

# **TRIBOLOGICAL BEHAVIOUR OF ZIRCONIUM DI-OXIDE CERAMIC**

A THESIS SUBMITTED IN PARTIAL FULFILMENT  
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## **CERTIFICATE**

This is to certify that the thesis entitled, “**STUDY OF TRIBOLOGICAL PROPERTY OF  $ZrO_2$  CERAMIC**” submitted by **Mr. DIGVIJAY SINGH & Mr. PUSHPA NARAYAN DHAKAL** in partial fulfillment of the requirements for the award of **Bachelor of Technology Degree in Mechanical Engineering** at **National Institute of Technology, Rourkela** is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma.

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**DIGVIJAY SINGH**

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## **ABSTRACT**

Ceramics are gaining importance due to their non-carcinogenic and bio-degradable nature. The ZrO<sub>2</sub> ceramic material is of prime importance. The structure of ZrO<sub>2</sub> is monoclinic crystalline structure. It occurs in form of mineral baddeleyite. The properties of ZrO<sub>2</sub> are: - Density= 5.68 g/cm<sup>3</sup>, molar mass=123.218 g/mol. It has good thermal insulation, high electron conductivity due to present of free oxygen ion. It is used in refractory, sensor, diesel engine and also as diamond simulant.

Keeping this in view the present research work has been under taken with an objective to explore the tribological properties of ZrO<sub>2</sub> ceramic. The ZrO<sub>2</sub> ceramic has been made by epoxy and hardener in ratio of 10:1 and ZrO<sub>2</sub> weight is 10%, 20%, 30%, 40%, 50% by cumulative weight of epoxy and hardener weight. Basically. To study the wear properties the components made from this ZrO<sub>2</sub> ceramics is subjected to wear test using a PIN-ON-DISC machine. Experiments have been conducted under laboratory conditions to access the wear behavior of the ceramics. The loss of weight has been measured in different percentage of ZrO<sub>2</sub>, with different load applied and with velocity varies from 200 to 500 rpm. The results have been drawn on graph and this shows the wear property of ZrO<sub>2</sub> ceramics with sliding distance and load applied.

The result shows that on increasing the load of pin on disc apparatus, the wear rate will increase. The structural property before and after the experiment have been drawn seen by scanning electron microscope (SEM).

Wear test shows that ceramics are less prone to the variation of speed as compared to the normal load applied. The lowest wear is occurred at 30% of the ceramic and highest wear occur at 40% of zirconia with epoxy and hardener. Wear occurs due to plastic deformation. The surface formed after the wear is studied in the SEM (scanning electron microscope). It shows that, as the percentage of zirconia increases, the surface fracture decreases up to 30% of zirconia but after that, the surface fracture increases.

**KEYWORDS-** monoclinic, ZrO<sub>2</sub> ceramic, PIN-ON-DISC a machine, sliding distance, SEM, zirconia

## **CHAPTER-1**

### **INTRODUCTION**

#### **1.1: Background**

The developments of ceramic material after meeting the challenges of aerospace industry have cascaded down for catering to domestic and industrial applications. Ceramics, the wonder material with light-weight, high strength-to-weight ratio, thermal insulation, electron conduction and stiffness properties have come a long way in replacing the conventional materials like wood, plastics and metal etc. The material scientists all over the world focused their attention on ceramic to cut down the cost of raw materials. Over the last thirty years ceramic materials have been the dominant uprising materials. Number of applications of ceramic materials is growing steadily, penetrating and conquering new markets relentlessly. Present day ceramic material constitutes a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications. They are usually optimized to achieve a particular balance of properties for a given range of applications.

While ceramics have already proven their worth as weight & volume saving materials, the present challenge is to make them cost effective. The present days efforts is to produce economically attractive ceramic have resulted in several innovative manufacturing techniques currently being used in the industry. It is obvious, especially for ceramics, that the improvement in manufacturing technology alone is not enough to overcome the cost difficulty. It is very essential that there should be an integrated effort of material processing, tooling, designing, manufacturing, quality assurance and even program management for ceramic to be competitive with metals and plastics.

Ceramics are generally inorganic, non-metallic solid that are prepared by action of heat and cooling. This present work aims at the present the wear properties of zirconia ceramic. Due to this wear property, other properties can also be studied like hardness and surface roughness.

In recent years ceramics have attracted substantial importance as a potential structural material. The most common attractive & basic featured ceramics that make them useful for industrial applications are light weights, low costs & high specific modulus.

## 1.2: WEAR

When two surfaces comes in contact with each other and there is a relative motion between these two surfaces. This leads to loss of material from the surface. This loss of material is called as wear. Wear lead to the loss of mechanical properties of material. It occurs due to the plastic deformation of the material.

Today there are many methods to determine the wear of a material under the specific condition. ASTM international produces a standard wear testing for specific application.

