

**FABRICATION OF Al7075/ ZrB<sub>2</sub> SURFACE COMPOSITE  
VIA FRICTION STIR PROCESSING**

**A**

**Project Report**

*submitted in partial fulfillment of the requirement for the award of the  
degree of*

**MASTER OF TECHNOLOGY**

*In*

**PRODUCTION ENGINEERING**

*By*

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## DECLARATION

I, **Manoj Kumar Sah**, hereby declare that the project work, which is being presented in this dissertation entitled “**Fabrication of Al7075/ ZrB<sub>2</sub> Surface Composite via Friction Stir Processing**” in the partial fulfillment for the award of degree of Master of Technology in Production Engineering, is an authentic work carried out by me at **Delhi Technological University** under the guidance of **Dr. Vipin** , Professor and **Sh. N. Yuvraj**, Asst. Professor of Mechanical Engineering Department.

I have not submitted the work in this dissertation for the award of any other degree or diploma to any other university.

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## CERTIFICATE

This is to certify that the project report entitled “**Fabrication of Al7075/ ZrB<sub>2</sub> Surface Composite via Friction Stir Processing**” submitted by **Manoj Kumar Sah** (Roll No. **2K13/PIE/25**) in the partial fulfillment for award of degree of Master of Technology in Production Engineering at **Delhi Technological University**, is a bonafide record of his own work carried out under our supervision and guidance.

To the best of our knowledge, the result contained in this thesis have not been submitted in part or full to any other university for the award of any degree or diploma.

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## ABBREVIATIONS

HAZ	Heat Affected Zone
TMAZ	Thermo Mechanically Affected Zone
UTS	Ultimate Tensile Strength
COF	Coefficient Of Friction
FSP	Friction Stir Processing
FSW	Friction Stir Welding
SEM	Scanning Electron Microscopy
EDS	Energy Diffraction Spectroscopy
XRD	X -Ray Diffraction

## ABSTARCT

Friction stir processing is new surface modifying technique to fabricate surface composite layer. A specially designed rotating tool with shoulder and pin is plunged into the surface of material and then processed. The rotating tool pin causes intense plastic deformation of material due to stirring action. The processed region results in refined grain structure and uniform dispersion of reinforcement particles in the matrix. In this project work different volume of  $ZrB_2$  reinforcement particles inserted into the Al 7075 alloy and then processed by FSP. Fabricated surface composites were analyzed with microstructural, EDS and XRD studies. Mechanical and wear properties were also examined. To understand the wear and fracture mechanism of composite SEM study were also carried out .The results shows that composites are having higher mechanical and wear properties than the base material. Around 10 % increase in mechanical properties and more than 50% reduction in wear rate was observed. The higher volume content of  $ZrB_2$  particles exhibited higher tensile and wear properties compared to lower content of  $ZrB_2$  particles. However at higher content of  $ZrB_2$  particles there was marginal increase in tensile strength but great reduction in elongation.

# CHAPTER 1

## INTRODUCTION

### 1.1 Friction Stir processing

Friction stir processing is a new developed solid state technique and based on the principle of friction stir welding which was invented in 1991 at The Welding Institute (TWI) of U.K [1]. Initially friction stir welding was used to join two soft metals but based on that friction stir processing is used for improving the mechanical and wear properties of the metals like Aluminum, magnesium, copper and their alloys etc. Material selection with specific properties is very important parameters in many industrial applications like in aircraft, shipbuilding and automotive industry. High specific strength can be achieved by very fine and homogeneous grain structure. Sometimes increase in strength is required without much loss of ductility. Friction Stir Processing refines the microstructure by thermo-mechanically stirring the material locally .A non-consumable tool having pin and shoulder is rotated and plunged into the surface of the material(Fig.1).Heat is generated between the contact surface of pin, shoulder and the material due to friction(Fig.2). This frictional heat generated is below melting point of the material and enough for plastifying the material .There are some forces which are acting on the stirring zone. Vertical downward force exerted by shoulder allows the pin to plunge into the material after sufficient heating, the transverse force is also developed by the tool movement in longitudinal direction. Faster the tool feed rate, higher will be the transverse force. The tool geometry can have many pin profiles like square, cylindrical, tapered cylindrical threaded, triangular etc. with different shoulder diameter based on base material properties. The ratio of pin diameter to shoulder diameter is very important parameter which affects the quality of processed area. The ratio of pin to shoulder dia. ( $d: D$ ) generally 1:3 to 1:3.5 has shown better results [5].Friction stir processing can also be used for fabricating metal matrix composites. Initially a groove or set of holes are made, the reinforcing particles such as  $Al_2O_3, SiC, TiC, B_4C, ZrB_2$  etc. are filled in that groove or holes and then rotating tool is plunged and traversed along that groove or set of holes. The number of processing passes can be increased to mix the particles in the metal matrix uniformly. The metal matrix composites prepared by this technique have shown improvement in mechanical as well wear properties.

