Study of Wear properties and Microstructural Analysis on 10% of Al_2O_3 and 10% of ZrO_2 with Polyurethane Resin Polymer Composite as an Orthopedic Implant

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Abstract- *The paper deals with the study on mechanical properties of Hybrid polymer composite material [1] for 10% of Alumina (Al_2O_3) and 10% of Zirconium Dioxide (ZrO_2) with polyurethane resin mixed with hardener for medical applications. It starts with the preparation of material by weight fraction method and fabrication of these basic materials by hand lay-up moulding technique. By this the round shape material is prepared with the specific size, later it is cut into required size as per ASTM standards for conducting wear test to study the wear rate of this composite material. These results are compared with femur bone material properties. And also we are studying the microstructure of the fractured surface of these same composites by scanned electron microscope (SEM Images). How the powder particles are interfaced with each other and it shows the good bonding properties. From this we know the internal structure of polymer material.*

Keywords- Hand lay-up moulding, polyurethane resin, wear test, SEM analysis

I. INTRODUCTION

This paper deals with the study and preparation of composite material for orthopaedic implant i.e. for medical application. This involves the concept of study of mechanical properties of femur bone and the composite material also the comparison of results for both the material by conducting the different material property tests. There are lot of research work is going on in the medical field and also there are many research works are already done on orthopaedic implants. By taking the reference from all those research works on the orthopaedic implants we are doing a project by preparing the composite material for the replacement of femur bone. In this project we are trying to introduce the new composite material and we expected to have the better mechanical properties like high strength, low density and resistance to wear and corrosion.

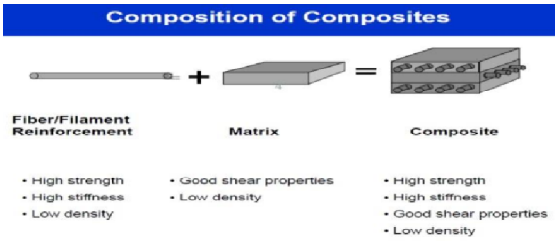


Fig.1.1 Composite structure

This can conclude by conducting the different types of material testing like tensile, compression and bending tests to find out the material behaviour under different load. These tests are conducted in various Research and Development institutes. The composite material is prepared by adding the 2 to 3 materials having the different compositions and different chemical and physical properties. The composite is prepared by Hand Lay-Up moulding technique in the rod form of size 150mm length and 10mm diameter. Later the fractured surfaces are subjected to SEM analysis.

II. LITERATURE SURVEY

- Based on this method the composite material is prepared by the Sandeep kumar. He prepared and studied the material property for 5%, 2.5%, 7.5% and 10% of SiC with combination of 5% of fly ash. [2]
- Biomed Research International Value 2015.[3]
- Investigation of .composite material of Al6063 matrix material includes zircon sand and alumina by 8% was studied by Mr.Jenix Rino [4]
- K. B. Girisha et al. investigated the effect of different weight fraction of zirconium oxide nanoparticle reinforced Al356.[5]
- Slivia suner Moreno theory of ultra-molecular weight polyethylene for joint replacement [6]
- Evaluation of mechanical properties of existing material SS316L by jagadish S P Dr A Thimmana Gouda[7]

- A Review on Natural Fibers D. Chandramohan& .K. Marimuthu.[8]

III. PROBLEMS IDENTIFICATION

It is identified that the problems associated with the orthopaedic implants. In our present generation, peoples are facing so many problems in health because of change in environment i.e. the problems may arise from genetic factor, effect of food, living style and factor of age. The strength of human bones are going to wear day by day i.e. decrease in the bone strength. This affects the mechanical properties of the bone. The femur bone can bear the major loads in regular activities like running, jumping, lifting and dancing etc. apart from this the femur bone may fracture from several reasons. They are as follows.

1. Heavy load
2. Car accidents
3. Falling from height
4. Slip in bath room
5. Natural disaster
6. In old age
7. Defective bone.

In these situations there is a maximum chance for the fracture of femur bone. So doctors suggested that patients may undergo 2 types of surgeries. They are

a) Inserting the bio material into the bone
b) Replacement of bones by new biomaterial. Biomaterials are also referred as implants.

The main reason for the failure of this material is progressive application of stress, change in the cross section and increase in the wear rate by solid surface mechanical actions. In many cases doctors preferred stainless steel 316 materials and some other materials for the replacement of femur bone as the effective solution for the fracture of femur bone. And also in many research works the different composite materials are also tried for this replacement.