When the loss of dimension of an engineering component exceeds the limit of tolerance, then the life of that engineering component ends. Wear is an ageing process and as the age increases it leads to the failure of material. Like wear, other ageing processes are fatigue stress which depends on the fatigue concentration and repeated load.

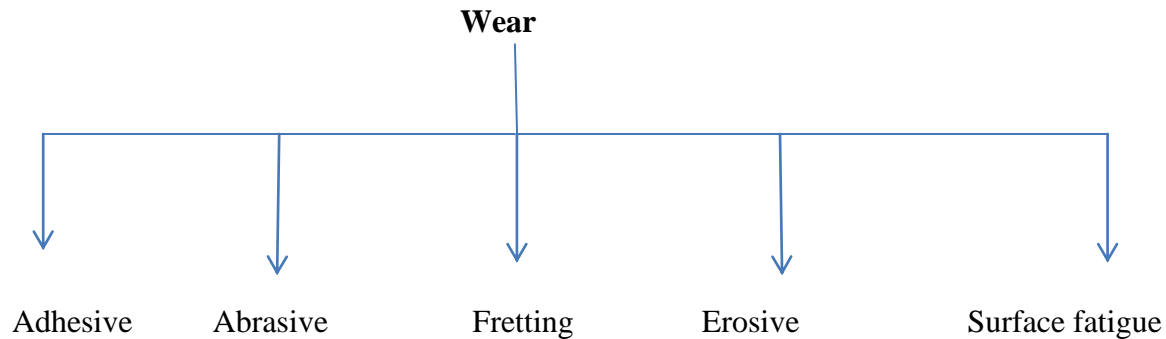
Wear is one of the slow processes. Due to wear, component losses its enormous properties and leads to failure.

There are three stages of wear in any components.

- Primary stage is run in period, In which the material contact is necessary. In this stages the wear may be high and low
- Mid age process, In which the steady rate of ageing occurs. This is the period for most of operational component life
- Old age period, in which the components are failing very high rate.



### 1.3 TYPES OF WEAR-



**Adhesive wear**- It occurs due to the surface friction between two contacting surfaces and are refers to as unwanted displacement and attachment of wear particle. Adhesive wear occurs due to the relative motion of the contacting surfaces and cohesive forces between the materials in contacts. When one body slides over other then adhesive wear occurs. It leads to the surface roughness and lumps.

**Abrasive wear**- it occurs when a harder surface slides over the softer surfaces. It depends on the type of contact & contact environments. It further classified as two bodies and three body abrasion wear. Two body occurs when a hard surface grit remove the material from other surface that is opposite to it. In three bodies, abrasion wear the particle in between two surfaces is free to move and roll. Mechanism for the identification of abrasion wear is plowing, cutting and fragmentation. Abrasion wear can be measured by loss of mass by Taber Abrasion Test

**Surface fatigue**- it is process by which the surface of a component weakens by the cyclic loading. Due to cyclic loadings the micro-crack forms, which are sub-surfaces cracks or superficial crack?

**Fretting wear**-fretting means repeated cyclic rubbing between two surfaces and this leads to the removal of material from one surface or both surfaces. Bearing is an example of this type of wear. Fretting leads to the fretting corrosion in presence of water.

**Erosive wear**-it exists for short period of time. Erosive wear occurs when particle or liquid forces to impact on the material with a force. Due to impact of the particle, the wear occurs. It is widely seen in industry. The rate of erosive wear depends on particle hardness, distance between nozzle and material surface, angle of impact and other factors. When Angle of impact reaches  $30^{\circ}$  the highest wear occurs in ductile material. While that for brittle material, the angle of impact should be normal to the surface for maximum wear to occur.

#### **1.4: Aim of present work**

Zirconia is ceramic with excellent property such as light weight, thermal insulating property and electro conductivity. In present days ceramics are replacing metal because ceramic is non corrosive and have more life than metal. The preparation of ceramics material is very crucial stage because at a particular sample percentage, it shows excellent property. Its wear rate will be less and surface fracture will be less.

So this present work is related to the finding of the percentage of zirconia with the hardener and epoxy for which ceramics material shows excellent property.