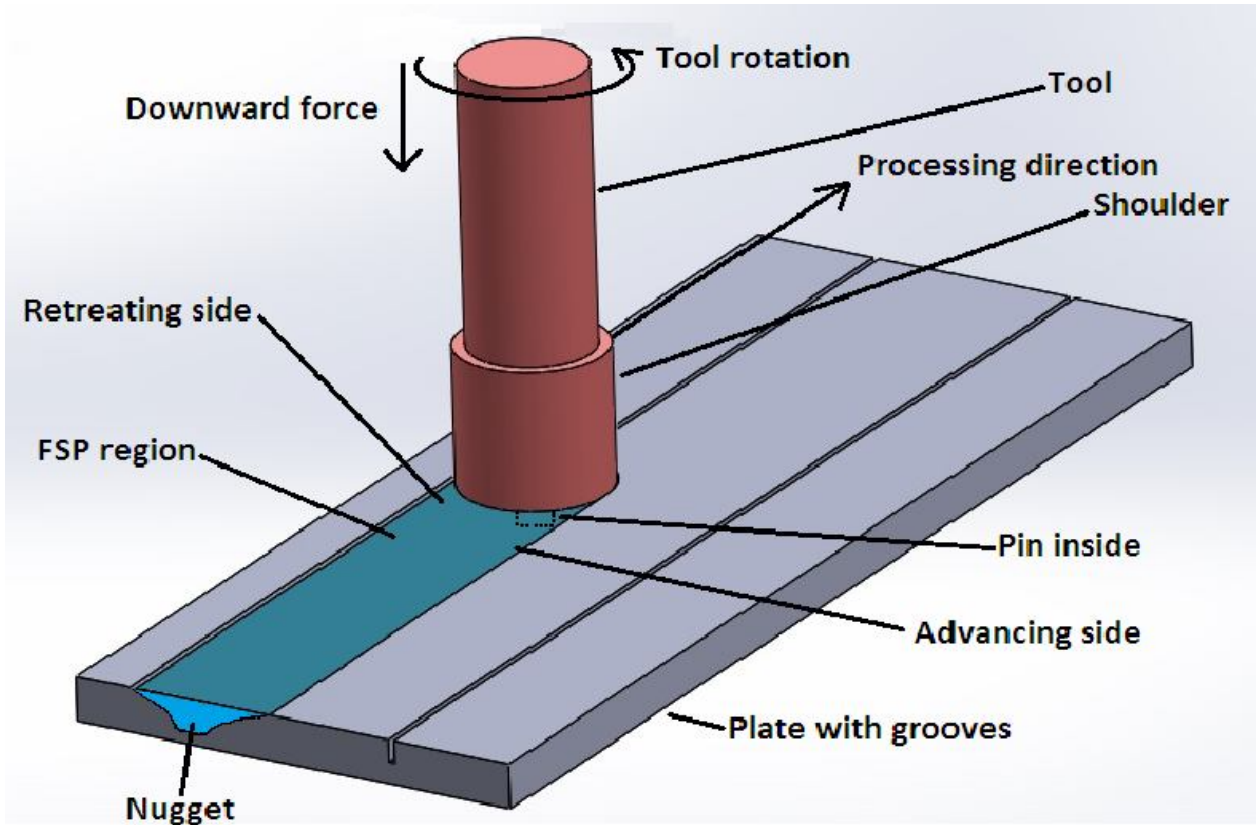


Fig.1. Schematic diagram of FSP



Fig.2.FSP of a plate

## 1.2 Aluminium and its Alloys

Aluminium is silvery white metal and possesses following properties:

Light in weight, density about one third of steel, high strength to weight ratio, good conductor of heat and electricity, high resistance to corrosion, very ductile, non-magnetic, have melting point about 660 °C. Aluminium when combines with other alloying elements becomes even stronger and achieves better properties. Some of the alloys of Aluminium are as listed below:

Alloy of Al-cu, alloy of Al-Silicon, alloy of Al- mg, alloy of Al-Zn-Mg, alloy of Al-Mn, alloy of Al-Mg-Silicon, alloy of Al-Lithium, alloy of Al-mg-Zn-Cu, alloy of Al-other alloying elements etc.

The demand of Aluminium and its alloy in industries arise from their attractive physical, mechanical and chemical properties. Their use includes:

Transportation industry, structural framework, engine parts, decorative items, hardware ,making doors, window frames, tanks, furnishing and fitting work, making parts of trucks, buses, automobile cars, aerospace industry, marine area, heat exchangers, food industry, refrigeration parts, storage containers, etc.

According to the Aluminium Association (AA), USA, there are some standard 4 digit designation codes to identify the composition of any particular Aluminium alloy. This standard is adopted by most of the countries.

The first digit indicates the major alloying element.

1000 series is pure Aluminium with 99% Aluminium by weight and it can be work hardened. It is widely used in food packaging industry.

2000 series contains copper as major alloying element and can be precipitation hardened. It is commonly used alloy for aerospace industry but it is easily subjected to stress corrosion cracking.

3000 series, it has manganese as major alloying element and it can be work hardened. Mainly used for roofing sheets.

4000 series, they are alloy of mainly silicon. It is also called Silium. Silicon lowers the melting point .So it is used as filler material in welding, soldering, and brazing.

5000 series are alloy of magnesium. It has high resistance to corrosion. Widely used in marine and offshore industry. The amount of magnesium should not be in excess otherwise the stress corrosion resistance will decrease.