Later it is found that there are so many problems that affects to the human body by using this type of material. Some of them are as follows

1. Heavy pain in body
2. Cause the infection
3. Sometimes this may undergoes deformation
4. Loosening
5. Fatigue
6. Gradual wear
7. Corrosion
8. Toxicity

IV. METHADOLOGY

In order to achieve these objectives we are following the different methodologies for the preparation of composite material for orthopaedic implants i.e.in medical applications with required characteristics. The steps involved in our project are as follows.

1. The first step is literature survey to study and identify the problems occurred in orthopaedic implants surgery. Based on this survey, the planning is done to select the basic materials for the preparation of composites.
2. The composites material is prepared by combining the different powders like aluminium oxide and zirconium dioxide with polyurethane resin.
3. These basic materials are mixed by the weight fraction method. We have selected and studying the material for 10 % of Al2O3 and ZrO2 and 80% of polyurethane resin.
4. The rod is prepared with a size of 150mm length and 10mm diameter. Later the fractured surfaces are subjected to SEM analysis.

V. EXPERIMENTAL PROCEDURES

The procedure involves the steps in fabricating the polymer composite material. The fabrication process involves the weighing, preparing the solution, testing and comparing the results. After preparing the composite material, it is cut into different shapes for conducting the various tests. These tests help to find out the material properties of the polymer composite material. The detail procedure of preparing the composite material is discussed here.

5.1 Materials used for Fabrication Composite Material

1. Aluminium Oxide (Al_2O_3), Active Neutral.
2. Zirconium dioxide (ZrO_2).
3. Polyurethane resin.

1. Aluminium Oxide (Al_2O_3), Active Neutral:-

The Aluminium Oxide (Al_2O_3), Active Neutral is used as the basic raw material for preparing the polymer composite material for medical applications. Aluminium Oxide is basically a chemical material composed of aluminum and Oxygen.

Fig.5.1 Aluminium Oxide (Al_2O_3), Active Neutral

The chemical formula is Al_2O_3 and it is the most abundantly available chemical and available in the crystalline polymorphic phase. Because of its good material characteristics, it has got wide range of applications.it is used in ceramics for fabricating the different composites materials.

2. Zirconium Dioxide (ZrO_2) :-

The Zirconium dioxide (ZrO_2) is used as another basic material for preparing composite material for medical applications. The Zirconium dioxide is basically a chemical compound. It is white coloured oxide of zirconium and chemically it is not reactive. The basic nature of Zirconium dioxide is resistance to wear and resistance to the forces under the different loads in the structural materials. Because of its hardness nature it has got the wide range of applications.

Fig.5.2 Zirconium dioxide (ZrO_2)

3. Polyurethane Resin:-

Polyurethane resin is used as a basic matrix material in this project. It is basically a polymer joined by the several urethane links. This is produced by the combinations of isocyanates and polyols. It is used as the catalyst in the chemical composition. This cannot be melt by heating because it is thermosetting polymer. The polyurethane resin has got different applications in health and industrial applications. This is mainly used in the gaskets, insulation panels, microcellular foam seals etc.

Fig.5.3 Polyurethene Resin

5.2 Requirements for composites material fabrication:-

1) Zirconium Dioxide
2) Polyurethane Resin
3) Aluminium Oxide
4) Roller
5) Stirrer
6) Furnace or Oven for heat Treatment

5.3 Steps in Fabrication of composite material:-

The fabrication of polymer composite material i.e. for 10% of both Al_2O_3 and ZrO_2 consists of following steps.

1. Cleaning the surfaces
2. Weighing
3. Preparation of solution
4. Moulding

1. Cleaning The Surfaces:-

The surface is cleaned to remove the dirt and other foreign particle from the surfaces by using acetone. Acetone is used to clean the surfaces before moulding.

Fig.5.4 cleaning the surfaces

2. Weighing:-

Rule of Mixtures:-

In many research works related to material science, the different material properties of composite materials are measured by using this Rule of mixtures. In order predict the actual weight required to measure the percentage of material is added to composite material. This is done by weight fraction method.