## CHAPTER-2

### LITERATURE SURVEY

*In 1990*, V. Maurice, M. Salmeron, G.A. Somorjai, the zirconium di-oxide surface are deposited and the surface properties are viewed by XPS, AES AND LEED. Fewer than 900 k zirconia is formed in fcc structure. The (111) plane of zircon growth parallel to (111) substrate plane. [1]

*In 1993*, R. Stevens, W.M. Rainforth, there are 3 mechanism are identified for the wear of ceramics. Transformation occurred along the [100] directions that led to formation of micro-crack. Wear rate increases due to transformation from tetragonal to the monoclinic phase. Secondly, clear tribo-chemical wear mechanism of the zirconia was found out. A minor role was played in Transformation of the tetragonal to monoclinic phase up to 200nm depth only .Finally, abrasive grooving causes extensive plastic deformation. Interestingly, there is no evidence of transformation or fracture was found at the abrasive grooves. This is demonstrated that under the particular sliding conditions, transformation toughening can have a beneficial as well as detrimental effect on the tribological properties of zirconia.[2]

*In 1994*, A. Tucci, L. Esposito., they studied the zirconium di-oxide and alumina. They studied sliding against the pin-on-disc tribometer. They found that the at all velocity the coefficient of friction is very low. They studied the surface by SEM and found out that the wear is due to abrasion and plastic deformation. [3]

*In 1997*, Carl G. Ribbing, Arne Roos ,zirconium nitride has sufficient hardness, young's modulus and high melting point. The high strength of zirconium nitrite is due to the formation of covalent bond between the nitrides. [4]

*In 2006*, Andrew W. Batchelor, Gwidon W. Stachowiak, the empirical method are employed for surface coating so there is not any clear idea about which of the surface treatment is best suited for the specific application. Wear resistance substances are very expensive in bulk but for small film they provide good economic and minimizing the wear problem. Lubrication position method is should be critically efficient because even powerful lubricant can scrape off if the deposition method is incorrect.[5]

**In 2010**, V. Naglieri, M. Lombardi, L. Montanaro , L. Joly-Pottuz, J. Chevalier, thermal treatments of a surface modified  $\alpha$ -alumina powder to which a zirconium oxide precursor was imbedded can be tailored to obtain an alumina–zirconia composite powder with well-controlled size and phase distribution. Transformation of the zirconia precursor to zirconia Nano-grains bonded to the alumina particles from a starting amorphous phase was followed by X-ray diffraction at room temperature and different thermally treated powders, as well as high-temperature in situ ) and by both conventional and transmission electron microscopy of high resolution (HRTEM and TEM ). Nanostructure and Phase evolutions were followed on a large temperature span. Nucleation-growth mechanisms by exploiting Avrami–Johnson–Mehl–Kolmogorov formalism were used to explain the crystallization kinetics.[6]

**In 2011**, Michael Behr, Peter Proff, Carola Kolbeck, Sabine Langrieger, Johannes Kunze, Gerhard Handel, Martin Rosentrit, the bond strength of zirconia with resin cannot found by bond strength test. Silicon contains phosphine and mono and di-phosphate do not provide sufficient tensile strength in long terms. [7]

**In 2013**, M.J. Hadianfard, E. Salahinejad , M. Mozafari ,D.D. Macdonald, D. Vashae, L. Tayebi, preparation of Zirconium titanate multilayer thin were done by particulate sol-gel process and after that spin coating. Obtained structures were viewed by SEM, TEM and atomic force microscope. The result showed that up to three layer sound films were developed and accompanied by increase in thickness. The coating consist of average dia. Of 50 nm of globular Nano-particles. [8]

**In 2013**, Johannes Karl Fink, epoxy-resins, which are formed from an oligomer that contains a curing agent and at least two epoxide groups. A variety of such monographs and resins is available on the market. The method of manufacturing and the specialties of these epoxy resins are elaborately explained. To enhance and strengthen it for all conditions there are many special additives used in the formation of epoxy resin. Few possible reactions are used for curing in practice. Mechanical properties of epoxy resins can be traced back and correlated to the constituting monomers. It is very difficult in recycling of wastes of epoxy resins.[9]

## CHAPTER-3

### APPARATUS AND MATERIAL USED

#### 3.1. Test tube-12X75 5ml



**Fig 1: test tube for preparation of sample**

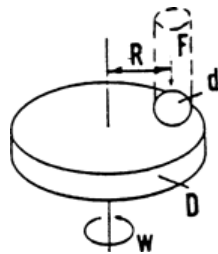
Test tube is used for casting or preparation of the sample. It is made by borosil company, which is largest glassware manufacturer in India.

### 3.2- PIN ON DISC MACHINE



**Fig-2:** set up for study of abrasive wear of sample

Pin on disc machine is used for study of wear properties of ceramic.