6000 series, they are mainly alloyed with magnesium and silicon. Their machinability and weldability is good and can easily be precipitation hardened. It is used in building construction, transportation etc.

7000 series, this is having Zinc, Magnesium as main alloying element. It has highest strength among other Aluminium alloys. It can also be precipitation hardened. It is somewhat difficult to fabricate by conventional technology as it requires proper control of fabricating parameters. It is very costly and hard as compared to other Aluminium alloys.

8000 series, it is alloy of Aluminium and lithium. Lithium is very light in weight because of low density. It has density even less than Aluminium. So it is used to make the alloy even lighter and increase the strength to weight ratio. Its uses includes as bearing components of cars, trucks and nuclear power generation etc.

### **1.3 Composite Materials**

**They are classified as:**

**Metal –Matrix- Composites (MMCs)** - mixture of ceramics and metals .

**Ceramic-Matrix-Composites (CMCs)** –ceramic particles like SiC, Al<sub>2</sub>O<sub>3</sub> etc. imbedded with fibers to improve properties, especially in high temperature applications.

**Polymer-Matrix-Composites (PMCs)** – thermosetting plastics/resins are widely used.

Examples: epoxy and polyester with fiber reinforcement, phenolic with powders.

**Functions of the Matrix Material** (primary Phase)

It forms the bulk, base or mass of the product of the composite material, it holds the imbedded phase or reinforcing in place, the matrix shares the load with the secondary phase, it sometimes partially deforms to transfer the stress to the reinforcing particles or fibers.

**Functions of the Reinforcing Phase** (Secondary Phase)

It supports the primary phase, shares the load of primary phase, provides extra strength and rigidity to the composite. The secondary phase may be in the form of Fibers, Particles, and

Flakes. The secondary phase can also be used as an infiltrated phase in a porous matrix or skeletal.

## **1.4 Metal Matrix Composites (MMCs)**

Metal as a matrix and ceramic particles as secondary phase. The secondary phase may be :

1. Particles of ceramic (these MMCs are also called CERMETS)
2. Fibers of various materials

The secondary phase or reinforcement can have volume fraction in the range of 10-70%. .MMCs offers a wide range of properties enhancement over monolithic-alloys. MMCs have found application in many areas of our daily life over the last years. Materials like cast iron with graphite or steel with high carbide content as well as tungsten carbide, consisting of carbides and metallic binders, also belong to this group of composite materials. Metal matrix composites become interesting for use as constructional and functional materials, if the property profile of conventional materials either does not reach the increased standards of specific demands, or is the solution of the problem.

### **The reinforcement of metals can have many different objectives**

Increase in yield strength and tensile strength at room temperature and above while maintaining the minimum ductility or rather toughness. Increase in creep resistance at higher temperatures compared to that of conventional alloy. Increase in fatigue strength, especially at higher temperature. Improvement of thermal shock resistance, Improvement of corrosion resistance, Increase in Young's modulus, Reduction of thermal elongation etc.

## **1.5 Advantages of MMCs**

Light Weight, Performance at higher temperatures, High Strength, Low Density, Better wear resistance

## **1.6 Production of MMCs**

Powder metallurgy, casting, rolling, diffusion bonding, friction stir processing etc.

## **1.7 Advantage of the FSP process**

It improves the mechanical properties like tensile ,yield and fatigue strength etc., it refines the grain size and increases hardness, improves superplasticity of the material, less

distortion ,no spatter, no porosity, and there is no fume, better efficiency in terms of energy saving, less or negligible shrinkage of processed zone, no filler material is required, the tool is non-consumable, no shielding gas required, processing is possible in all direction, no ultra-violet radiation etc.

### **1.8 Limitations of the FSP process**

Tight and rigid clamping of material is required, sometimes backing plate is required to support from bottom, high shoulder pressure is required, a key-hole is left at the end of the process, tunnel defect or wormhole may occur if the processing parameters are incorrect.

### **1.9 Application of FSP**

Shipbuilding, offshore, marine area, automobile industry, aerospace industry, fabrication of metal-matrix surface composites, changing the mechanical property of any selected region or part locally etc.



# CHAPTER 2

## LITERATURE STUDY

### 2.1 Literature Review

Friction stir technology is a very innovative technology in the field of material joining or processing. It has shown great results in processing of some light materials like Al, Mg and Cu etc. Friction stir processing can be considered as a hot working process in which the work piece undergoes a large amount of deformation locally through a rotating tool having shoulder and pin. This highly deformed area (nugget) is covered by pin diameter and the rest of the area covered by shoulder. The shoulder provides extra heat from rubbing at the surface and softens the region ahead of the pin which helps the pin to traverse in linear direction smoothly. The area surrounding the nugget is TMAZ (thermo mechanically affected zone) and HAZ (heat affected zone). The nugget comprise of very fine, equiaxed and dynamically recrystallized grains. Researches have shown great improvement in mechanical as well as wear properties at nugget zone.