Fig.5.5 Weight fraction method

Formula is,

$$\mathbf{M_c = \rho_c \times V_c}$$

Where, M_c –Mass of composite
ρ_c –Density of composite
V_c –Volume of composite

1. Density (ρ) of the resin in g/cm^3 (Density= Mass/Volume (or) Volume=Mass/Density)

1. ZrO2 = 5.68 g/cm^3
2. Al2O3 = 3.9 g/cm^3
3. Polyurethane resin=1.2 g/cm^3

2. Volume

A) Volume of Tensile Test Specimen= (L*W*T) mm
= (300*300*2.5) mm
V=Volume for Tensile -225 cm^3

B) Volume of compression & bending specimen= (300*300*3.2) mm
V=Volume for compression & bending -288cm^3

C) Volume of wear specimen= $\pi/4*D^2*L$
$= \pi/4* 10^2* 300$
V=Volume for Wear-225 cm^3

Volume of Composite = Volume of Epoxy + Volume of Carbon Fiber

$V_c = V_{Al2O3} + V_{Poltyrethane} + V_{ZrO2}$
(Mass of Composite / Density of Composite) = (Mass of resin/

Density of resin) + (Mass of ZrO_2 / Density of ZrO_2) + (Mass of Al_2O_3 / Density of Al_2O_3)

$Mc/\rho c = m_{resin}/\rho_{resin} + m_{ZrO2}/\rho_{ZrO2} + m_{Al2O3}/\rho_{Al2O3}$
(Dividing the Mass of composite (m_c) on both sides

$1/\rho_c = 1/\rho_{Resin}\ (m_{Resin}/m_c) + 1/\rho_{Al2O3}\ (m_{Al2O3}/m_c) + 1/\rho_{ZrO2}\ (m_{ZrO2}/m_c)$

$1/\rho_c = (0.8 / 1.2) + (0.10 / 3.9) + (0.10/5.68)$

$1/\rho_c = 0.70\ cm^3/g$

$M_c = \rho_c \times V_c = 1.4232 \times 225\ m_c m$

1. 319.5*10% Al_2O_3=31.95 gms
2. 319.5*10% ZrO_2=31.95 gms
3. 319.5*80% polyurethane resin=225.6 gms

Similarly we calculated for Compression Test & Bending Test and Wear test.

3. Preparation Of Solution:-

- The previously measured both Al_2O_3 and ZrO_2 for required proportion is mixed with the polyurethane resin.
- This is stirred slowly by using stirrer. Hardener is mixed with this solution having 10% of both Al_2O_3 and ZrO_2 with 10:1 proportion.

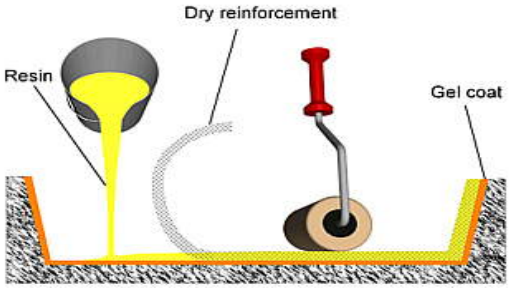
- Now the polymer matrix material is ready to pour into the mould for preparing the laminates.

Fig. 5.6 Preparation of solution

4. Moulding:-

The polymer composite is prepared by Hand Layup technique. This technique involves the following steps

1. In this method the both Al_2O_3 and ZrO_2 powders are blended by weighing.
2. The polyurethane resin is mixed with hardener by proper proportion.
3. The mould with the size (300*300*3) mm and placed on the flat surface and wax is applied on the flat surface.
4. Then pour the prepared polymer matrix material into the mould.
5. The solution is rolled slowly to remove the air bubble and for each corner material is circulated.



.Fig.5.7 Hand Lay-Up Technique

6. By using cylindrical mild steel rod the rolling process is carried out.
7. The coating is done for top layer for good surface finish.
8. The Teflon paper or glass cloth is added to cover the layer and the rolling is slowly carried out.
9. Allow the mould for 80 min for sufficient curing and so as it dries the hardness increases.
10. After the laminates are prepared, it is cut into required shape to carry out the different tests for determining the material properties.

VI. TESTING METHODS

1. **Wear Test:-**To measure the wear rate of the samples and to determine the final weight of the sample.

The wear test is conducted for following load, time and RPM

1. Load-20 N
2. Speed-300 RPM
3. Time-1800 Sec. (30 min)

From the mold the rod sample was cut with the dimensions of 10 mm diameter and 150mm length.