**Fig-3:** attachment of sample on disc

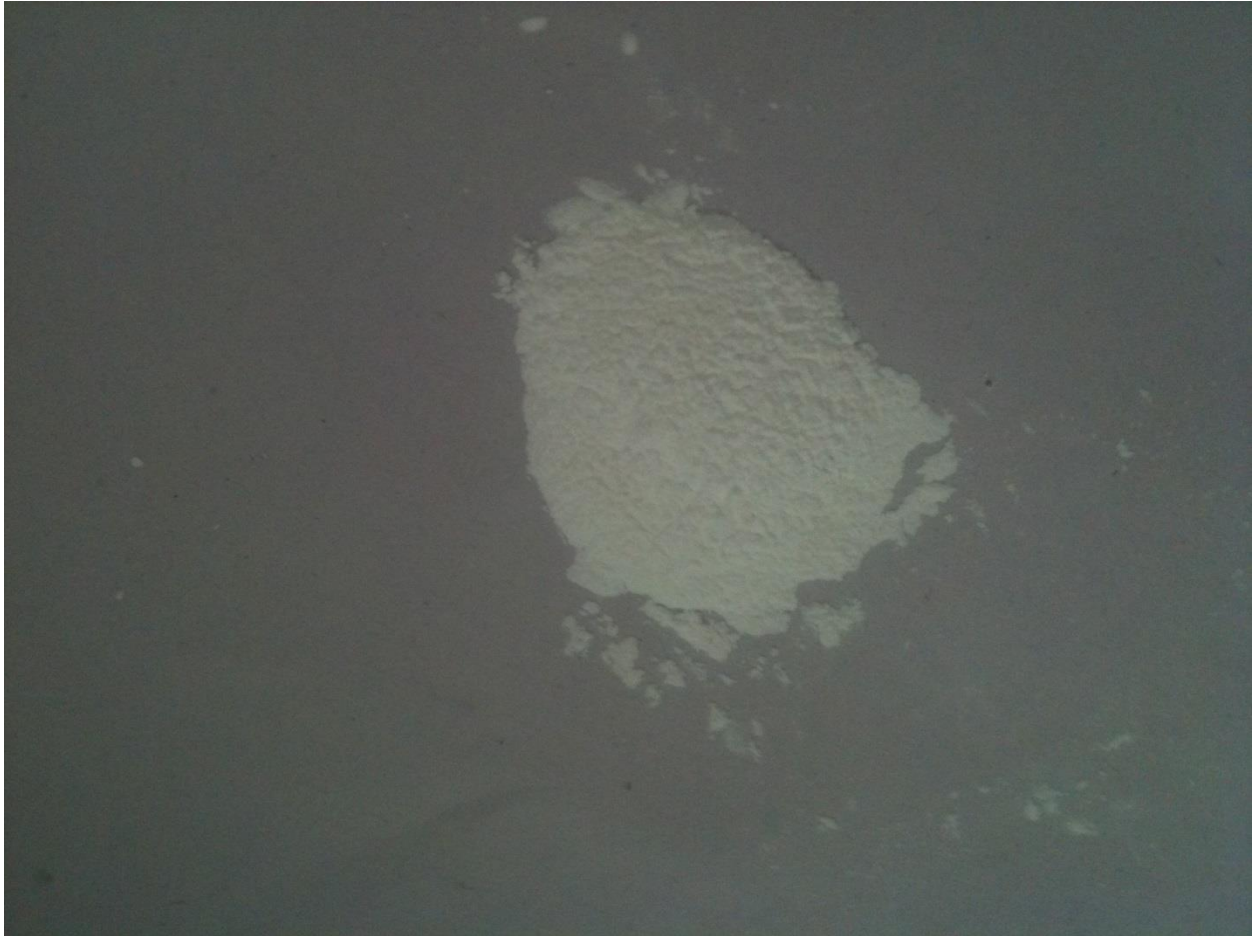


the load is applied by the application of hanging load. The load acts normal to the disc. The disc is 190 mm dia. and 9mm thick. It is rotated by motor. Disc can rotated up to 2900 rpm speed. The load is applied normally.

### **3.3- ZrO<sub>2</sub> SAMPLE-**

Molar mass=123.22

It is white in color.



**Fig-4**-zirconium dioxide in powder form

### **3.4- HARDNER**

Density-0.90gm/ml

### **3.5- EPOXY RESIN**

Density-1.12gm/ml

### Specification of pin-on-disc machine:

Load applied	Up to 3.5 kg
rotational speed	Upto500 rpm
Loading Lever Ratio	1:1
Disc Type	180 mm dia,9 mm thick
Pin diameter	1.25 to 2 mm
Wear or Displacement	- 2000 microns to + 2000 microns
Frictional Force	Up to 2.5 kg
Drive	1.15 kilowatts D.C. motor, constant torque
Motor Control	Thyrister converter
Power	230 Volt Line, 15 amps, single phase, 50 Hz. A.C.

### 3.6 Sample preparation

Taking 10 ml of epoxy and 1 ml of hardener. Take zircon 10%, 20%, 30%, 40% and 50% by the sum of weight of epoxy and hardener. mix them completely. Pour the sample in the test tube up to 3ml only. Leave them for 24 hours, so that they mix completely.

By breaking the test-tube, sample is taken out. Clean the surface and take the initial weight in weight measuring machine.