There has been extensive researches and study on microstructures of the processed zones. There are also studies on temperature distribution and its effect on microstructure of the processed region. Some studies are also available on precipitation phenomenon. Hardness profile of different region of processed zones has also been studied experimentally. Studies on tool wear and different optimum design of tool are also available. Mechanical and wear properties have also been studied.

Some overview of researches in the field of friction stir technology is presented in this section as below:

**K. Elangovan and V. Balasubramanian [2]** studied about the effect of different tool pin profiles and tool shoulder diameter on FSP of Al 6061 (Al–Mg–Si alloy). The tool pin profile used were straight cylindrical, tapered cylindrical, threaded cylindrical, square and triangular having length 5.5 mm. Each pin with three different shoulder diameter as 15, 18 and 21mm. So total tools used were 15 in numbers. FSP was carried out with 1200 rpm and 1.25 mm/sec of traverse speed at 7 KN of shoulder pressure. They found that out of 15 tools, the tool having square pin showed better properties of the stirred zone irrespective of the

shoulder diameter and the tool having 18 mm shoulder diameter showed good quality of zone. In overall the tool having square pin with 18 mm shoulder dia. showed better results.

**G. Casalino et al. [3]** studied about the Influence of Shoulder Geometry and Coating of the Tool on FSW of 3 mm thick 5754H11 Aluminium alloy butt weld Plates .They varied the diameter and slopes of the shoulder and tested the coating of Tungsten Carbide on the tool. They found that the conic tool produces smooth surface and little flash, the large shoulder coated carbide tool gives defect free weld and the shoulder diameter affects the grain size of TMAZ and hence the micro-hardness profile.

**L. Trueba et al. [4]** studied about how does tool shoulder affects the tensile properties of friction stir welded aluminum 6061-T6. Six different tool shoulder's bottom surface (raised, recessed, spiral and ramp type with different variation in surface texture and roughness) of the material Ti-6Al-4V was used. The speed taken was 1200 rpm at traverse of 8.10 cm/min. The shoulder dia. of the tool was 20mm with 6 mm length and width. The tool pin was of non-threaded, flat-bottom, cylindrical type. The tool having raised spiral surface produced better quality weld.

**S. Malarvizhi and V. Balasubramanian [5]** studied about influence of ratio of shoulder diameter (D) to plate thickness(t) of dissimilar friction stir welded joint of aluminum-magnesium alloys( AA6061- AZ31B).Plate thickness of 6mm and different shoulder dia. of 12,15,18,21 and 24 mm respectively. He studied that D/t ratio of 3.5 i.e. shoulder dia. of 21mm and plate thickness of 6 mm gives better quality weld with maximum tensile strength of 192 MPa.

**V. Malika et al. [6]** studied and analyzed the effect of various tool pin shape on FSW of Aluminium alloy(2024-T3 ).Various tool pin shapes like straight and frustums of circular ,triangular, square, rectangle, pentagon ,hexagon were used. After making a model, FEM analyses was conducted and concluded that pin having square profile produced less defect and providing frustum aids to the less power consumption.

**Magdy M.El-Rayesa et al. [7]** studied about the effect of number of passes on FSP of 6mm thick plate of 6082-T651 AA. Tool rotation taken was 850 rpm and traverse speed of 90, 140 and 224 mm/min with one, two and three number of passes .Tool made of Mo-W steel with shoulder dia. of 15mm and square pin. Increasing the number of passes led to the

increase in grain size of stir-zone and more heat production leading to dissolution of  $\beta$ -phase which further leads to the reduction in UTS, increasing the traverse speed reduces the size of second phase particle and increases the UTS.

**K. Elangovan and V. Balasubramanian [8]** studied about the effect of pin profile and rotational speed of the tool on stir zone of 6 mm thick rolled plate of AA2219 Aluminium alloy. He used 5 different tool pin profiles (straight, cylindrical, tapered-cylindrical, threaded-cylindrical, triangular and square) with three different tool rotational speed. He found tool with square pin at rotation of 1600 rpm produces defect free and good quality stir region.

**Weifeng Xu et al. [9]** studied the influence of various pin profile and other parameters on the weld quality of Aluminium alloy 2219(Al-Cu alloy). Studies have shown that because of copper content the 2xxx series of Aluminium alloys have poor weldability by fusion welding processes. The copper content causes poor solidification, hot cracking and porosity in the fusion area. So this research was carried out to see if these defects could be minimized by friction stir processing or not. The speed ranged from 300 to 500 rpm and traverse speed varied from 60 to 100 mm/min. The tool used were of threaded pin with three spiral and threaded pin with triangle. Better plastic flow was observed by threaded tool with three spirals. The speed at 300 rpm produced more fine grains and distribution of secondary phase was more diffused as compared to 500 rpm. Hardness at 300 rpm was more than 500 rpm as obvious because of fine grains. The traverse speed did not show any notable change in hardness.