Fig.6.1 Specifications for wear test

Fig.6.2 Specimens before wear test

INSTRUMENT USED FOR WEAR TEST

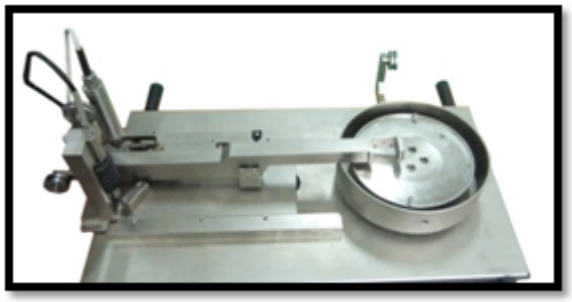


Fig.6.3 wear testing machine

Table No. 6.1 wear test machine specification

SL NO	ITEM	SPECIFICATIONS
1	Test rig	Wear And Friction Monitor, Tr-201
2	Controller	Electronics controller
3	Software	Winducom 2010
4	Computer	Pentium 4,512MB RAM, 2GB, 17"color monitor

2. Scanned Electron Microscope (SEM Analysis) :-

The main objective of study of SEM analysis is to study the microstructure of the surface or fractured surface of the bulk specimens. It used to study the bonding of material, composition and surface roughness of the specimens. So we consulted the JSW steel Ltd. We are using the S-3400 N Hitachi scanned electron microscope for SEM analysis. In this method a beam of electrons are directed towards the material surface that we want to study. These electrons interact with atoms of material and produce the signal which contains the information about the sample's surface, composition topography and other properties such as electrical conductivity.

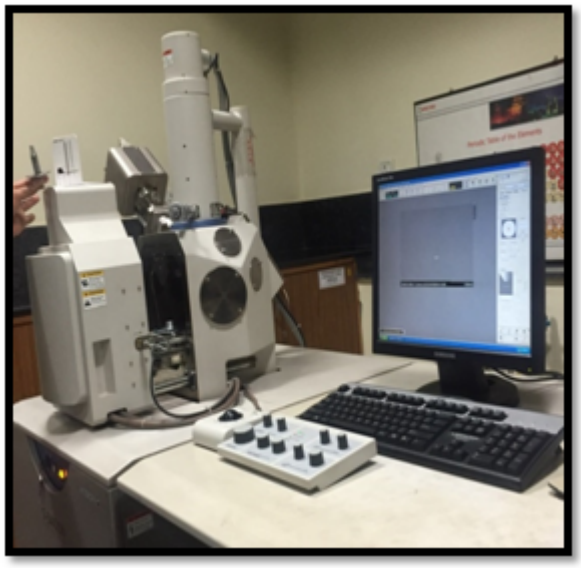


Fig.6.4 Scanned Electron Microscope (SEM Analysis)

The electrons are accelerated to the energy from 1 keV to 30 keV and surface diameter from 2-10nm. The specimens are observed with the different magnification for depth analysis. The magnification range is from 5x to 300000x. This is 150 times more than the optical microscope.

Salient features:-

1. Resolution of the image-3 to 10 nm
2. Accelerating voltage-0.3 to 30 keV
3. Magnification-5x to 300000x
4. Maximum specimen size-200 mm diameter
5. EDX spectroscopy-Metallic or oxide sample.