Weight of zirconium di-oxide=(weight of epoxy resin+weight of hardener)x percentage required

$(\text{Weight})_{\text{ZrO}_2} = (1.12 \times 10 + 0.90 \times 1) \times \text{percentage required}$

**10% sample** (Weight)  $\text{ZrO}_2 = 12.1 \text{ gm.} \times 10\% = 1.21 \text{ gm.}$

**20 % sample**(Weight) $\text{ZrO}_2 = 2.42 \text{ gm.}$

**20% sample** (Weight)  $\text{ZrO}_2 = 3.63 \text{ gm.}$

**40% sample** (Weight)  $\text{ZrO}_2 = 4.84 \text{ gm.}$

### 3.7 Experimental procedure

After the preparation of sample, clean the disc of pin on disc machine. Measure the weight of the sample. Put the sample on the pin and tighten it with the bolts so the sample does not vibrate .give the weight of 1.5 kg to the normal to the disc. Give the power to the machine and vary the speed from 200 to 500 rpm. After 5 minute, remove the sample and measure its weight.

Find the weight loss



Then again put the sample take the 4 consecutive readings. Again increase the weight to 2.5 kg and then repeat the process and take the 5 consecutive readings

Weight loss = initial weight- final weight

$$\Delta W = IW - FW$$

Wear rate in N/m was calculated by using the following formula:

$$WR = \Delta W / d_{sd}$$

Where

WR is wear rate

$d_{sd}$  is sliding distance

Sliding distance was calculated by using the following formula:

$$d_{sd} = 2\pi r N t$$

Where;

$r$  is radius of wear track,

$N$  is sliding speed in RPM of disc

$t$  is time for which disc rotates.

Sliding speed ( $V$ ) in m/s is calculated by using the following formula:

$$V = 2\pi r N / 60$$

Wear track radius was measured and its value was 5.5 cm.

Wear rate was calculate for different load

$$SWR = \Delta W / d_{sd} \rho F_N$$

Where:  $F_N$  is normal load (in N)

## CHAPTER-4

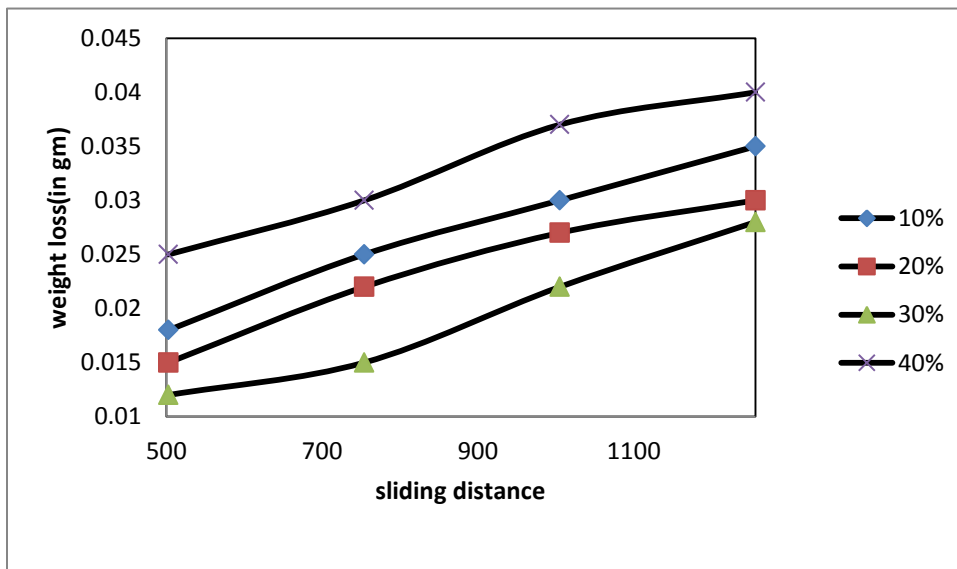
### RESULTS AND DISCUSSION

The result of the abrasive wear are shown by the graph with the variation of 15 N load and 25 N load