**H. Jamshidi Aval [10]** studied the effect of pin profile on weld quality of dissimilar Aluminium alloys (AA6082-AA7075). AA6082 is an alloy of Al-Mg-Si and AA7075 contains Al-Zn-Mg as main alloying elements. Three different tool pin profiles, tool 1 with square frustum pin 3 to 9 mm in dia. ,7.9 mm in length ,tool 2 with square frustum pin 4.5 to 9mm in dia. and 7.9 mm in length, tool 3 with conical frustum pin with 4.5 to 9mm in dia. with three grooves were used .All the tools were having 2° concave at the bottom of the shoulder surface to accommodate the stirred material. The speed ranged from 1000 to 1200 rpm and traverse speed varied at 9 to 12cm/min. It was observed that the temperature generated by square frustum pins was higher and produced fine grains with proper mixing of material at weld zone as compared to conical frustum pin. The Slope of the decrease in hardness at the AA7075 side was very sharp than the AA6082 side.

**M. Salehi et al. [11]** studied and optimized the process parameters of producing surface composite of AA6061-SiC nano composite via FSP by applying Design of Experiments(DOE).Investigation was done on the effect of mainly four factors rotational speed, traverse speed, pin profile and tool penetration depth. Total 16 runs of FSP was done .The parameters range taken were ,tool speed 800-1600rpm, traverse speed 40-160 mm/min, pin depth 0.12 to 0.24 mm. The pin profile included was square and threaded only. Their influence on the quality of the processed zone in terms of percentage was investigated and it was found that the effect of tool rpm is 43.70%, traverse speed 33.79%, depth of tool penetration 4.21% and pin profile 11.22%.The optimum condition were found to be speed at 1600 rpm, traverse speed at 40mm/min, pin depth 0.30 and with threaded type of pin.

**S. M. Bayazid et al. [12]** studied the effect of different pin profiles like triangular, square and cylindrical on FSW of AA 7075-T6, 5 mm plates at tool rotation of 1600 rpm and tool traverse speed of 63mm/min. it was observed that tunnel defect occurred in cylindrical and triangular pin. Other defects like kissing bond, zig-zags were found in cylindrical pin only. The triangular pin produced some cracks on advancing side. But in overall the square pin produced defect free stirred region.

**J. M. Piccini and H. G. Svoboda [13]** studied about the effect of pin length of friction stir spot welding of lap joint of dissimilar metals, Aluminium alloy and steel. An Aluminium alloy AA6063 plate was welded with a galvanized low carbon steel plate. Different pin lengths were used ranging from 0.65 to 1.5 mm. The shorter pins resulted better quality of welds.

**M. Felix Xavier Muthu and V. Jayabalan [14]** studied the effect of traverse speed on friction stir welding of Aluminium AA1100-H14 with pure copper .Tool traverse speed varied from 50 to 90 mm/min and other parameters were kept constant. The welding was done in perpendicular direction to the direction of rolling of plates formation of layers of intermettalic compounds like Al<sub>2</sub>Cu, AlCu, Al<sub>4</sub>Cu were confirmed by X-ray diffraction technique(XRD), thermal imaging microscope(TEM ) and energy diffraction spectrum(EDS).It was observed that traverse speed of 70 and 80 mm/min resulted in better quality of joint. Also uniform distribution of copper particles over Aluminium resulted in better tensile strength.

**M. Ghosh et al. [15]** studied optimization of parameters of friction stir welding of dissimilar Aluminium alloys A356 and AA6061. The speed ranged from 100 to 1400 rpm and traverse speed at 80 to 240 mm/min. Other parameters were kept constant. The tool used was of high speed steel having 15 mm shoulder diameter, 5 mm cylindrical pin diameter with pin length of 2.6mm. the shoulder pressure used was 5KN with tool tilt angle of 3°. It was observed that low tool speed and low traverse speed resulted in fine grain size, less thermal stress, uniform distribution of Si rich particles. In overall low traverse speed and low rotation resulted in superior mechanical properties.

**L. Karthikeyan et al. [16]** studied about the effect of various processing parameters on friction stir processing of AA2285 alloy. Tool speed used were 1400 and 1800 rpm at different traverse speed of 10,12,15 mm/min respectively. Tool made of high speed steel was used with 16 mm shoulder dia. and cylindrical pin having 5 mm dia. and 4.7 mm length. It was observed that increasing the rpm of tool there is increase in tensile strength, increasing the traverse speed decreases the tensile strength. with increase in rotational speed increase in micro hardness was also observed. Optimum result was observed at 1800 rpm at 12 mm/min of feed.

**N. Yuvraj et al. [17]** studied the effect of micro and nano sized B<sub>4</sub>C particles on mechanical and wear properties of Al 5083 Aluminium alloy. Effect of one pass and three number of passes were also studied. The tool used was of cylindrical threaded with 18 mm shoulder dia. and 5mm pin length and 6 mm dia. It was observed that processing with nano size B<sub>4</sub>C particle with three number of passes resulted in better mechanical and wear properties than micro sized B<sub>4</sub>C particles with one and three pass.

**N. Rajamanickam et al. [18]** studied the effect of various process parameters on generation of heat in stirred zone of Aluminium alloy AA2014 at different tool rotation and welding speeds. H13 tool material was used having 20mm, 6mm shoulder dia. and threaded cylindrical pin dia. Different tool rotations 355,710,1120rpm and different traversing speeds 30, 60,120 mm/min were used. So total of nine samples of different combination of parameters were made. The analysis of all these parameters were done by analysis of variance (ANOVA) and the effect on heat generation on processed zones were studied. It was analyzed that temperature of the area near the tool is more dependent on tool rotation as compared to traverse speed.

