer sheet and high resistance to fracture, while trabecular bone is located in the areas that need to absorb energy that is ribs and skull.

The cortical bone consists of three subtypes, interstitial, osteonal and plexiform. The cortical bone is found in the diaphysis of the long bones and additionally as a protective layer outside the spongy bone. The osteonal bone is the basic unit of cortical bones. It is made up of cylindrical shape that is typically several millimeters long and approximately 0.2mm in diameter. The space between adjacent osteons is occupied with interstitial lamellae, which comprise of remnants osteons after remodeling. The plexiform bone is built of lamellar bone sheets that are perforated by a plexus of blood vessels [7]. The femur and tibia bones are investigated by different methods and technique. The hip bone is less investigated.

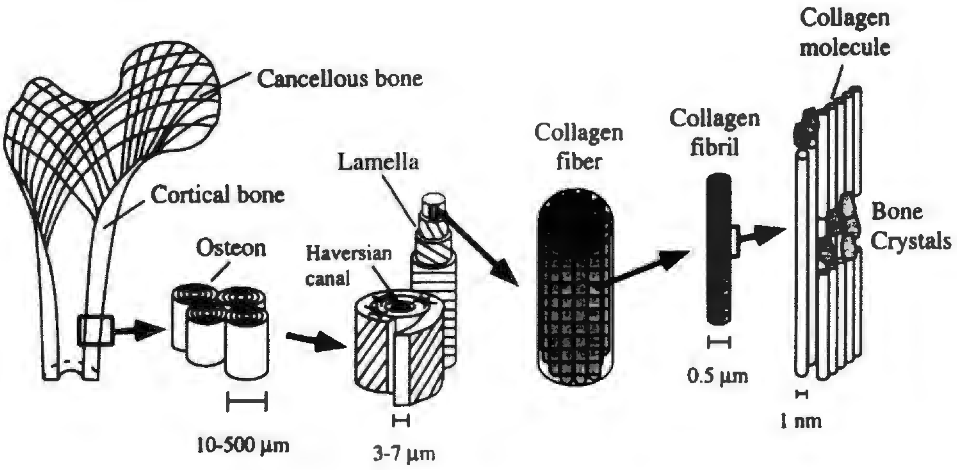


**Figure 1.1:** Hierarchical structure of organized bone [1].

## 1.2 Hip Bone

Hipbone is the major bone that constitutes pelvis. Embryologically it is made up of three bones ileum (the winged or expanded upper portion) ischium (the lowermost portion) and pubis the front or interior portion as shown in figure 1.2. After puberty, all these three bones start fusing forming one bone call hipbone. The hipbone articulates with the sacrum through sacroiliac joint, in between of two hipbones below both sides articulate with the femur to transfer the force of back bone to lower limbs. The joint of femur and hipbone is a ball and socket joint in which femur head serves as a ball and the hipbone acetabulum serve as socket. While sacroiliac joint is a fibrous joint which allow limited movement and is supported by a lot of strong ligaments [8].

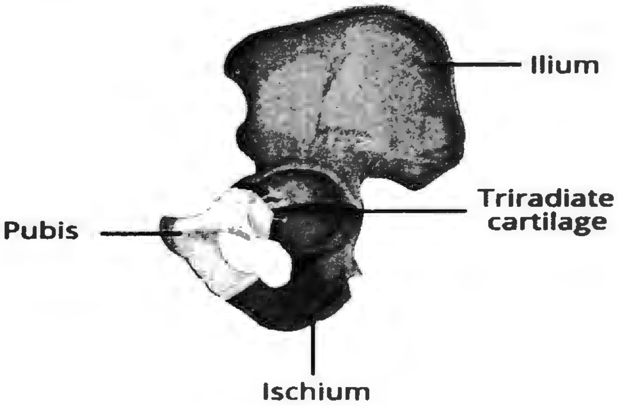


**Figure 1.2:** Hip Joint Bone structure [9]

Hipbone has broadened portion of the skeleton like skull, which allow movement of many vitally important pelvic organs like urinary bladder, uterus, rectum, prostate, etc. The fracture of hipbone is very common and makes the whole pelvic unstable by affecting vitally important organs like the uterus, bladder, rectum, prostate etc.

## 1.3 Bone evolution with age

The pattern of change with age is found in all animals, including humans and rumens like other tissues of the body it starts with fragility, malleability, elasticity and reaches to maximum of strength, then declining in strength and malleability until it reaches a stage not favorable for life compatibility. Bone serve many important function for animals i.e. organ protection locomotion, weight bearing and calcium homeostasis [10]. All these properties start from immaturity reaching to climax in young adulthood and then start declining unless it reaches the most unfavorable conditions.

Bone start as procure cartilage from mesenchyme tissues in the organogenesis period of prenatal life (before birth). Extracellular matrix mineralized to HA [11] crystal and collagen type 1 [12] serve like steel bars of a building which is woven around HA with cementing materials GaGs (glycosaminoglycan's). Bone is the masterpiece for locomotion, protection and weight bearing due to its fine geometrical microstructure and gross structure.

In a prenatal age of animal cartilaginous precursor of bone start ossification [13] (the process of bone formation) until puberty where by all of the long bone become ossified except the ends which contain estrogenic cartilage making new bones and subsequent mineralization as shown in figure 1.2. This collar of cartilaginous bone forming factory is called growth plate [14, 15] which fuses after puberty where by the animal attain maximum linear growth and no further tallness occur [16].

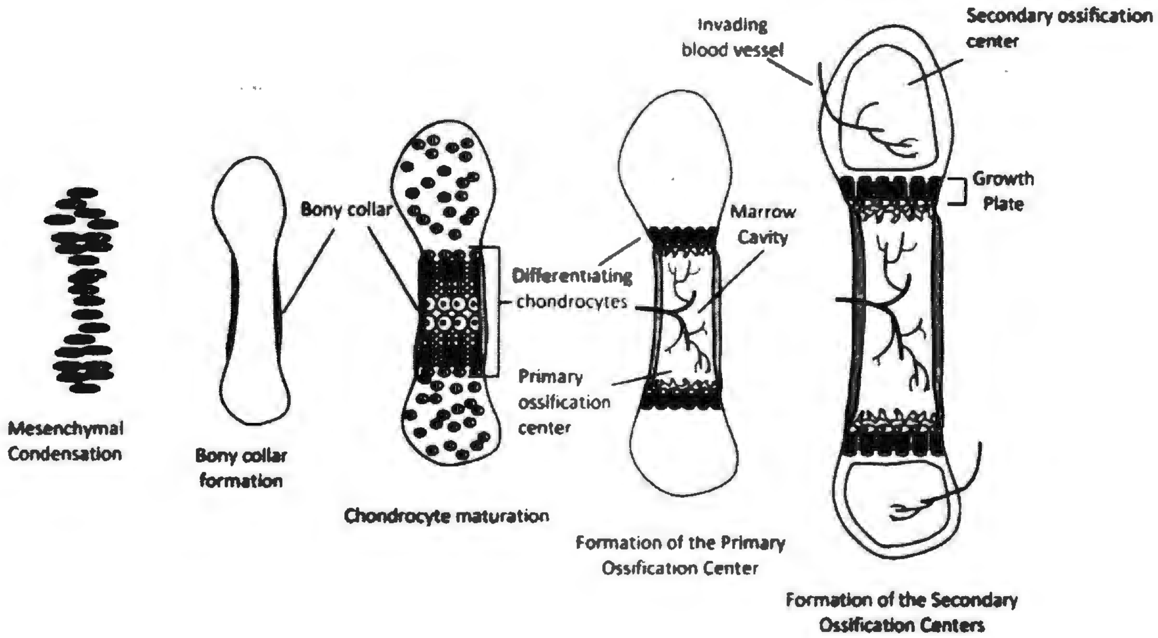


**Figure 1.3:** Mesenchymal of condensation cells and the formation of common shape of long bone [17]

The process of bone remodeling occurs throughout life in which bone reaches a maximum compatibility from the function in which collagens are densely interwoven and bone mineral density (BMD) are at the peak. This is a shift from the maximum malleability to stiffness and perfect strength. At this age, bone can withstand maximum stress and shear force and there are very less chances of fracture.

After this peak BMD starts decreasing [18-20], the process mineralization slows down collagen cross linkages decreasing leading the animal to a state called osteoporosis, in which the decrease BMD is insufficient to withstand the daily demands, a time where the bone is weak and chances of fracture are higher. This phenomenon of bone remodeling is not only dependent on age other factor also contribute much like exercise, use and disuse, diseases, nutrition, genetics and acquired factors.

## 1.4 Problem Statement

Different researchers have used different bones, i.e. femur and tibia bone and various methods and techniques, i.e. three point bending tests, flexural tests, and compression tests etc for research. The hipbone is less investigated with age. Therefore, this research is focused on the hipbone. The main purpose of this study is to examine the failure behavior of bovine bone (male calf Hipbone) with the variation of age. This investigation will be both analytical and experimental.

## 1.5 Objectives of the Thesis

The basic purpose of this thesis is to investigate the effect of age on fracture toughness of the bovine hip cortical bone. The secondary aim of this thesis is to investigate the effect of age on hip bone's,

i. Young modulus
ii. Ultimate tensile strength

## 1.6 Research Methodology

The research has been carried out analytically and experimentally. The analytical portion is included in order to investigate the stress field at the cracks in the bones. For analytical analysis, the fracture mechanics approach is used.

The displacement field (strain distribution) at the crack tip has been analyzed using digital image correlation (DIC) while using MATLAB.

## 1.7 Organization

In this thesis, several mechanical properties of the bovine hip bone such as fracture toughness, young modulus and ultimate tensile strength of different ages are investigated. The next chapter is the literature review of the previous studies for fracture toughness, young modulus and ultimate strength. Chapter 3 describes the experimental setup for fracture toughness tests and tensile tests. Chapter 4 presents the results and discussion of our experimental work. Chapter 5 describes the discussions and chapter 6 ends this dissertation the conclusions and future recommendations.

## Chapter 2

# LITERATURE REVIEW

This chapter covers the literature review of the mechanical behavior of different bones with the variations of age. The age effect on mechanical properties of several bone types of human beings and bovine has been extensively investigated in the past few decades. Following paragraphs describes prominent studies on the age effect on tensile strength, fracture toughness and modules of elasticity of femur, tibia, and cortical bones.

Leng et al [21] investigated the response of mechanical properties by considering the age effect of human cortical bone and for collagen phase in different orientations. Micro tensile tests were performed on young, middle age and old age human bones donors demineralized cortical bone and aging effects for the different orientations for the mechanical properties of the collage. The analysis indicated that the ultimate strength and elastic modulus of the demineralized bone specimens decreased with aging in both the transverse and longitudinal orientations and there is failure strain has no changes in failure strain in both directions irrespective of aging. Results suggested that collagen strength and stiffness of bone deteriorated with aging for both directions as shown in the table 2.1. The failure strain of the collagen phase in both orientations irrespective of aging is not observed and it is concluded that the failure of the collagen phase does not depend upon aging and orientation.

**Table 2.1:** Mechanical properties of collagen fiber with age

| Age group | Ultimate stress Transverse (MPa) | Ultimate Stress Longitudinal (MPa) |
|---|---|---|
| Young Age | 4.48 | 19.9 |
| Middle Age | 2.91 | 15.4 |
| Elder Age | 2.31 | 13.2 |

Zherrina et al [7] performed compression and optical microscopy test in the transvers and longitudinal directions for protein and mineral portion of bone to investigate the mechanical properties of bovine femur bone of different ages. The optical microscopy test of microstructure of young and mature, the results show that mature bone is less porous and more stable than the young bone, and mature bone is more stronger and less tough in both directions longitudinal and transvers for the case of mineral and protein part of bone. For the protein, part of the mature bone is less strength than young bone. The elasticity of bone is 68 % increases from young age to mature age.