#### 4.1 VARIATION OF WEIGHT LOSS & SPECIFIC WEAR RATE WITH SLIDING DISTANCE

For

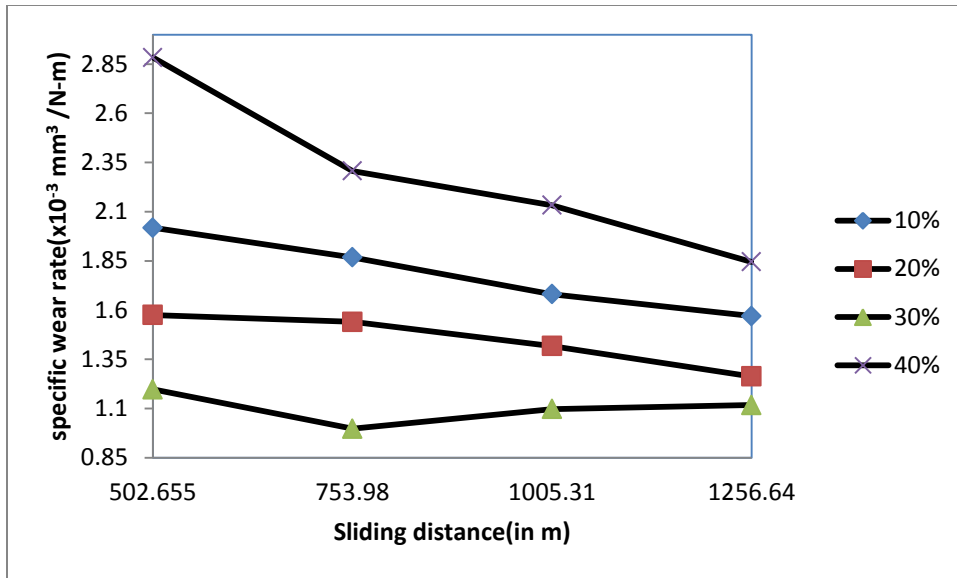
normal load=15N, r=8 cm, time(t)=5minute



**Fig-5** variation of weight loss with sliding distance

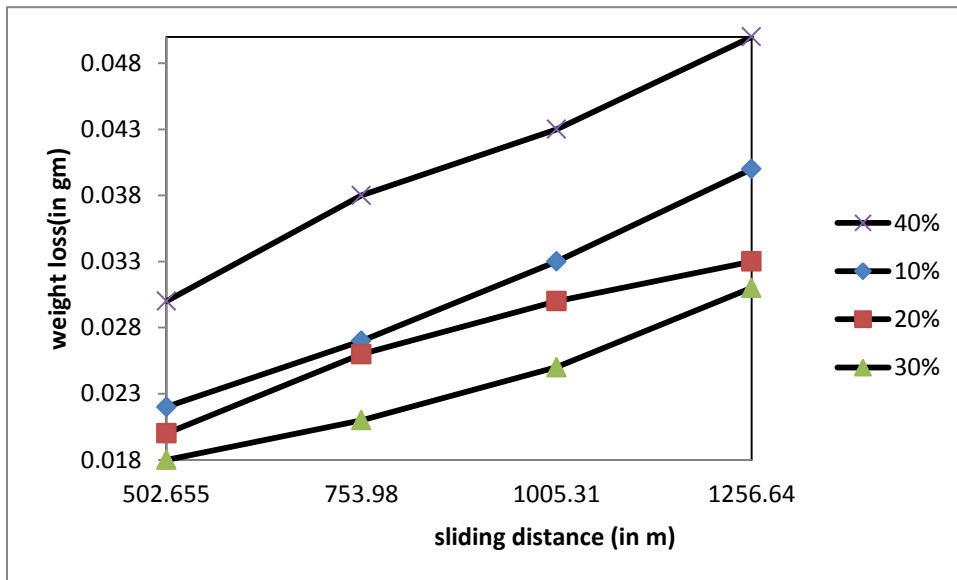
The above graph is clearly showing that as the sliding distance increases, the weight loss will increase. Highest wear loss is with 40% zircon with epoxy and hardener. And lowest with 30%

The graph for specific wear rate with sliding distance shows that as the sliding distance decreases the specific wear rate increases.

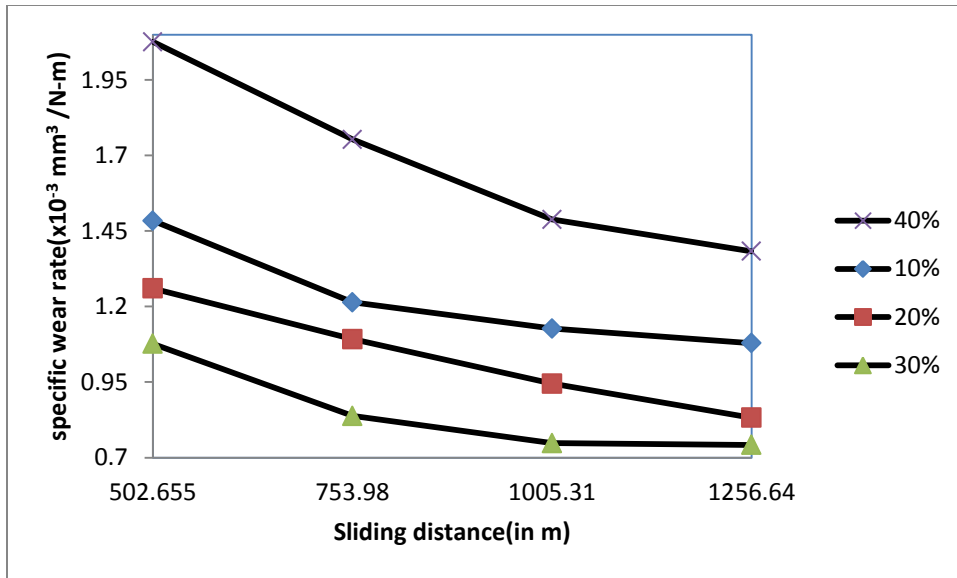


**Fig-6:** specific wear rate with sliding distance for 15 N load

**For normal load=25 N, r=8cm,time(t)=5 minutes**



**Fig:7-**variation of weight loss with sliding distance



**Fig-8:** specific wear rate with sliding distance for 25 N load

The variation of wear loss with sliding distance for 25 N normal loads is drawn on above figure. The specific wear rate vs. sliding distance is drawn on figure 8.