VII. RESULTS AND DISSCUSSIONS

1. WEAR TEST RESULTS:-

WEIGHT OF SPECIMENS AFTER THE CONDUCTING THE TESTS:-

Table 7.01: Specimen Weights after conducting the tests

Specimen No's	Wear Test Specimens (gms)
1	2.7486

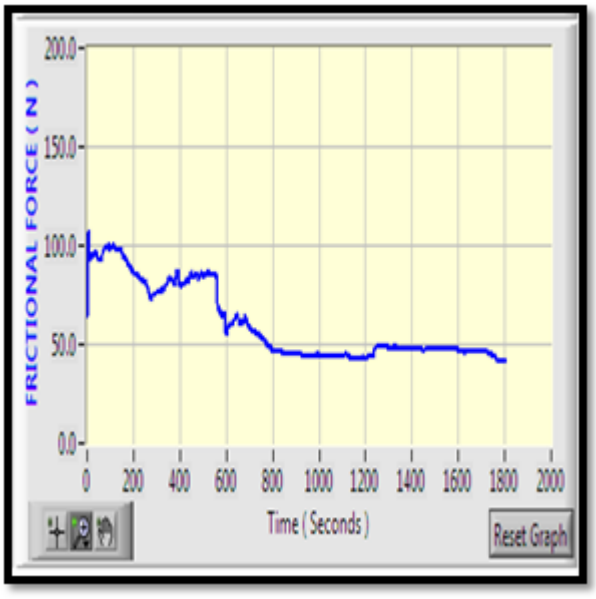
SPECIMENS AFTER TESTING:-

A) WEAR TEST SPECIMEN:-

Fig.7.1 specimen after wear test

For wear testing we consulted Ducom industries Bengaluru, in order to get computerized graph which is more accurate. The sample having the size 10mm diameter and 150mm length is subjected for wear test. The load is 200N, speed is 300rpm and time is 1800 sec. The initial weight, final weight, weight loss is calculated.

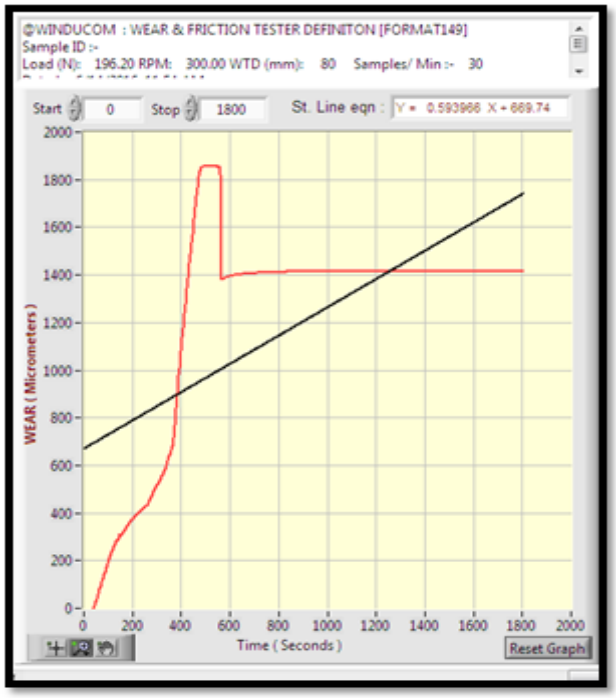
GRAPHS:-



Graph 7.1 frictional force v/s Time

Table 7.1 Wear Test Results

Sl No	Load (N)	Wear Track (Mm)	Speed (Rpm)	Time (Min)	Temp (°C)	Wear (Microns)	F.F (N)	Cof	Weight before Test (Gm)	Weight after Test (Gm)	Weight Loss (Gm)	Graph
1	200	80	300	30	-	1416	61.2	0.306	3.24609	2.74864	0.49745	obtained



Graph 7.2 Wear v/s Time

Graph 7.3 Coefficient of friction v/s Time

2. SCANNED ELECTRON MICROSCOPE (SEM ANALYSIS):-

To study the microstructure of composite material having the 10% of both Al_2O_3 and ZrO_2, we consulted the JSW steels Ltd.in order to get the quality SEM images. So here we observed for different magnifications like 50,100,200,400 and 500.