Ani Ural et al [22, 23] investigates age-related toughness loss in human cortical bone by performing interrelated finite element (FEM) modeling. They performed the qualitative analysis of cortical bone for treatment and diagnostic modalities in the old age population at risk of failure. They show loss of bone and crack growth behavior and toughness experimentally with variations of age. They obtained two types of results from this research. First, the analytical result shows that the toughness of cortical is decreasing up to 15 % per ten years of age and the finite element analysis shows that the toughness decrease 13 % per ten years of age. The results obtained from this study are shown in the following figures 2.1.

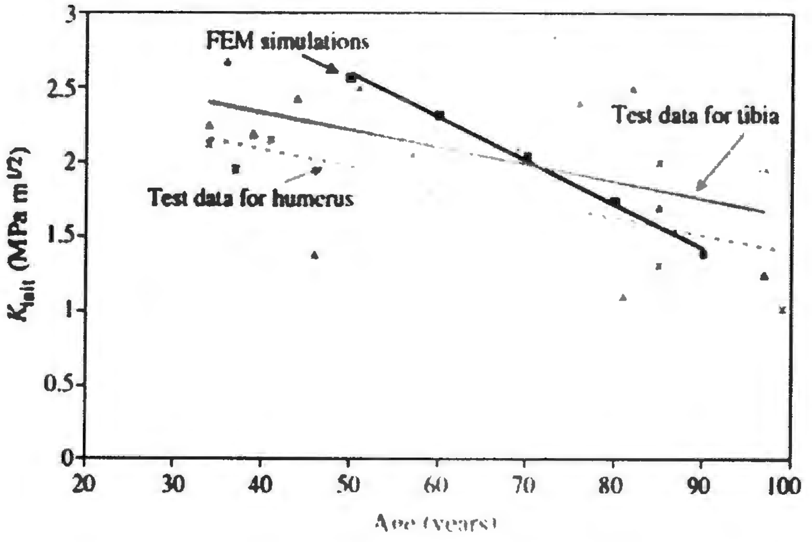


**Figure 2.1:** FEM simulation of age-related initiation toughness loss and its comparison with experimental results [22].

Elizabeth et al [24] investigated toughness and plasticity of human cortical bone of several lengths with respect to age. They analyzed the strength properties and vitro toughness of hydrated cortical bone specimens of the fresh-frozen hummer of old and young age group.

Three-point bending testing of unmatched beams were used to determine the strength and toughness measurements and it showed a 10% loss of peak strength and 5% loss of yield strength with age. The results of this study are shown in the following figure 2.2.

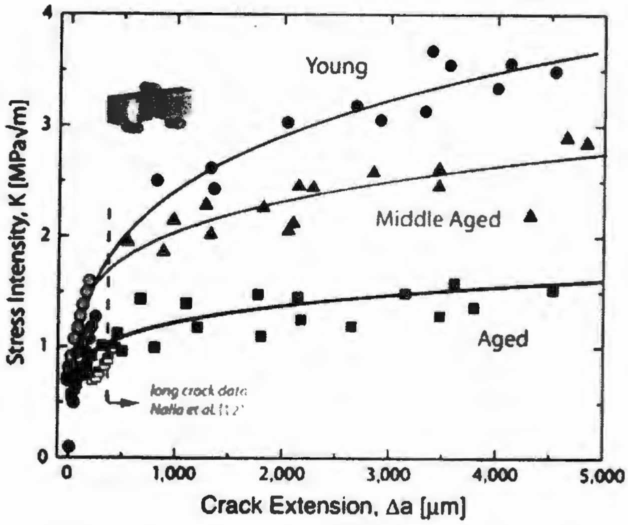


**Figure 2.2:** Stress intensity versus crack extension [24].

Nalla et al [25] investigated the toughness of human cortical bone by R-curves and determine the aging effects. They performed the experiments on ex vivo to examine the fracture toughness properties of human cortical bone in the longitudinal direction to measure quantitatively the effect of aging. The toughness with crack extension in longitudinal direction of human cortical bone were studied with age. They obtained a wide range of bone specimens (34-100) by the American Society for Testing and Materials (ASTM) standard E-399. The fracture toughness of cortical bone, expressed in terms of rising R-curve behavior as show in the figure 2.3, shows significant deterioration with aging. They found that the toughness decreases from 2.12 to 0.92 MPa√m in six decades.

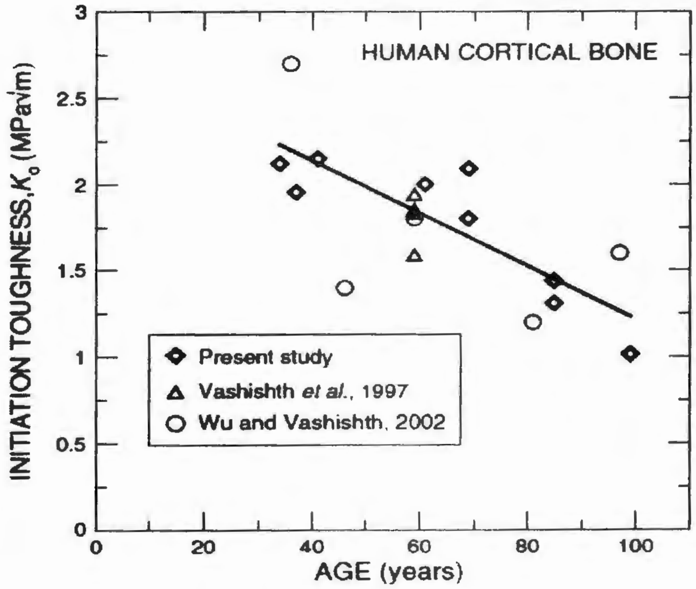


**Figure 2.3:** Variation in the toughness with age. Data obtained from Vashishth [26] and Vashisth and Wu [27] are plotted to compare [25].

Kulin et al [28] performed number of tests on femoral specimens obtained from post mortem equine donors ranging in age of 0.5 to 28 years to study the effects of age and loading rate on failure of cortical bone. In this study, the loading rate effect and the mechanical behavior of the cortical bone of different ages were determined by performing the fracture toughness and compressive tests under monotonic and cyclic loading conditions. Fracture toughness experiments were performed on single and double notch specimens using a four-point bending loading conditions to measure fracture toughness and to observe the differences in crack initiation between quasi-static and cyclic experiments as shown in figure 2.4. The behavior of the propagation of fracture was analyzed using electron microscopy and scanning confocal. From these results, it may be observed that the bone has lower fracture toughness, but the high value of compressive strength, when tested under cyclic loading as compared to static experiments. The decrease of fracture toughness is also observed when measured statically, but there is a slight change of fracture toughness under cyclic loading conditions by considering the age, where brittle failure behavior dominates.

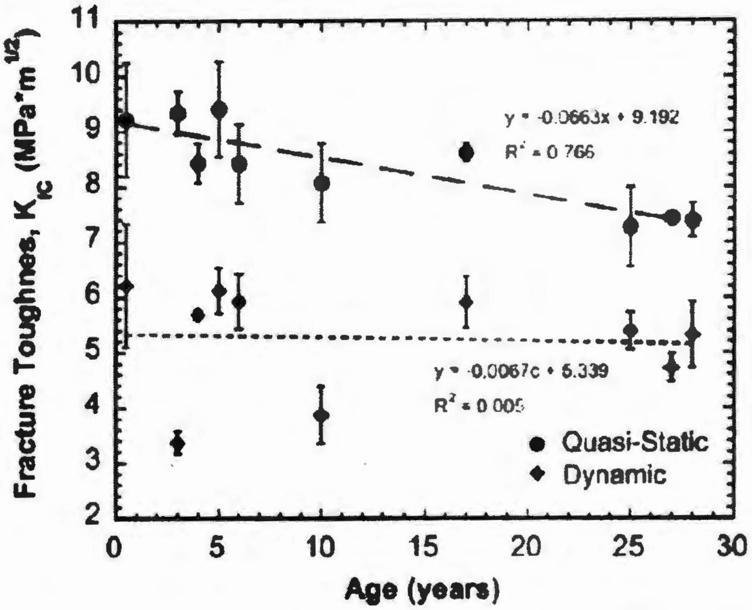


**Figure 2.4:** Fracture toughness measurement comparing he changes in in quasi-static and dynamic results as a function of age [28].

McCalden et al [29] investigated the tensile failure of 235 cortical bone specimens. The donors have in age ranged from 20 to 102 years. They found the microstructure, mineralization, mechanical testing and the porosity of cortical. The ultimate strength and energy absorption capability decreases by 5 to 12% per ten years. The microstructural analysis showed that the haversian system of the bone increases with the age. With the age, the porosity of bone has significantly increased while the mineral content does not change. The change in porosity of bone caused 76% decrease in strength. The mechanical properties of bone have significant effects on the quantitative change rather than qualitative change.

Wang et al [30] investigated that the mechanical reliability of the collagen fiber in bone declines with age, and such adversarial changes are related to the reduced strength of aging bone. They proposed that the adversarial variations in the collagen fiber network occur with age of peoples and these variations can lead to the reduced fracture toughness of bone. In addition, the results indicate that the nonenzymatic glycosylation of protein may contribute significant changes in collagen fiber and, therefore, leads to the age-related decline of bone quality.

Ping-Cheng Wu et al [27] investigate the crack propagation methodology to identify bone toughness with respect to age. Different tests were performed on human bone to investigate the initiation and propagation of crack. It was investigated that toughness of cortical bone after the initiation and propagation decreases twice abrupt and reaches to zero value during the 10th period. These results show that a decrease in bone's fracture with age may be more radical than presently proposed and the propagation-based extent may be more sensitive to changes in bone with respect to age.

Zioupos et al [31] performed 3 point bending static tests to describe the overall effects such as stiffness, degradation and strength of cortical bone with age and shows that stiffness reduces with age. They determined the fracture toughness, young modulus, and work fracture of human male aged bones between 35-92 years by doing experiments. They found that all these mechanical properties decreases with age as shown in figure 2.5 and 2.6.