It is clearly shown in figure that there is almost linear weight loss with the sliding distance. So, we can predict that it is a steady wear. Because the wear rate will be almost same .highest loss of mass is with the ceramic that is made up of 40% zirconium di-oxide with epoxy and hardener. The lowest wear mass loss is with 30% zircon with epoxy. The wear mass loss is decreasing from 10% to 30% and then it increase from30% onward.

The lowest specific wear is  $0.9 \times 10^{-3} \text{ mm}^3 / \text{N-m}$  and highest specific wear is  $2.88 \times 10^{-3} \text{ mm}^3 / \text{N-m}$  with the load of 15 N. Specific wear rate is decreases as the sliding distance increases. lowest specific wear rate is  $0.74 \times 10^{-3} \text{ mm}^3 / \text{Nm}$  and highest SWR is  $2.2 \times 10^{-3} \text{ mm}^3 / \text{Nm}$  with the load of 25 N.so from above graph we can predict that as the load increases the SWR decreases. So as the load increases the SWR decreases.

#### **4.2 Hardness**

As the percentage of zirconia increases, the hardness increases up to 30% of zirconia from 10% zirconia. It shows that the ceramics with around 30% of zirconia shows the best hardness. But after 30% zirconia the hardness decreases. Hardness decreases is due to void formation and improper chemical reaction of zirconia and epoxy resin.

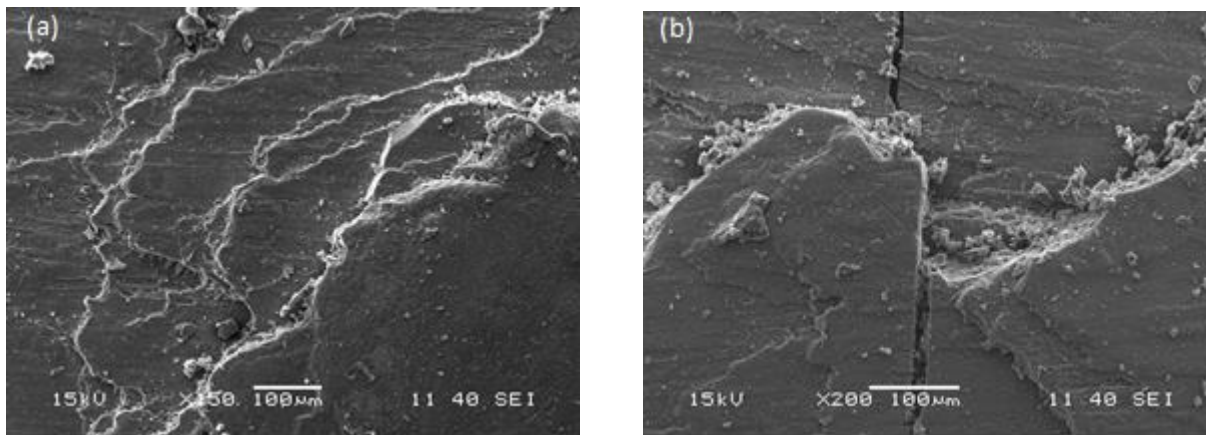
It increases from 10% to 30% because of increases of proper reaction.

### 4.3 Density-

Density calculated by formula of weight fraction is not same as the calculated by experiment. This in agreement can be understood by the void formation and incomplete zircon ceramic. The sample is prepared in some container and then it is poured in test-tube. In this whole process, there is some material loss. This loss, result in decrease in density of actual sample as compared to the formula density. It also affect some of mechanical properties of the ceramics i.e. tensile strength, hardness etc. also fatigue resistance is decreases as the void formation increases