**L. Dubourg et al. [19]** studied the lap welding of Aluminium alloy AA7075 and AA 2024 by friction stir processing. These 7 series and 2 series alloys are mainly used in aircraft structural elements and are very difficult to be weld by other conventional welding. Four different variations of the welds having single pass, double continuous pass, double discontinuous pass and a riveted joint were studied. Other parameters taken were 500-2000 rpm rotational speed and 50-100 mm/min traverse speed, H13 tool with shoulder dia. 19, threaded cylindrical pin dia. 6.3 mm. It was observed that double pass continuous weld resulted in improvement in tensile as well as fatigue strength as compared to other welds.

**Zhikang Shen et al. [20]** studied the mechanical strength of the weld of AA7075 plates welded by friction stir spot welding. It is variation of friction stir welding. In this case the rotating tool is plunged for some seconds of time and then retracted back. It costs 25 % less than resistance spot welding. The plates of AA7075 of 2mm thickness was welded at different tool speed of 1500,1750 and 2000rpm for the time period of 3 ,4,5 sec respectively. Tool shoulder was 15mm with cylindrical pin dia. 5mm. It was observed that defects like hook, voids and bonding ligament were present at the weld area. Overall it was observed that the better weld joint is achieved at low rotational speed for shorter period of time.

**L. B. Johannes and R. S. Mishra [21]** studied about the effect of multiple number of passes on superplasticity of AA 7075 Aluminium alloy plate of 6.4 mm thickness. The processing was carried out at 400 rpm and 50 mm/min of traverse speed. The tool used was made of high strength cobalt alloy (MP159) with shoulder dia. 25 mm and pin dia. and length of 6.4 mm each. The tool pin profile was cylindrical threaded. After processing the tensile specimens were made and polished upto 1 micron surface finish. The tensile tests were carried out at different temperature range of 673 K to 763 K at different strain rate ranging from  $1 \times 10^{-3}$  to  $1 \times 10^{-1} \text{ s}^{-1}$ . It was observed that single pass processing resulted in higher elongation of upto 1200 % at temperature 740 K and at strain rate of  $1 \times 10^{-2} \text{ s}^{-1}$ . Although increasing number of passes resulted in slightly less superplastic property but still that values are in the region of superplasticity. It was also observed that grain boundary sliding mechanism was the main reason of superplasticity.

**I. Sudhakar et al. [22]** studied the wear and ballistic resistance of AA7075 Aluminium alloy by friction stir processing. Surface composite was made with B4C particles of different sizes 30, 60 and 120 micron. One more variation of B4C 30 micron+MoS<sub>2</sub> was there for

ballistic testing. H13 tool with 20 mm shoulder and 6 mm cylindrical pin dia. was used. The length of the pin was 3 mm. Tool rotation was 960 rpm with traverse speed of 30 mm/min and 50 mm/min. The samples were prepared for wear as well as ballistic testing. The ballistic testing was carried out as per the military standard (JIS.0108.01). It was observed that the sample with B4C 30 micron + MoS<sub>2</sub> resulted in better ballistic property. It was also observed that the insertion of B4C particles on the surface changes the wear mechanism from abrasion to adhesion. The wear rate was significantly reduced after insertion of B4C particles. The sample with B4C 30 micron and 60 micron resulted almost same wear rate but B4C + MoS<sub>2</sub> resulted in significant improvement in wear properties. Coefficient of friction was also reduced significantly.

**H. G. Rana and V. J. Badheka [23]** studied the wear and hardness properties of Aluminium alloy AA7075 surface composite with B4C particles. The size of the boron particles was almost 15 micron. A groove was cut in 6.5 mm thick plate of Aluminium alloy 7075. The approximate percentage of particle insertion was 12 to 15 % of volume fraction. The tool used was of WC-Co(12%) with tapered cylindrical pin with 18 mm shoulder dia. and 5 to 3 mm pin dia. the length of the pin was 3mm. Tool speed was 545 rpm with tilt angle of 3 deg. Traverse speed ranged from 50 to 120 mm/min. Wear test was carried out for 2000 m of distance at a load of 60N. It was observed that tool traverse speed has less effect on dispersion of particles. The hardness was decreased while increasing the tool traverse speed. Max. hardness was observed 144 HV at 50 mm/min traverse speed. On average basis there is 1.3 to 1.6 times increase in hardness and wear properties after FSP and FSP with particles.

**T. Dieguez et al. [24]** studied the superplastic behavior of Aluminium alloy AA7075-T651 by tensile testing at the temperature ranging from 350 to 750 °C. The strain rate ranged from  $5 \times 10^{-3}$  and  $2.5 \times 10^{-2} \text{ s}^{-1}$ . The tests were done on friction stir processed 4 mm plate thickness. The used was of H13 material with shoulder dia. 12.5 mm and square pin of side 2.5 mm. The shoulder was somewhat concave to accommodate the plasticized material. The tool speed was 514 rpm at 50 mm/min of traverse speed. The tilt angle of the tool taken was 1.5 deg. After FSP average size of grain size of stirred zone was 4.65 micron which is very refined size. The best results was reported at 400 °C at strain rate of  $1 \times 10^{-2} \text{ s}^{-1}$ . The percentage elongation observed was 900%. The maximum stress reached was 9 MPa.