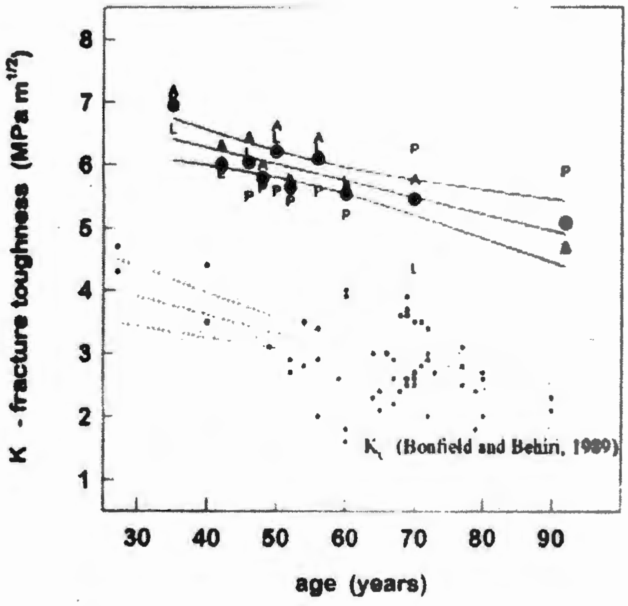


**Figure 2.5:** Fracture toughness versus age, the dotted line shows the Bonfield and Behiri [32] results

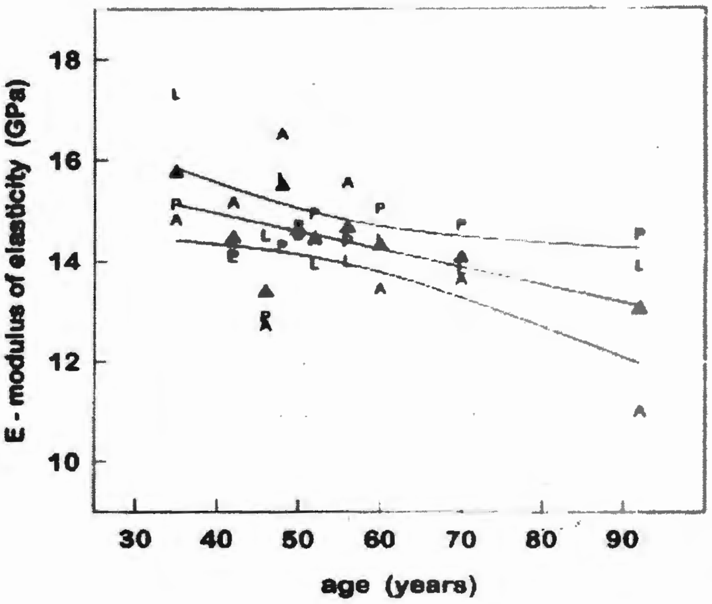


**Figure 2.6:** Elastic modulus versus age [31].

Phelps et al [33] describes the differences in material properties of osteonal and interstitial regions of cortical bone with age. The parameters such as porosity, longitudinal and transverse toughness, osteonal and interstitial micro hardness, strength to weight ratio, density of the bone and organic and inorganic phases of bone were investigated for female baboon femurs as a function of age. The longitudinal and transverse fracture toughness decreases while the interstitial microhardness increases. However, in other parameters no major differences were found with age. To measure the differences in the material properties in these two regions the ratio of interstitial to osteonal microhardness have been used, correlation analysis shows that the longitudinal fracture toughness of bone has an important relationship with this ratio. Local differences in material properties may result in high stress concentrations at adhesive lines and enable longitudinal crack propagation.

Raghavendra et al [34] investigated the fracture toughness or R- curve of cortical bone using a static fracture test. The bovine cortical bone shows significant anisotropy in compression property. The strength in the longitudinal (parallel to the axis of bone) direction is higher than the transverse direction. It was also observed that the dry bone strength is greater than wet bone.

Manilay et al [7] investigated microstructure of young and old femur bones using optical microscopy and compression testing in the transverse directions as well as longitudinal direction for the cases of deproteinized, dematerialized and untreated femur bones.

It was observed by optical microscopy that the mature bone is more stronger in both direction as compared to young bone. Mature bone was also found to be stiffer and less tough compared to young bone as shown in figure 2.7. These results are related to the increase the amount of minerals in mature bone with the age. Young bone was found to be stronger because of the less amount of minerals.

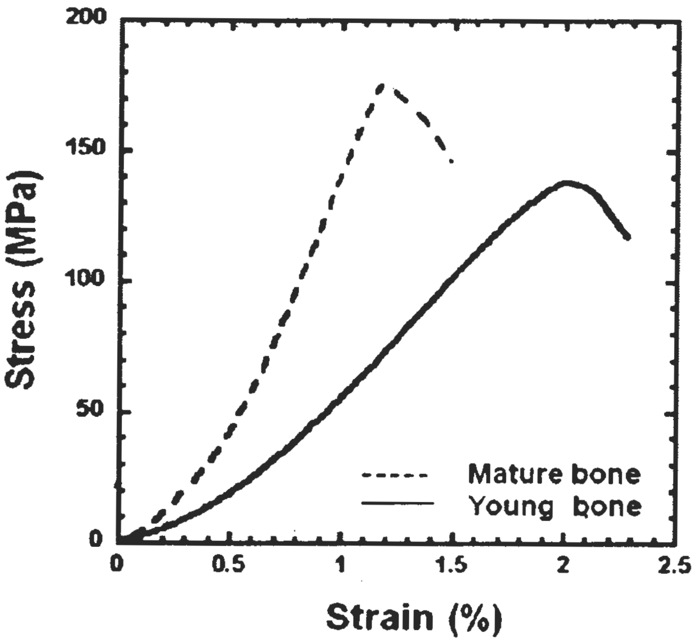


**Figure 2.7:** Representative compressive stress–strain curves. Untreated mature (dashed) and young (solid) bones for (a) longitudinal [7]

Nagaraja et al [35] investigated the change of quality, quantity and microstructure of bone with the age. The clinical observations of these changes show an abrupt increase in fracture rate with age. In this research, they compared younger (average age 2 years) and older (average age 10 years) bovine bone to evaluate how change in bovine bone quantity and quality with age. The results show strong positive correlations between microdamage and local stresses and strains for both younger and older bovine trabecular bone. The maximum compressive principal stress in microdamaged bone from younger bone was significantly lower (144 MPa) compared to microdamaged bone from older bone (219 MPa)

Flavia et al [36] performed the experimental fracture toughness of bovine cortical bone using single edge three point bending, and compact tension test. The tests were carried out by the crosshead speed 0.1 mm per mint. The results showed that the crack path in the LCT specimens is perpendicular to the collagen fiber as shown in the figure 2.8 and fracture toughness for compact tension test is 5.8 MPa√m.

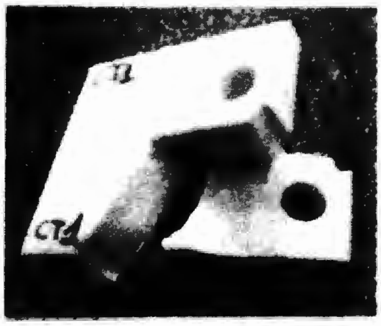


**Figure 2.8:** Fracture toughness of LCT specimens [36]

The literature survey reveals that considerable work has been done for characterization of age effect on cortical and femur bones. However, studies describing age effect on hip bone are very less. Being an important bone, the investigation of hip bone behavior with age is vital for the treatment and modeling. Owing to the research gap for the hip bone behavior with age, this study is focused on the characterization of mechanical properties variation of hip bone with age. The following chapter describes the experimental testing of the hip bone specimens tested for age effect on fracture toughness, young modulus, and ultimate strength.

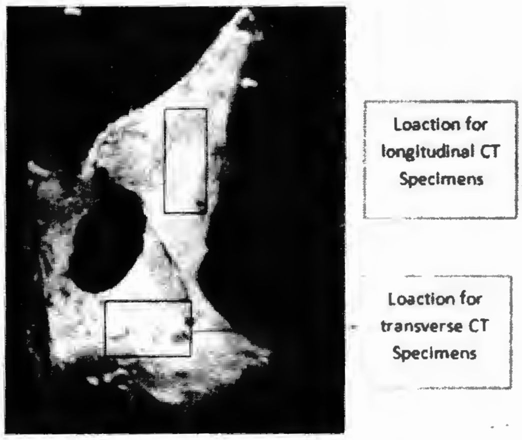


**Figure 3.1:** Location of the specimen extraction on from hip joint bone

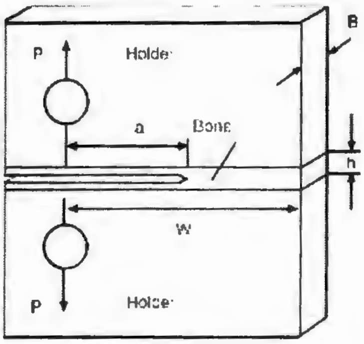


**Figure 3.2:** Geometry of a typical CT Specimen

**Table 3.1:** Test Matrix of CT Specimens

| Sr No | Specimen Code | Age [Year] | LCT | TCT | Width (w) [mm] | Crack Length (a) [mm] | Thickness (b) [mm] |
|---|---|---|---|---|---|---|---|
| 1 | S1 | 1 | ✓ |  | 31.11 | 12.4 | 2.15 |
| 2 | S2 | 1 |  | ✓ | 29.00 | 11.60 | 2.3 |
| 3 | S3 | 2 | ✓ |  | 30.70 | 11.61 | 3.19 |
| 4 | S4 | 2 |  | ✓ | 29.72 | 11.19 | 3.7 |
| 5 | S5 | 2.5 | ✓ |  | 31 | 12.9 | 2.00 |
| 6 | S6 | 2.5 |  | ✓ | 30.82 | 12.13 | 2.4 |
| 7 | S7 | 3 | ✓ |  | 30.26 | 10.61 | 2.7 |
| 8 | S8 | 3 |  | ✓ | 30.57 | 12 | 2.6 |
| 9 | S9 | 4 | ✓ |  | 31 | 12.40 | 3 |
| 10 | S10 | 4 |  | ✓ | 29.92 | 11.26 | 3.3 |
| 11 | S11 | 5 | ✓ |  | 20.06 | 6.33 | 1.8 |
| 12 | S12 | 5 |  | ✓ | 15.77 | 4.21 | 2 |

### 3.1.2 Test Procedure

The test was conducted in universal testing machine (UTM) WDW-E-100 at HITECH University Taxila. WDW-E Series is a new kind of electronic PC control UTM. The loading capacity of this UTM is 1000 KN. They measure and plot the loading force, deformation and crosshead stroke etc. The displacement range of the machine was +/-80 mm.

The specimen was bolted in the steel fixture and held in the machine grips. The test setup is illustrated in figure 3.3. The test was performed at normal room temperature and pressure conditions. The load applied by machine at a crosshead speed of 0.5 mm/minute. During the test, the computer connected to the machine recorded the applied load and corresponding deformation. The specimen was loaded until the crack started to grow and at this point, the test was stopped.

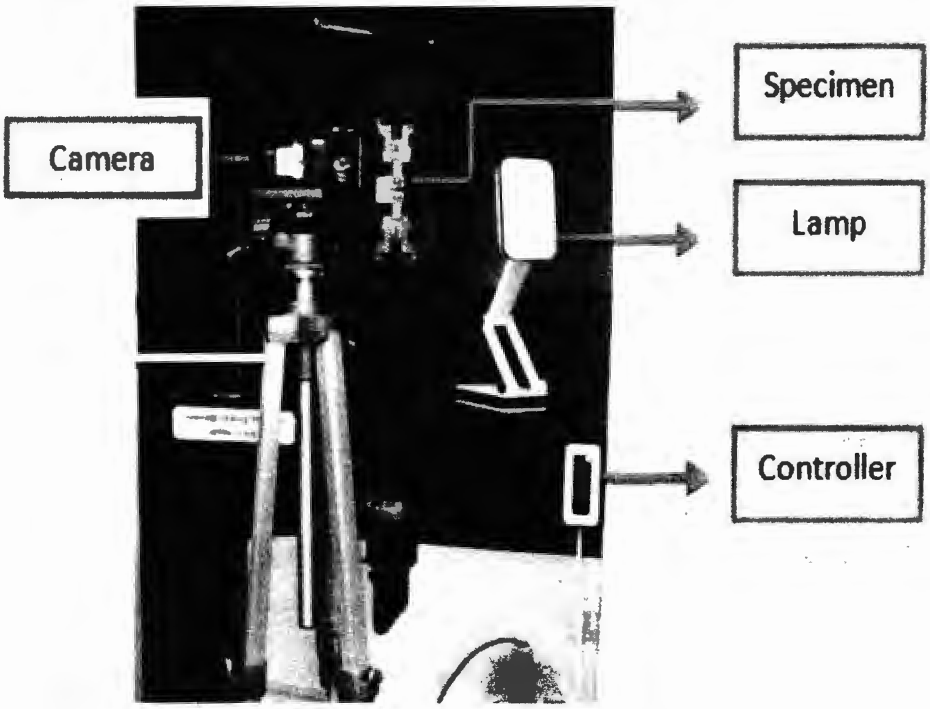


**Figure 3.3:** Fracture toughness test setup

## 3.2 Tensile Testing

### 3.2.1 Material and Specimen Preparation

The test material was collected in a similar manner as described in section 3.1. The tensile test specimens were prepared according to ASTM D3039 [38]. The geometry of a typical tensile specimen is shown in figure 3.4. The ends of specimens were covered with a scotch tape. Twelve specimens of hip joint bone were prepared as given in test matrix in table 3.2.