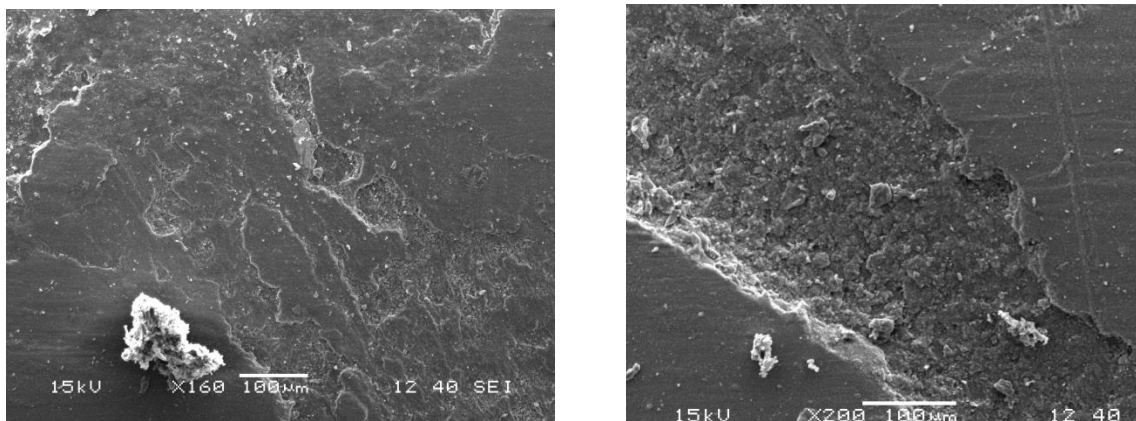
### 4.4 Surface morphology.

For the view of wear surface better, SEM machine is preferred.

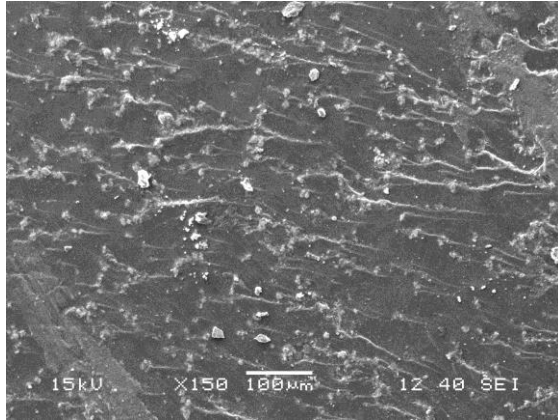


**Fig-9:** photo micro graph of worn out surfaces of 10% sample with 15N load

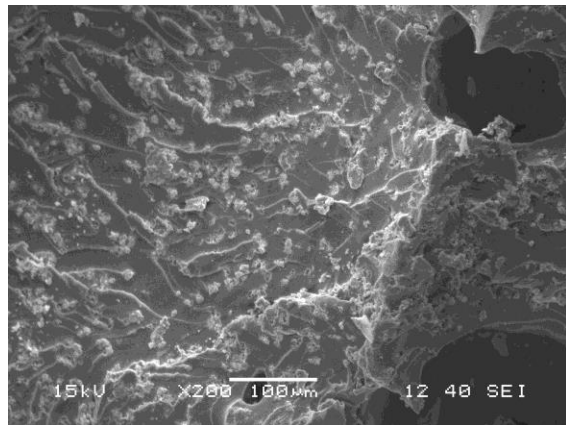
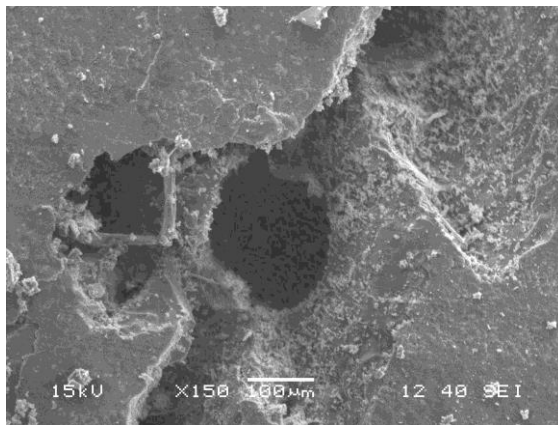
From figure it is seen that with increase in load the worn out surfaces will be more and rougher.



**Fig-10:** with 20% sample of zirconium di-oxide



**Fig-11:**with 30% sample of zirconium di-oxide



**Fig-12:**with 40% sample of zirconium di-oxide

From the figure it is clearly visible that the pattern followed by the worn out surfaces.

As the percentages of zirconia increases, the surface roughness and crack decreases up to 30% of zircon. But for the 40% of zircon, the void is seen and surface crack also exist.

From above figure, it is seen that with 30% of zirconium di-oxides the worn out surfaces is good and there is no any crack is seen.

## **CHAPTER-5**

### **CONCLUSION**

Based on the above experiments, the following conclusion is drawn.

1. The density and hardness is less as compared to the formulated density because of the void formation.
2. The wear loss will be increase from the 10% to 30% of the zirconium di-oxide sample and then it increases from 30% to 40% of the sample.
3. As the load increases, the wear loss is increase.
4. As the speed of rotation increases the wear loss is increases.
5. As the speed of rotation increases, the specific wear rate decrease
6. The crack formation and surface worn out decreases from 10% sample to 30% sample and again it increases from 30% to 40% sample

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