**A. Hamdollahzadeh et al. [25]** studied about micro-structural and mechanical properties of friction stir processing of AA7075-O 6 mm plates with reinforcement of SiC particles of 45 to 65 nano-meter of size. H13 tool hardened to 58 HRC was used .The tool shoulder dia. 16 mm with 4.54 mm side of square pin was used having pin length of 5.7 mm. The volume fraction of particles was around 20 %. The tool speed was 1250 rpm at 40 mm/min of traverse speed. It was observed that particle distribution in 2 pass was better than single pass. Ultimate strength of 2 pass was somewhat reduced as compared to single pass because of coarsening of MgZn<sub>2</sub> precipitates in HAZ. 2 pass resulted in increase in heat input which led to increase in grain size which future led to increase in ductility.

**R. S. Mishra et al. [26]** studied the superplastic behavior of AA 7075-T651 Aluminum alloy of 6.35 mm thick plate. A single pass of 0.3 meter was made at traverse speed of 15 cm/min .the strain rate was varied from  $10^{-4}$  to  $10^{-1}$  s<sup>-1</sup> and the temperature range was 420 °C to 520 °C. The optimum result was observed at 490 °C of temperature having 1000 % elongation at  $10^{-2}$  s<sup>-1</sup>.

**M. Bahrami et al. [27]** studied the effect SiC particles of nano size (45-65 nm) on weld quality of AA7075 Aluminium alloy 6mm thick plates. The SiC particles were inserted into a groove of 5 mm deep and 0.4 mm wide. The volume fraction of particle were around 20 % .the tool speed was 1250 rpm at 40 mm/min of traverse speed. H13 tool was use with threaded cylindrical pin. It was observed that the grain size of Aluminum matrix was around 3.85 micrometer and cluster size of SiC particle was 80 nm-430 nm in stir one of specimen having SiC particles. The specimen which was not having SiC reported grain size 4.83 micrometer. The tensile test was carried out along transverse section and in both the cases the fracture was taken place from TMAZ area which shows that the bonding was better in the stir zone. Overall The Specimen With SiC particles resulted in better mechanical properties.

**Dongxiao Li et al. [28]** studied the effect of stationary shoulder friction stir butt welding of Aluminium alloy AA7075-T651 of 5mm plates. This technique is a variation of friction stir welding. In this there is rotating pin but the shoulder remains stationary and just slides over the surface of welding. It was reported that the stationary shoulder creates focused heat input and eliminates unnecessary flow of material. This results in narrow HAZ. The Tool Speed Was 1000rpm and 1500 rpm at traverse speed range of 30 to 50 mm/min. The samples for tensile test, hardness and toughness test were cut across the transverse section. It was



observed that both UTS and elongation was decreased as compared to base metal. But the specimen at 1500 rpm and 50 mm/min of traverse speed resulted maximum hardness.

**T. Srinivasa Rao et al. [29]** studied the friction stir welding of AA7075-T651 Aluminium alloy. Two different weld joints of plate thickness 10 mm and 16 mm were made. Tool material was M2 for both the welds but having different dimensions. Both Tools were having tapered cylindrical threaded pin. Tool one was having shoulder dia.22 mm and pin dia. at top 7mm and 4 mm at bottom. Tool second was having 30 mm shoulder dia. And pin dia. at top 10 mm and at bottom 8 mm .The length of the pin for tool first and second were 9.5mm and 15 mm respectively. Tilt angle for both tools was 1.5 deg. The tool speed and traverse speed for first tool was 700 rpm and 120 mm/min and for the second tool it was 500 rpm and 25 mm/min .Tensile test and micro structural observations was done on the specimens across the transverse section of the joint. Both the specimens were fractured from HAZ area. There is reduction in tensile strength and elongation of both the specimens as compared to base but the weld with 10 mm thickness was having somewhat higher strength than 16 mm thick weld. However impact strength of 16 mm thick weld was better than 10 mm thick weld.

**P. Cavaliere and A. Squillace [30]** studied the friction stir processing of Aluminium alloy AA7075-T6, 7mm thick plate .The FSP was perpendicular to the rolling direction of plate. The tool speed was 700 rpm at traverse speed of 2.67 mm/sec. The pin was 6.5 mm in dia. and 6.8 mm in length.The shoulder dia. was 20 mm. The tool was having tilt angle of 3 deg. The specimens were prepared for tensile and hardness tests. The tensile test was carried out at room temperature and at 150 to 500 deg. Celsius for different strain rates ( $10^{-2}$  and  $10^{-4}$  per second).The tensile strength was decreased almost 23 % ,and elongation 36 % across transverse section .the yield strength reduced from 524 to 325 MPa. In longitudinal direction the yield strength observed was 355 MPa and tensile strength was better than transverse direction. The elongation was better 500 deg. Celsius at  $10^{-3}$  per sec.