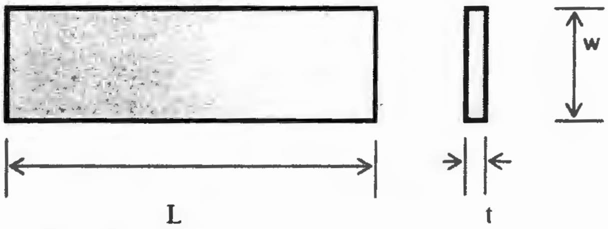


**Figure 3.4:** Geometry of tensile test specimen

**Table 3.2:** Test matrix for tensile test

| Sr No | Specimen Code | Age | Longitudinal Tensile Test Specimen | Transverse Tensile Test Specimen | Width (w) [mm] | Length (L) [mm] | Thickness (t) [mm] |
|---|---|---|---|---|---|---|---|
| 1 | S1 | 1 | ✓ | | 16.54 | 7.4 | 2.10 |
| 2 | S1 | 1 | | ✓ | 16.22 | 5.91 | 2.18 |
| 3 | S2 | 2 | ✓ | | 16.46 | 6.21 | 2.7 |
| 4 | S2 | 2 | | ✓ | 16.28 | 5.7 | 3.00 |
| 5 | S3 | 2.5 | ✓ | | 13.26 | 4.07 | 1.91 |
| 6 | S3 | 2.5 | | ✓ | 17.68 | 6.35 | 2.00 |
| 7 | S4 | 3 | ✓ | | 15.88 | 3.91 | 2.30 |
| 8 | S4 | 3 | | ✓ | 12.32 | 3.3 | 2.03 |
| 9 | S5 | 4 | ✓ | | 16.22 | 5.7 | 2.36 |
| 10 | S5 | 4 | | ✓ | 16.22 | 5.08 | 2.50 |
| 11 | S6 | 5 | ✓ | | 13.30 | 5.08 | 1.91 |
| 12 | S6 | 5 | | ✓ | 16.26 | 5.15 | 2.00 |

### 3.2.2 Test Procedure

The tensile test specimens were held in the UTM jaws as shown in figure 3.5. During the test, the specimen was loaded at a crosshead speed of 1 mm/minute. During the test, the computer connected to the machine in order records the applied load and corresponding deformation. The test was stopped as the specimen failed.

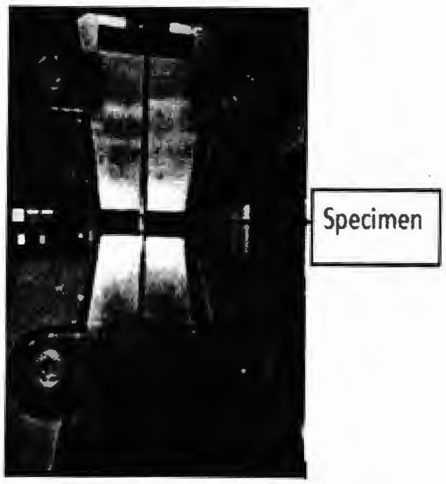


**Figure 3.5:** Experimental setup for tensile test

## 3.3 Experimental Data Analysis

### 3.3.1 Fracture Toughness Test Data Analysis

The fracture toughness, K was determined by using the equation [37].

$$K = \frac{F_C Y}{t w^{1/5}} \tag{3.1}$$

Where $F_c$ is the critical load at fracture, t is the specimen thickness and w is the specimen width as shown in figure 3.7. The value of Y is given by following equation:

$$Y = 29.6\left(\frac{a}{w}\right)^{0.5} - 185.5\left(\frac{a}{w}\right)^{1.5} + 655.7\left(\frac{a}{w}\right)^{2.5} - 1017\left(\frac{a}{w}\right)^{3.5} + 638.9\left(\frac{a}{w}\right)^{4.5} \tag{3.2}$$

Where “a” is the crack length of CT specimen in the above equation.

### 3.3.2 Tensile Test Data Analysis

The stress σ was calculated using the equation

$$\sigma = F/A \quad (3.3)$$

In the above equation, F is the load applied and A is the cross sectional area of the specimen. The strain ε in the specimen was calculated using the equation:

$$\varepsilon = \delta/l \quad (3.4)$$

Where δ is the total deformation of the specimen until failure and $l$ is the initial length of the specimen.

The young modulus E was calculated by the following equation

$$E = \frac{\sigma_{max}}{\varepsilon_{max}} \quad (3.5)$$

Where $\sigma_{max}$, and $\varepsilon_{max}$ are the maximum stress and maximum strain at failure.

## 3.4 DIC Experiment

DIC is an advance, non-contact optical technique for measuring strain and displacement. DIC technique is simple to implement providing cost effective explicit results leading to a massive range of potential.

This technique consists of capturing consecutive large images with a high-speed digital camera during the deformation period to assess the changes in specimen surface characteristics and understand the behavior of the specimen while being subjected to incremental loads [39-41].

In this study, Strain field is measured by the application of DIC at the CT specimen's crack tip. The DIC setup is shown in the figure 3.3. The specimens were speckled with black and white spray paint as shown in figure 3.6.

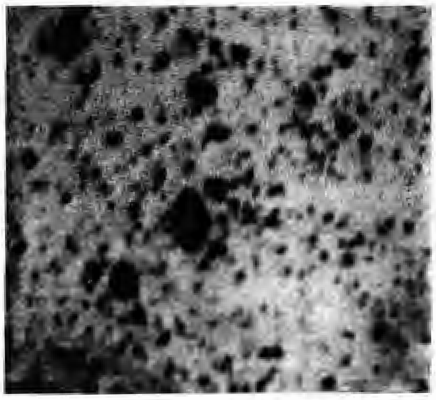


**Figure 3.6:** Applying speckle pattern

During the start of the fracture toughness test, the camera was focused on the crack tip and images were taken at a rate of four per second. The image recording were stopped after the specimen's failures [42, 43].

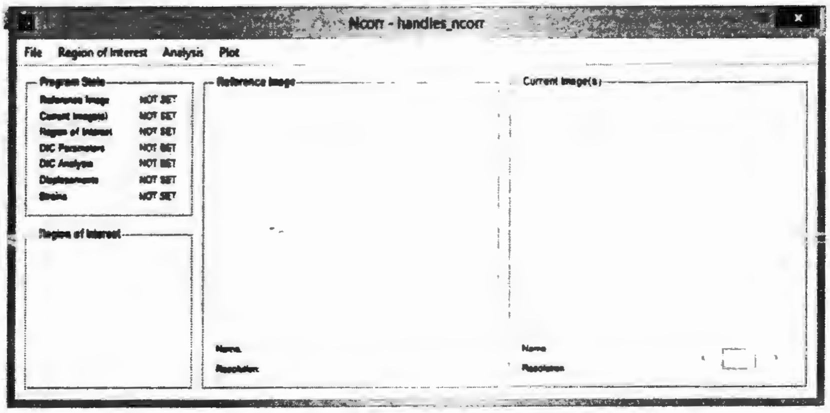
## 3.5 DIC Data Analysis

The images captured during fracture toughness tests were processed in MATLAB using NCORR software. NCORR is MATLAB based GUI tool used for performing DIC analysis.

The DIC data analysis consists of following steps:

i. **Handles_NCORR**

First we type the handles_Ncorr in the MATLAB window, then the NCORR GUI window appear as shown in figure 3.7



Program State
Reference Image NOT SET
Current Image(s) NOT SET
Region of Interest NOT SET
DIC Parameters NOT SET
DIC Analysis NOT SET
Displacements NOT SET
Strains NOT SET

**Figure 3.7:** NCORR GUI window

### ii. Loading Reference Image

By selecting file from the main menu of NCORR GUI window, we loaded the reference image from the main memory as shown in figure 3.8.

**Figure 3.8:** Reference image for DIC analysis

### iii. Load All Images

From the main memory, we selected all images of the specimens, which were processed in the NCORR software as shown in the figure 3.9.

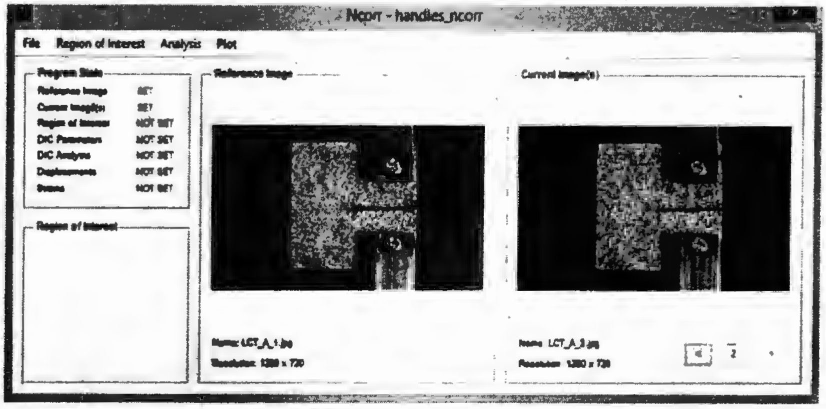


**Figure 3.9:** Image loading in NCORR for DIC analysis

iv. **Region of Interest**

We selected the region of interest from the reference image of the specimen through the main menu of NCORR GUI window as shown in figure 3.10.

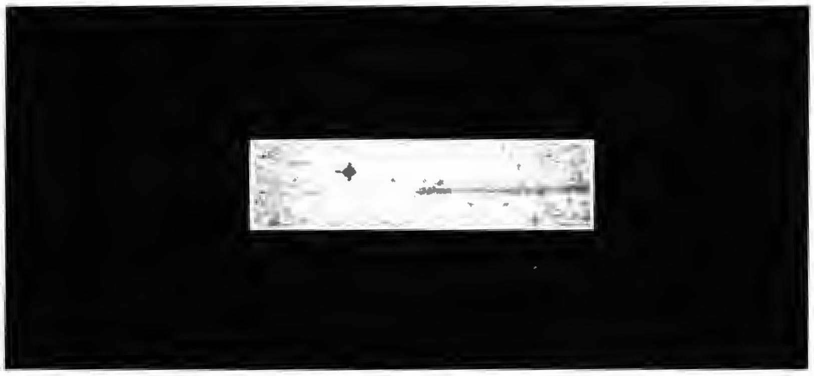


**Figure 3.10:** Selection of region of interest DIC analysis

v. **Analysis**

From the analysis tab, we select the DIC parameter, set iteration, seed propagation and perform analysis as shown in figure 3.11

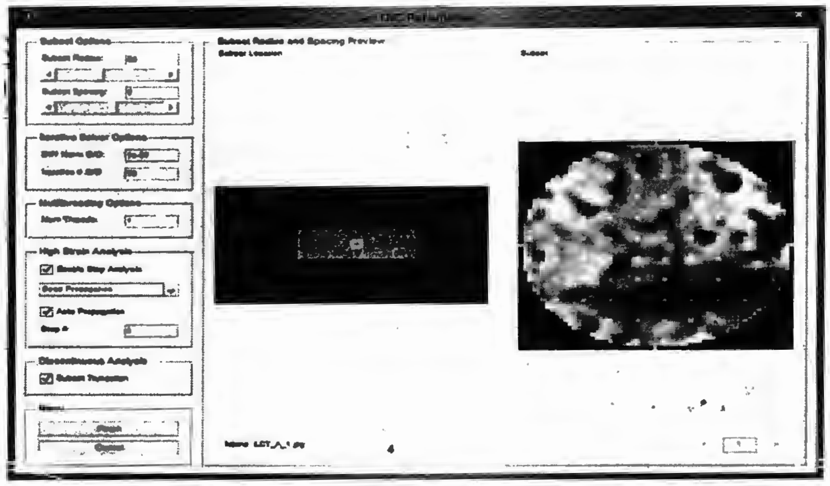


**Figure 3.11:** Setting the DIC parameter

### vi. Strain calculation and Plotting

After completing the analysis, we calculate and plot the strain for each specimen, as shown in the figure 3.12.