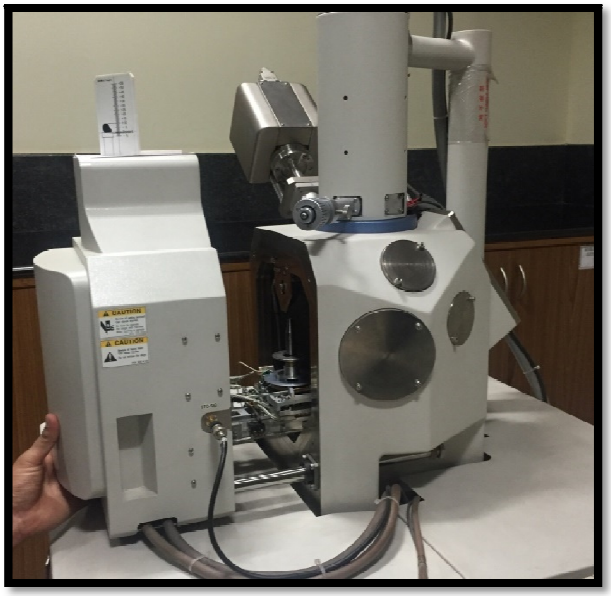


Fig.7.2 sectional view of scanned electron microscope

SEM IMAGES OF DIFFERENT MAGNIFICATION:-

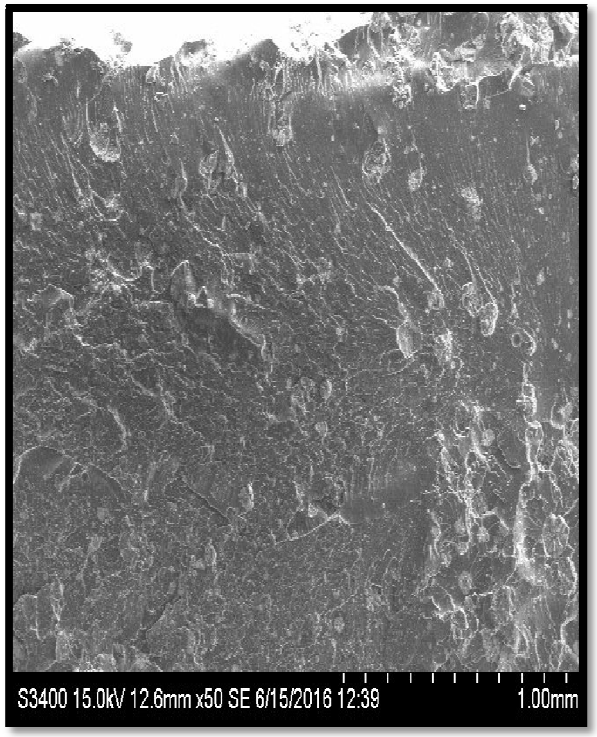


Fig.7.3 SEM Image for magnification 50

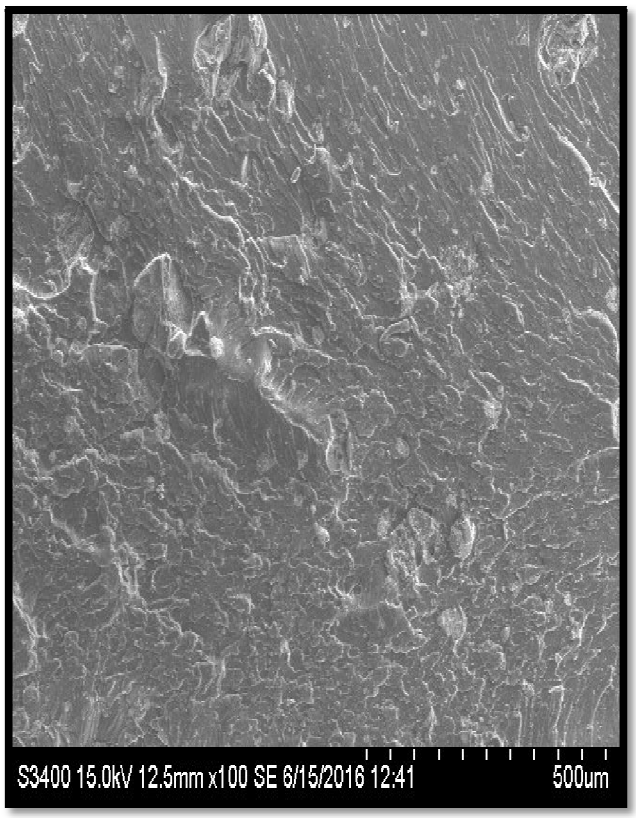


Fig.7.4 SEM Image for magnification 100

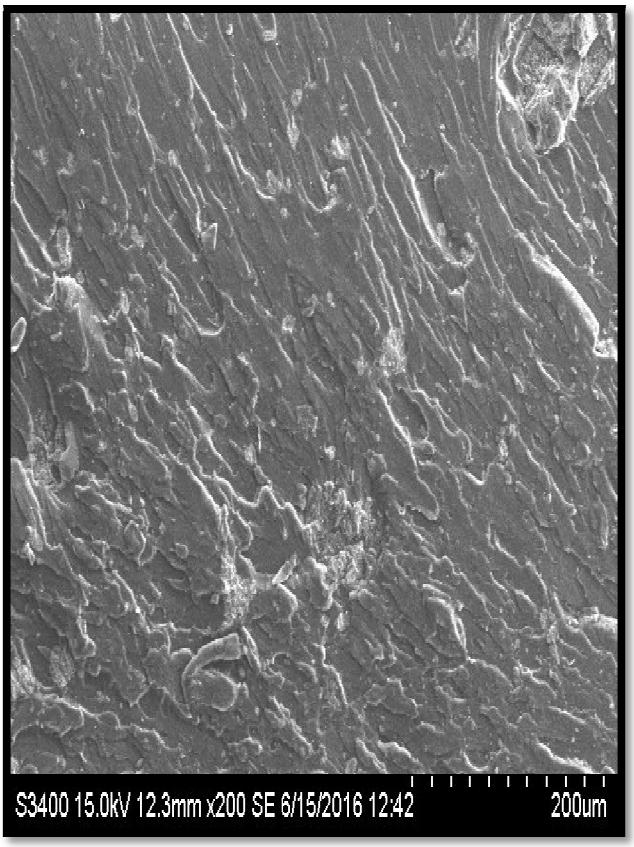


Fig.7.5 SEM Image for magnification 200

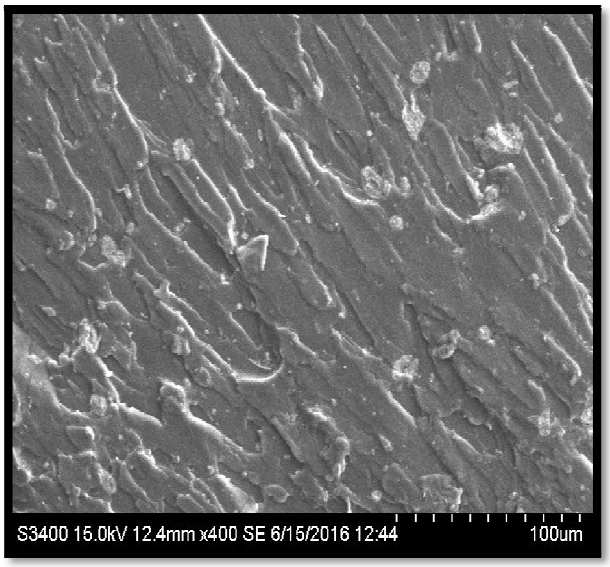


Fig.7.6 SEM Image for magnification 400

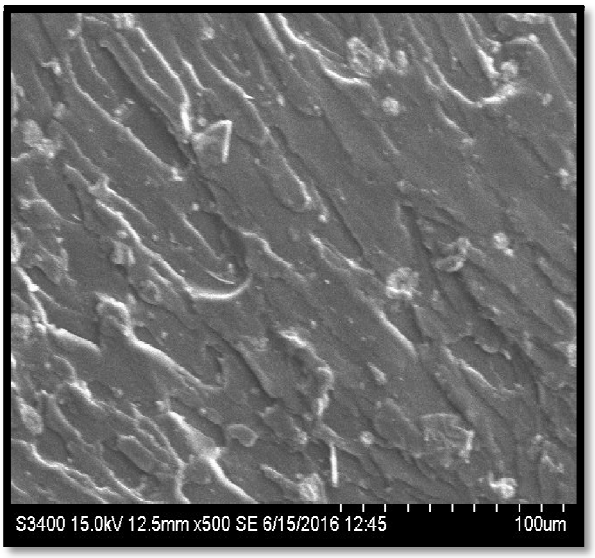


Fig.7.7 SEM Image for magnification 500

VIII. CONCLUSION

It conclude that the different tests are made to check whether the properties of polymer composite material will matches with the femur bone material properties at different ages or not. The wear test is conducted and tested for 200N, 30min for 300rpm. The initial weight is 3.246 gms, final weight after wear is 2.7486 gms. So weight loss we identified after the wear process is 0.4974 gms. And also by analyzing the SEM Images of fractured surface of the polymer composite material, it is concluded that still there are some places where the bonding is not uniform. Some projections are observed. So it is suggested to prefer vacuum pressure moulding technique or still more efficient moulding technique for uniform bonding of the material.

FUTURE SCOPE

1. The advanced bio matrix material i.e. high density polyurethane resin etc. and by using the different types of fibres the characterization can be done to improve the strength.
2. It can also possible to conduct DME analysis to study the damping analysis, temperature that material can withstand and stiffness of the material.
3. Fatigue test is done to measure the maximum strength that material posses. Coating test is made for better surface of implants and we can make different coatings like plasma spray coating and high velocity oxygen fuel spraying (HVOF).
4. Corrosion test can be used know whether the material will corrode or not. This test is done by keeping the material in artificial saliva for 48 hours.

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