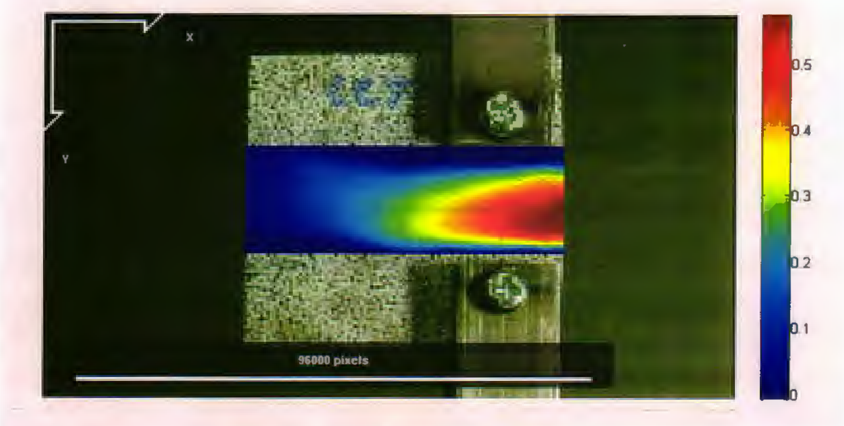


**Figure 3.12:** Graph of the strain from DIC analysis

After obtaining strain value from the digital image correlation (DIC) use in the equation 3.1, the ultimate strength of each age of the bone was calculated by the following equation.

$$\sigma_{ult}=E\varepsilon_{max} \tag{3.1}$$

## Chapter 4

# RESULTS AND DISCUSSION

This chapter presents the experimental results of the tests described in the chapter 3. Section 4.1 presents the results of fracture toughness tests; Section 4.2 discusses the results of young modulus variation with age. Section 4.3 presents the results of ultimate strength versus age and section 4.4 gives the details of DIC technique.

### 4.1 Effect of age on fracture toughness

The fracture toughness of hip joint bone is plotted against the age in figure 4.1. The figure shows that the fracture toughness is increasing with age up to three years for LCT specimens. The fracture toughness decreased from the value of 3.059 MPa√m to 2.688 Mpa √m over the time of four years. The fracture toughness in TCT specimens decreased from 4.05 MPa√m to 2.3201 MPa√m over the time of four years.

The figure shows that the toughness the fracture toughness slightly increases in the initial period from one to two years of age span in both LCT and TCT directions. However, the trend is mixed in the age span between 2 to 2.5 years as shown. The toughness decrease in both directions with the age over 2.5 years. In the initial 1-2 year period, the fracture toughness in the transverse direction is almost twice the longitudinal direction. The higher toughness value in this case may be attributed to the higher resistance to the crack growth in transverse direction. The crack path is nearly parallel to the initial notch plane in case of LCT specimens. In the LCT, the crack takes its path between collagen layers facing least resistance. Figure 4.2 shows the images of the LCT specimen before and after the failure. The fracture plane of the specimen is parallel to the initial crack as shown. In case of TCT specimen, the crack has to cross the collagen layer network due to which it faces higher resistance promoting higher fracture toughness. The crack path in this case is diverted from the initial artificial crack plane as shown in figure 4.3.

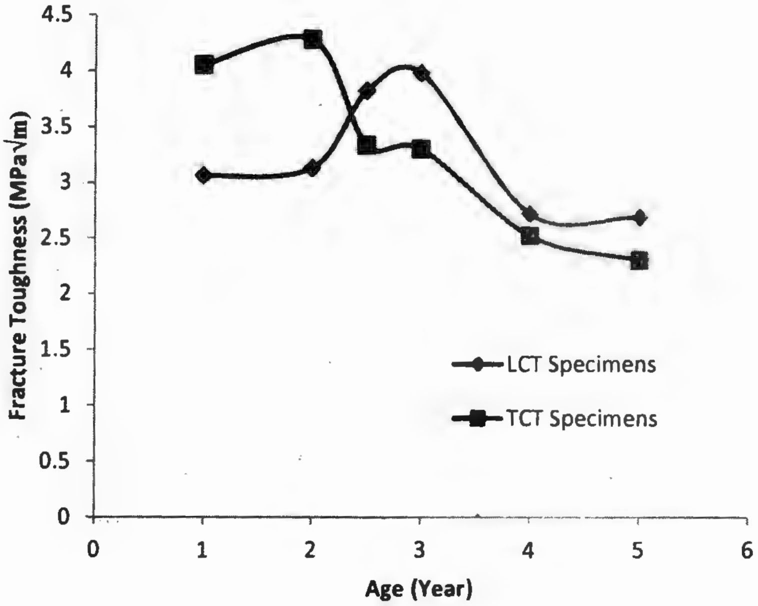


**Figure 4.1:** Hipbone fracture toughness versus age

In longitudinal orientations, the failure of specimen follows a straight path that was right angle to the loading axis. While in transverse direction, the failure of specimens followed a random orientation.

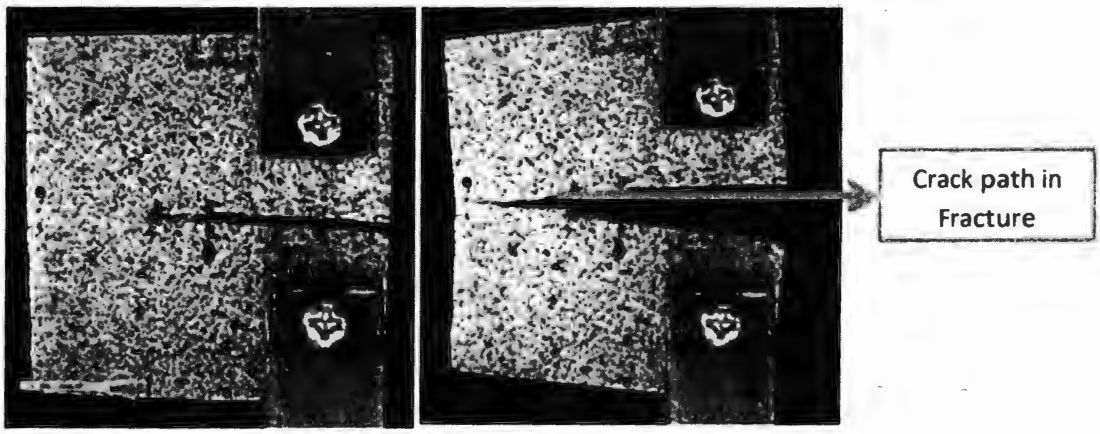


**Figure 4.2:** Longitudinal CT specimen before failure (left) and after failure (right)

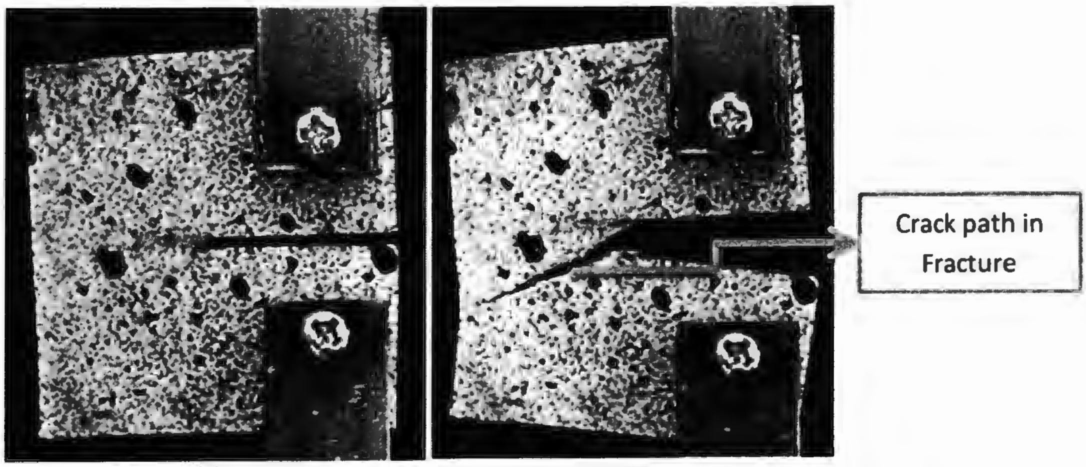


**Figure 4.3:** Transverse CT specimen before failure (left) and after failure (right)

The previous research performed on the bovine femur and tibia [44-46] gives the longitudinal fracture toughness in range of 2.2 to 5.8 MPa√m, and the transverse fracture toughness [44, 46-48] which gives the value in range of 0.23 to 6.12 MPa√m which are very close to the previous reported research. The values of fracture toughness (LCT) of bovine femur bone is 2.3 to 5.9 MPa√m . The longitudinal fracture toughness of bovine bone increases from 1 to 3 years in different rate as the human bone's fracture toughness increases from 1 to 20 years. After 20 years the fracture toughness of the human bone decreases with the age as shown in figure 4.4 [31, 49]. The results obtained from the experiments shows the same behavior for the bovine bone above the age of 3 years.

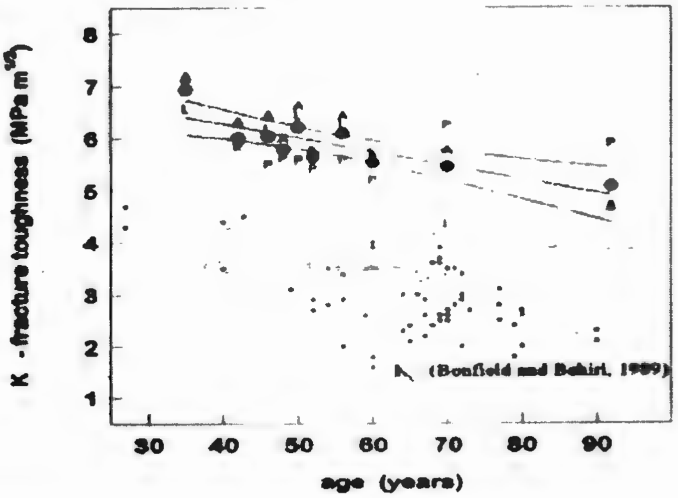


**Figure 4.4:** Some previous investigation of fracture toughness versus the age of the bone [31]

### 4.2 Young Modulus Variation with Age

The young modulus of the bone is plotted against the age in figure 4.5.

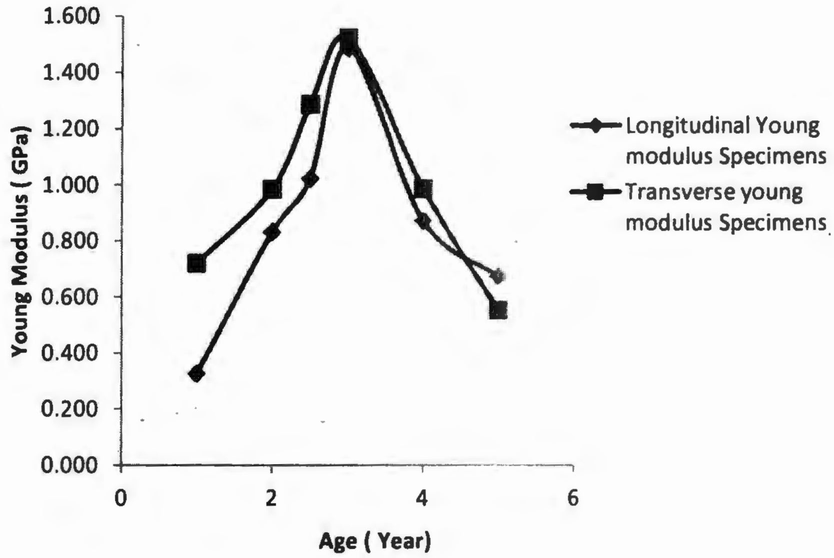


**Figure 4.5:** Young modulus of hipbone versus age of the bone

The modulus is lower at the young age of one, up to three years for both longitudinal and transverse specimens respectively. The modulus after three years decreases gradually. This trend is very rare in the bone and it matches the result of the experimental data of the other species bones like human, bovine for young modulus calculation.

The research done on the variation in the stiffness, strength, and toughness of human cortical bone with Age by Zioupos and Currey, [1, 30, 31, 50, 51] observed in figure 4.6 that the age significantly affects the young modulus and ultimate strength of human bone. They determined the fracture toughness, young modulus, and work fracture of human male aged bones between 35-92 years by doing experiments. They found that the all these mechanical properties decreases with age [52].

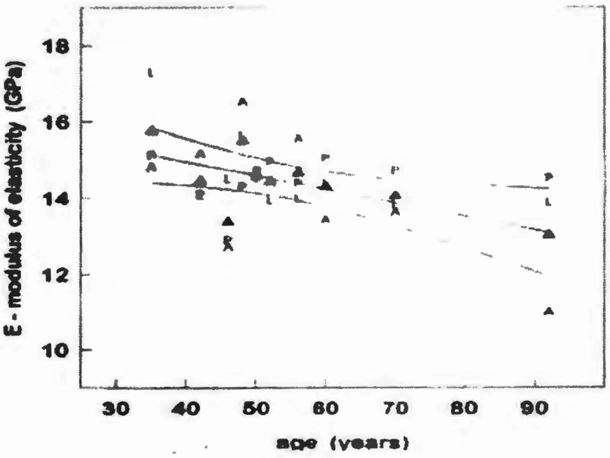


**Figure 4.6:** Some previous investigation of Young modulus versus the age of the bone [31].

## 4.3 Ultimate Strength versus Age

The tensile strength (MPa) of the bone is plotted against the age in figure 4.7.

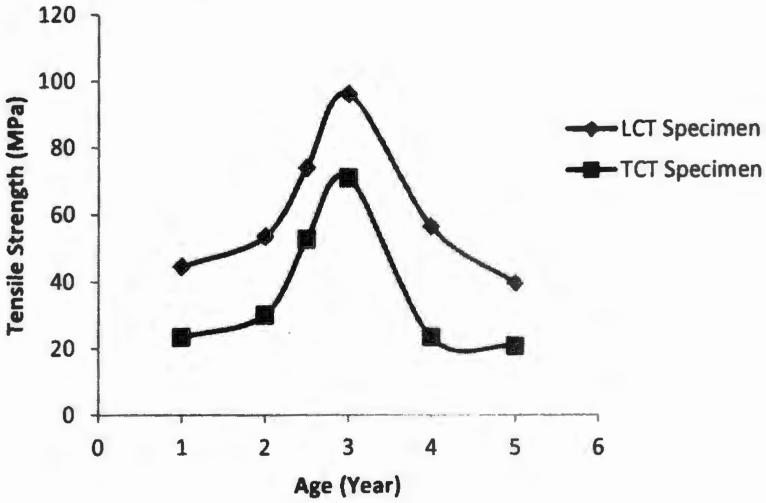


**Figure 4.7:** Ultimate Strength of the hipbone versus age

The tensile strength increases from one to three years age. The tensile strength decreases from three years onward. This trend is very common in the human, bovine, and other specimen's bones [31, 53].

The Wang et al [30] studied the changes in the toughness of bone. From their experimental work, they found that the mechanical properties of the bone decreases with age. The fracture toughness and ultimate tensile strength deteriorated with age due to demineralization of bone. Similarly, Ashman and Rao [1] studied the ultimate tensile strength of bovine femur bone. They found from the result that the ultimate tensile strength of the bone deteriorated with advancing age. This research validates our research work.

## 4.4 Ultimate Tensile Strength Using DIC Technique

The strain distributions were determined for both the longitudinal and transverse the images of the bone specimen shown in figures 4.8 to 4.19.

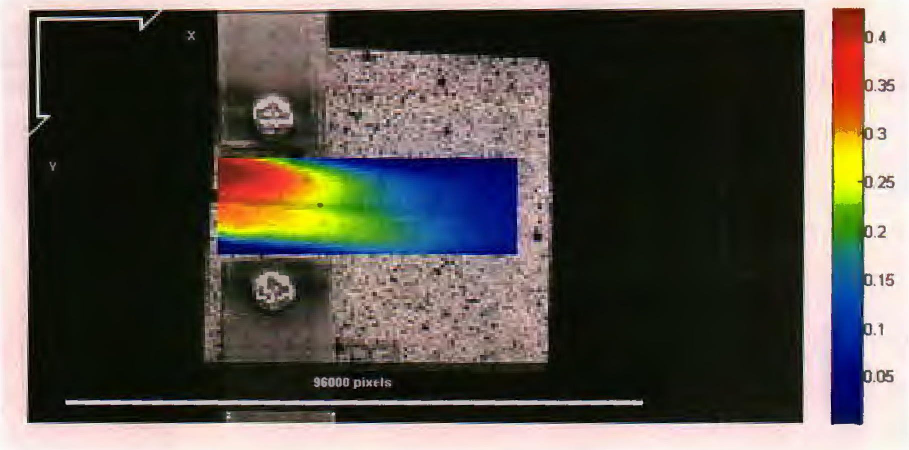


**Figure 4.8:** Strain distribution of S1 (1 Year) in the longitudinal direction

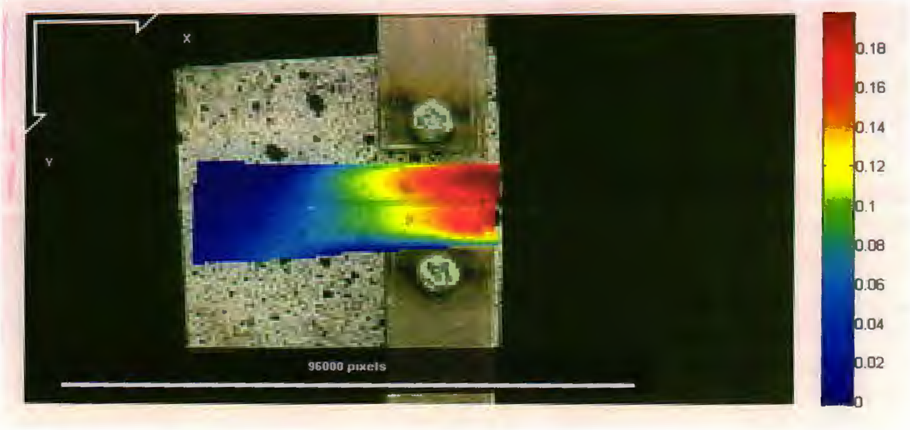


**Figure 4.9:** Strain distribution of S2 (1 Year) in the transverse direction

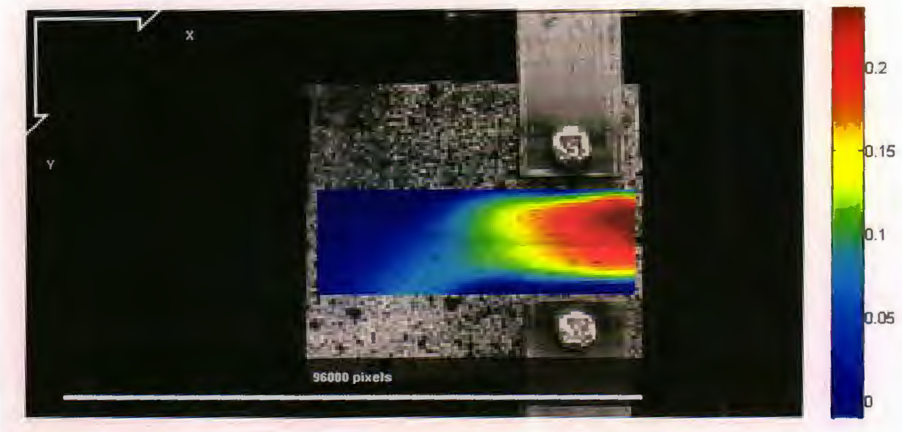


**Figure 4.10:** Strain distribution of S3 (2 Years) in the longitudinal direction

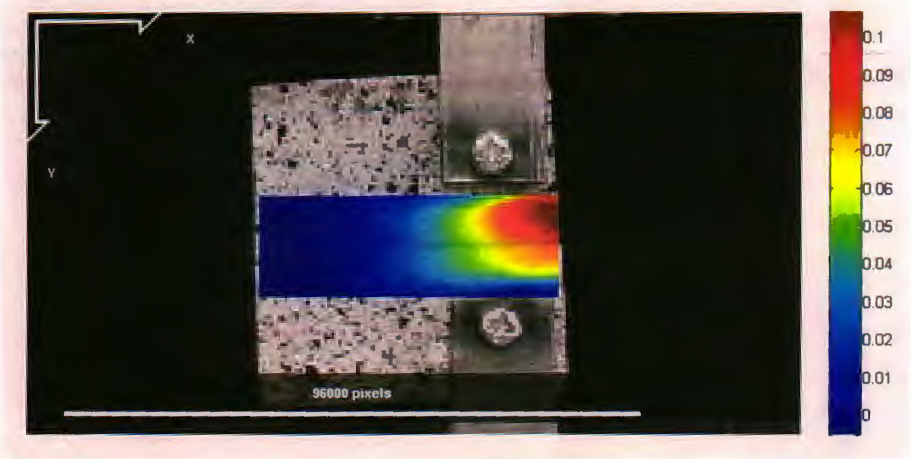


**Figure 4.11:** Strain distribution of S4 (2 Years) in the transverse direction

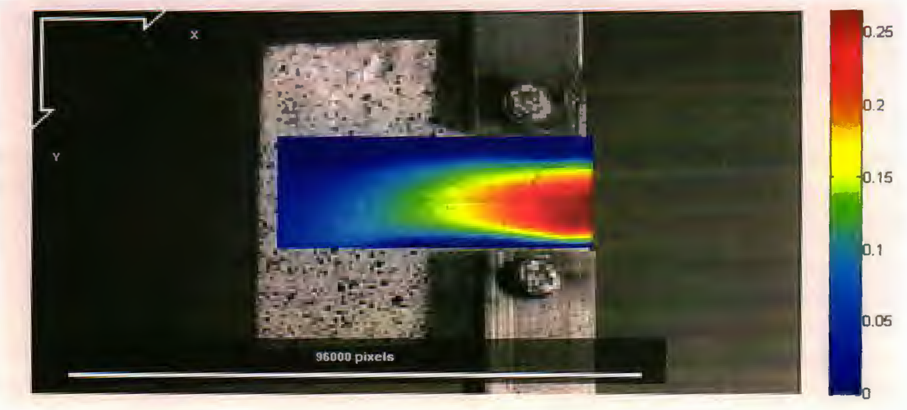


**Figure 4.12:** Strain distribution of S5 (2.5 years) in the longitudinal direction

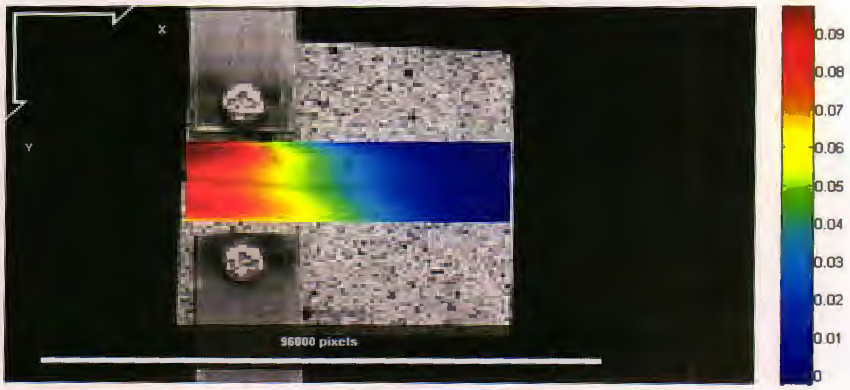


**Figure 4.13:** Strain distribution of S6 (2.5 years) in the transverse direction

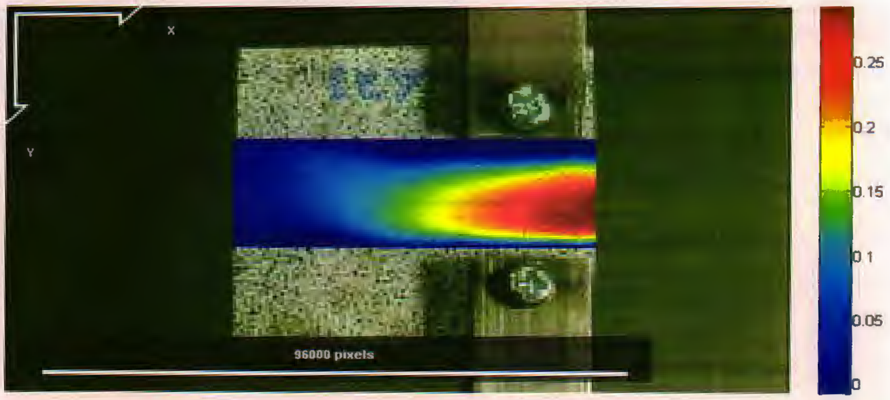


**Figure 4.14:** Strain distribution of S7 (3 years) in the longitudinal direction

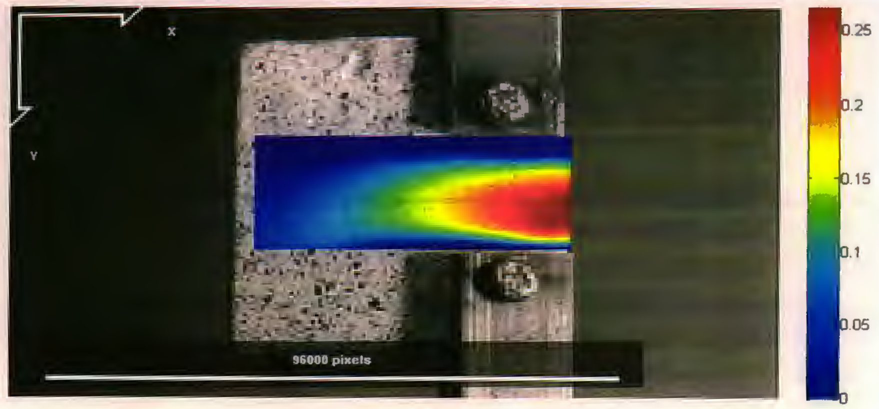


**Figure 4.15:** Strain distribution of S8 (3 years) in the transverse direction

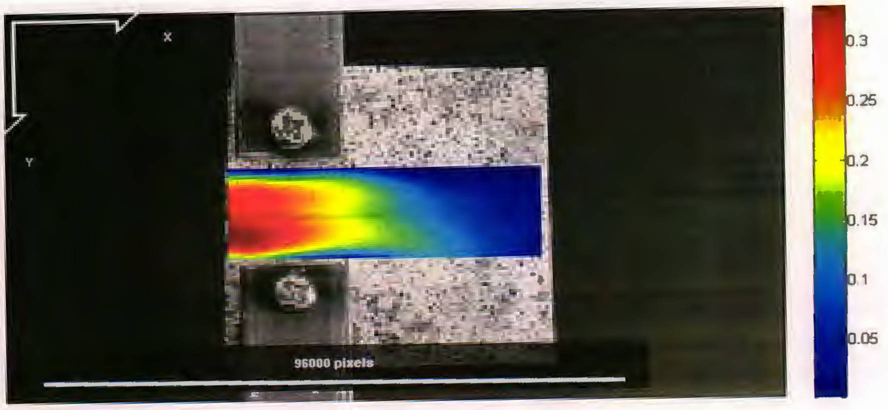


**Figure 4.16:** Strain distribution of S9 (4 years) in the longitudinal direction

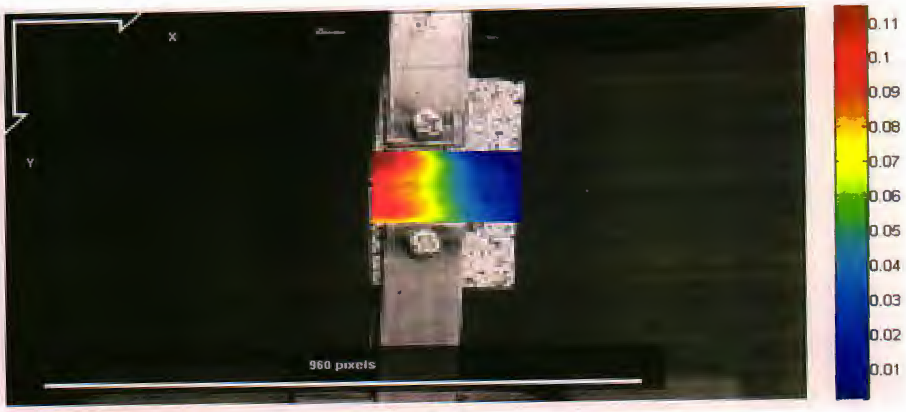


**Figure 4.17:** Strain distribution of S10 (4 years) in the transverse direction

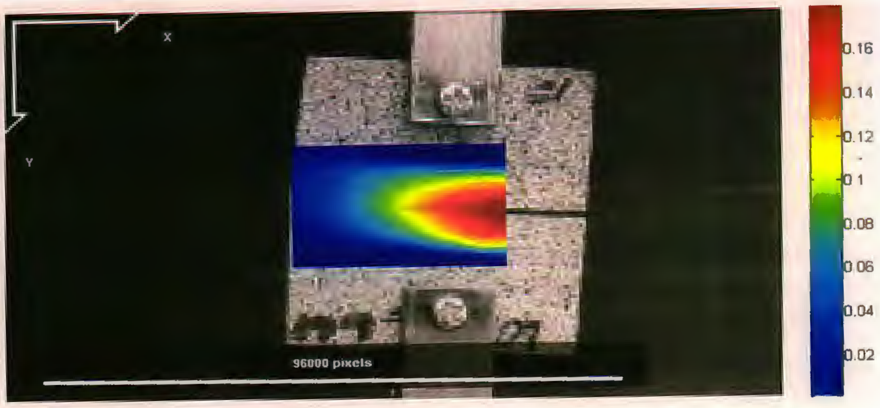


**Figure 4.18:** Strain distribution of S11 (5 years) in the longitudinal direction

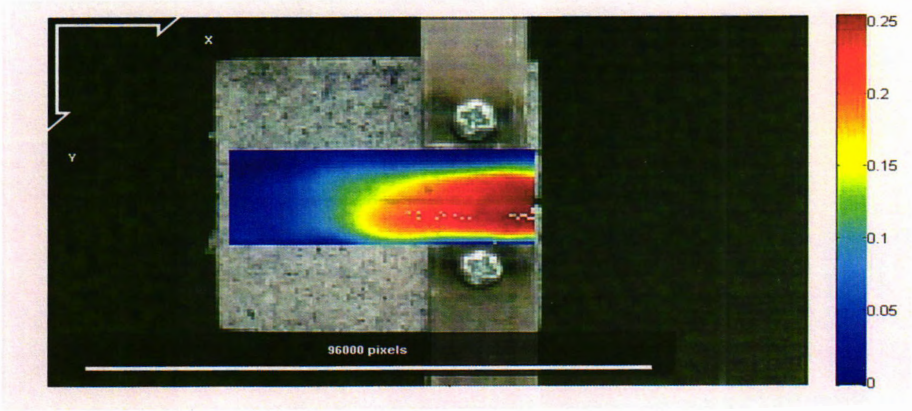


**Figure 4.19:** Strain distribution of S12 (5 years) in the transverse direction

The results of the strength (LCT specimens) obtained from both DIC and analytical are plotted in figure 4.20. The ultimate tensile strength of the cortical bone for both analytical and DIC strength first increases with age upto three years (53% and 58%) respectively, and the next two years decrease (58% and 59%) respectively. The difference between the analytical ultimate tensile strength and DIC strength is 10% to 30%.

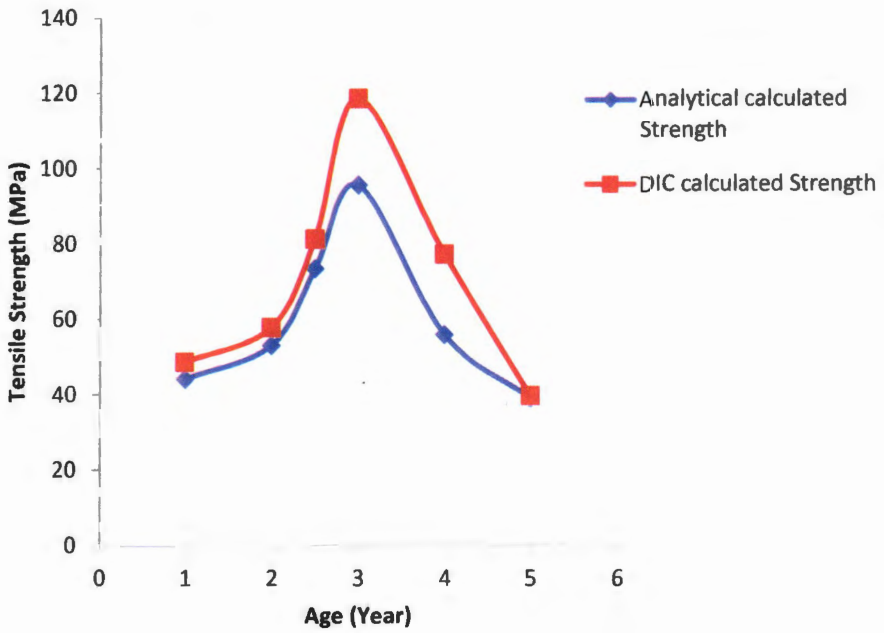


**Figure 4.20:** Comparison of analytical ultimate tensile strength of the DIC calculated strength in the longitudinal direction

The results of the strength (TCT specimens) obtained from both DIC and analytical are plotted in figure 4.21. The ultimate tensile strength of the cortical bone for both analytical and DIC strength first increases with age upto three years (23 MPa to 70 MPa and 28MPa to 91 MPa) respectively, and the next two year decreases (70 to 20 MPa and 91 to 33 MPa) respectively. The difference between the analytical ultimate tensile strength and DIC strength is 1% to 30%.

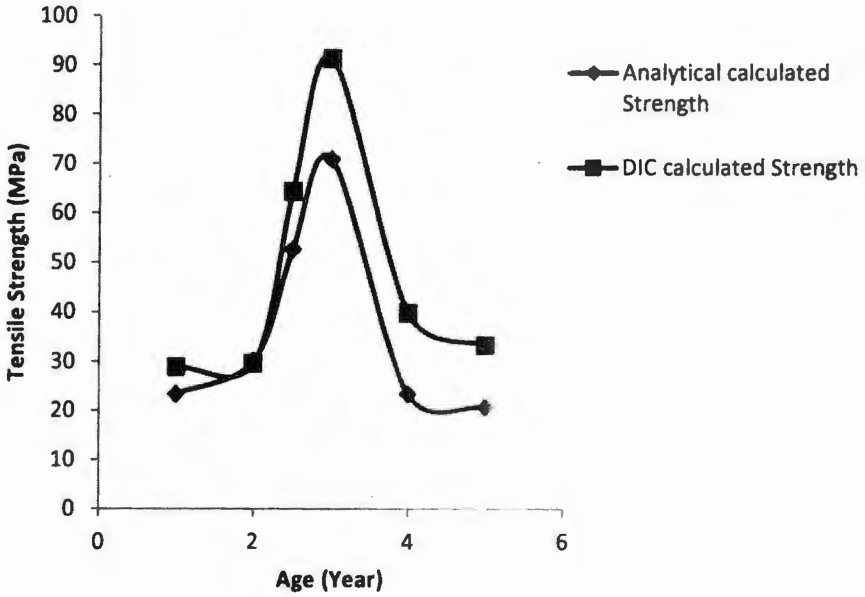


**Figure 4.21:** Comparison of analytical ultimate tensile strength to the DIC calculated strength in the transverse direction

# Chapter 5

# DISCUSSION

Previous chapters described the details of experiments and their results for the effect of age on mechanical properties of hip joint bone. This chapter highlights some of the issues with utilization of the research carried out in a broader spectrum of bone properties investigation and human bone treatment. Section 5.1 discusses age effect on bovine bone and its extension to the treatment of human bone. Section 5.2 discusses test techniques for characterization of bone behavior. Section 5.3 DIC techniques and its significance in the observation of localized deformation of bone under application of loads and section 5.4 discusses the fatigue behavior of bone. The last section of this chapter discusses Bone behavior under compression.

## 5.1 Age effect on bone and its application for the treatment of damaged bone

The fracture properties of bone do not remain constant with age rather they vary day by day in life, in some cases the function is improving, but in others decreases. The mechanical properties human bone increase from the embryo to 18 years and from 18 to 30 is static; (there are no mechanical properties in the static age) after that the mechanical properties of bone decline with age. In the bovine bone, the mechanical properties are increased from one to 18 months and after three years, the bone fragility start. The human bone mechanical properties increase slowly and the mechanical properties of bovine bone increase abruptly because the average age of male bovine is 5-8 years compared to the human average age which is 60-80 years. The chemical composition of human and bovine bone is almost same. For this reason, the bovine bone can be grafted in the human body; however, the difference in age factor effect should be accounted for in bone grafting.

Bone grafting is an advance surgical technique in which an immense research is going on along with bone prosthesis. The idea of bone grafting came to medical and biological science after successful kidney, pancreas, lung and liver grafts. An indication of bone grafting is loss of bone tissues during accidents and trauma, surgical procedures or disease causing bone erosion.

There are three types of grafts.

i. Autografts
ii. Allografts
iii. Xenografting

Auto or autologous graft means grafting one owns bone [54]. It is a common practice in orthopedics Operation Theater because bone tissue are lost in majority of trauma cases and elective surgeries. The usual bone donor sites are crest of iliac bone (part of hip bone) and floating ribs (11, 12). Autograft is the most successful method of restoring last bone tissues with no immunological reaction because the bones are from one's own body [55].

Allografts means transferring of organs from one individual another of the same species i.e. from human to human or bovine to bovine bone, from both living or dead donor but to overcome immunological reaction or chances of rejection, you have to give strong immune suppressant drugs [56].

Xenografts means transferring of organs from an individual to stranger i.e. of another species like transferring from bovine to human or bovine to some other species. There is many differences between bones of different species, therefore, the rejection chances are more but we can use it and decrease rejection chances upto some extent by treating it chemically e.g. corals of bovine is hydrochemically modified and calcified matrix can be used as xenograft. Utilizing the same technique age of the donor and recipient must be bear in mind because age contributes a lot to grafting phenomenon [57].

## 5.2 Tests techniques to determine fracture toughness of bones

The fracture toughness of bone can be determined by three point bending test, single and double edge notched test, charpy test and compact test. The three point bending test can be used for homogenous material to determine the fracture toughness and bending moment. The single and double edge test is used for measuring the fracture toughness of the specimens. The charpy test is used to measure the energy of the specimen and used to examine the fracture surface that whether the fracture surface is granular, fibrous or a mixture of both. Different zone of fracture surface helps us to determine the mode of fracture. The compact tension test is used to determine the fracture toughness in tensile direction.

The compact tension test has the advantage over with above all tests because this test is more efficient and can be used for thick plates like cortical bone. The bone is naturally a complex and hierarchal material, composed of different layers and inorganic minerals in the form of small crystal. Hence, the compact tension test can be used for measuring the fracture toughness of hip bone for more accurate and satisfactory results.

## 5.3 Use of DIC for the observation of localized phenomenon of yielding in bone samples

Digital image correlation (DIC) is an advance technique for measuring strain and displacement. This method is simple, most accurate and cost effective compared to other techniques. This method works by comparing digital photographs of a specimen at different stages of deformation. By following blocks of pixels, the system can measure surface displacement, build up the full field 2D deformation vector fields, and strain maps. The DIC can measure true strain at any point in the specimen, while the average strain is obtained with the traditional method. The DIC can be used to accurately analyze the necking process. The fracture strain is much higher than the total elongation measured by the traditional method.

## 5.4 Fatigue behavior of bone

Bones are subjected to fatigue due to the action of cyclic loading. A material under cyclic loading fails at stresses below the values that the material can bear under static loading [58]. Bones are subjected to fatigue loading as a result of daily activity or prolonged exercise, which occurs for instance in athletes. Fatigue loading is one of the primary causes of human bone failure [59, 60]. Therefore, it is important to characterize the residual strains that occur after cyclic loading. Fatigue loading is one of the primary causes of human and bovine bone failure. Fatigue fractures may occur in young adults. Failures due to stress concentration result from prolonged exercise and occur more frequently at the tibia and metatarsal bones, while fragility or osteoporotic fractures of elderly individuals take place on the proximal femur and on vertebrae where the trabecular bone supports most of the load. Fatigue damage results from accumulation of micro-cracks [61]. Micro cracks are repaired through bone remodeling. However, if micro-cracks are not repaired, they accumulate and coalesce, leading to a decrease in bone stiffness and to an increase of fracture risk [62].

## 5.5 Bone behavior under compression

The compression tests are the most frequently performed tests to determine the bone's mechanical properties, due to their simple procedure and to the fact that bones are often submitted to compression loads[63]. The cortical bone tested in the longitudinal direction exhibits an elastic region up to strains of 0.7% and a plastic deformation until 3% [64]. Bone has an anisotropic performance, as when is tested along the transversal direction is less stiff and strong [65, 66]. The compressive stress-strain curves of healthy cortical and trabecular bones are characterized by three distinct regions of shown as figure 5.1.

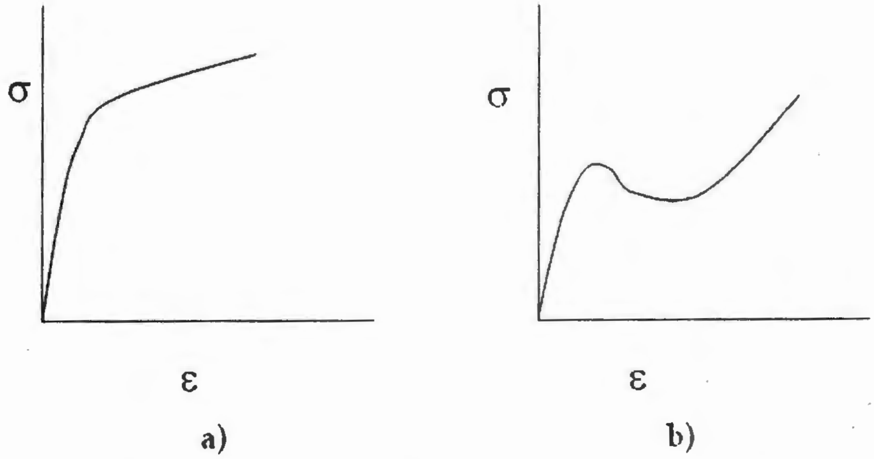


**Figure 5.1:** a) cortical and b) trabecular bone [67]

The initial elastic response is due to the bending of trabeculae, while the second region is associated with elastic buckling, plastic yielding or brittle fracture of trabeculae. Finally the third regime occurs when cell walls or trabeculae touch each other. It is generally accepted that the mechanical properties of trabecular bone are related to their density. Several works can be found on the compression of trabecular bone, using vertebrae, tibia and femoral heads [68]. For example, it was found that for a given density, samples from tibia had higher Young's modulus than the ones from vertebrae.

## Chapter 6

# CONCLUSION AND FUTURE WORK

## 6.1 Conclusions

This thesis experimentally investigated the age effect on fracture toughness, strength and young modulus of bovine's hip bone specimens. Fracture toughness was tested using CT specimens, tensile strength and young modulus were tested using flat specimens. Fracture toughness and tensile strength were investigated using ASTM E-399 and ASTM D-3039 respectively. The strain fields at the crack tip of compact tension (CT) specimens were examined using digital image correlation technique. The image files obtained during the DIC analysis were processed using free software NCORR.

These results show that fracture toughness, strength and young modulus are higher for transverse specimens. The higher values of transverse specimens are because of the presence of resisting collagen fibers. The variations in the values of the above properties with respect to age were more sensitive in the case of bovine bones as compared to the human bone behavior. This is due to shorter life span of the bovines than human. On the other hand, the properties of hip bones are similar to the cortical bone properties.

In the DIC analysis, the difference between the analytical ultimate tensile strength and DIC strength is 10% to 30% in LCT specimens. In TCT specimens, the difference between the analytical ultimate tensile strength and DIC strength is 1% to 30%. DIC results verified the experimental observations.

## 6.2 Future Recommendations

The variation of bone properties with respect to age can be explained properly if the mechanism of change of bone material is well understood. In future, the micromechanism of age effect on bone can be performed with the different size scale using the scanning electron microscope, CT scan or MRI techniques. These findings can be used for the development of mechanistic models for bone mechanical behavior. The focus should also be made to investigate bone mechanical behavior under compression load, as bone is often under compression during the service. Test procedures should be properly defined to simulate actual bone state during the service life of bone. Similarly the fatigue behavior is also important because 80 % of mechanical failures are caused due to fatigue. Fatigue behavior may be investigated using either S-N approach or fracture mechanics approach..

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## LIST OF PUBLICATIONS

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3. Waseem ur Rahman, Rafiullah Khan: Age effect on young modulus of hip joint bone, will be submitted to Technology and Health Care.