**S. D. Ji et al. [31]** studied the temperature effect on the behavior of material transfer of friction stir processed Aluminium alloy AA7075-T6. FSP was done on 6 mm thick plate. The tool was having threaded cylindrical pin with top dia. 6mm and bottom dia. 3 mm. The length of the pin was 5.6 mm. The shoulder dia. was 12 mm. The rotational speed of the tool ranged from 800 to 1200 rpm and traverse speed from 50 to 100 mm/min. The observations

of material flow was carried out at three stages initial, middle and final stage. The peak temperature observed in the initial stage was lower than the peak temperature in final stage. The material near the weld surfaces moves to downward and the middle of the weld material moves upward, and no material displacement near the bottom of the weld. Also the material transfer in advancing side was smaller than retreating side. It was observed that at final stage of welding the material transfer was highest.

**S. Gholami et al. [32]** studied the effect of post-process single and double aging treatment on friction stir processed AA7075 Aluminium alloy 10 mm thick plate. The H13 tool pin dia. was 5.5 mm and length 4 mm with shoulder dia. of 20 mm. The speed of the tool was 1200 rpm at 50 mm/min of traverse speed with 3 deg. of tilt angle. The bottom surface of the shoulder was 8 deg. Concave. After FSP the samples were cut into 15 X 50 X 10 mm and aging treatment was done that samples. In single aging the first sample was aged at 80 °C for 96 h, the second sample was aged at 120 °C for 72 h, and third sample was aged at 160 deg. Celsius at 16 h. In two step aging the fourth sample was first aged at 80 deg Celsius for 8 h and the temperature was increases at the rate of 2 degree Celsius per min and then again aged at 140 deg Celsius for 8 h. It was observed that around 50 % hardness was increased after FSP and around 30 % further increased after aging treatment .The hardness observed in single aging treatment was higher than double aging treatment.

**A. Goloborodko et al. [33]** studied the microstructural stability and mechanical properties of friction stir welding of aa7075-T6 Aluminium alloy by post heat treatment at different temperature of 623 k to 773K.The FSW was conducted on 3mm thick plate with tool speed of 1500 rpm at traverse rate of 300 mm/min. The samples were cut for testing the superplastic behavior and thermal stability of nugget zone. The different temperature range the samples were heat treated in air furnace for 30 min. of time. The tensile test was also conducted at different strain rate of  $1 \times 10^{-4}$  to  $1 \times 10^{-2} \text{ s}^{-1}$  at different temperature. It was observed that the average grain size of the nugget zone was 3 micron. After heat treatment the structure was stable upto 723 K .Above that temperature the the microstrucre becomes unstable and the grain size is increased in scale of mm. The maximum elongation of about 440 % was observed at 673k at strain of  $10^{-3} \text{ s}^{-1}$ .

**A. Sert and O. N. Celik [34]** studied the wear properties of AA7075-T651 Aluminum alloy and SiC particles surface composite made by friction stir processing. The processing

was carried out at 710 rpm tool speed at traverse rate of 20, 40, 56 mm/min. The test for wear behavior was done on ball on disc at sliding velocity of 2.5 cm/s. The load was varied in the range of 2, 4 and 5N. It was observed that the wear properties were improved. The rate of wear was increased at 5 N of load. It was also observed that the wear was mainly due to adhesion and abrasion.

**R. Ramesh and N.Muruga [35]** studied the hardness behavior of friction stir processed surface composite of AA 7075-T651 Aluminum alloy and B4C particles. The particles were filled in a groove of 0.5 mm width and 2.5 mm depth having 100 mm length. Different tool speed range of 425 to 575 rpm at different traverse speed range of 40 to 60 mm/min was used. The numbers of passes were varied upto 3 pass. So total of 16 experiments were conducted and a regression analysis was done based on these data. The equation obtained was  $BHN = 349.208 - 0.662 * RS - 6.387 * TS + 0.014 (TS * PS)$ . It was observed that maximum hardness obtained 64 BHN at 575 rpm at 60 mm/min of traverse speed.

**H. Bisadi and A. Abasi [36]** studied the effect of tool rotation and traverse speed on fabrication of surface composite of Al7075/TiB2 via Friction Stir Processing. The size of the particles used was 2.62 micron. H 13 tool was used with shoulder dia. 16 mm and pin dia. 3mm with pin length 5.4 mm. The tilt angle was 3 deg. Tool rotation was 450, 825, 1125 rpm at traverse speed of 32, 60 mm/min. It was observed that the hardness was increased by increasing tool rotation and traverse speed. The maximum hardness was obtained was 179 HV at 60 mm/min. There is also improvement in tensile strength and yield strength almost 2 times.

**G. Hussain et al. [37]** studied the microstructural and mechanical properties of surface composite Al7075-T651/TiN produced by multi pass-friction stir processing technique. Different tool geometries namely square, triangular, threaded cylindrical tapered. A groove of 1 mm width and 2.4 mm depth was made in 120 X100 X5 mm of plate. The insertion of TiN particles was around 18 % by volume. The traverse speed, rotation and number of passes were 1200rpm, 40 mm/min and 4 respectively. The tool tilt angle was kept at 4°. H13 tool was used with shoulder dia. of 15 mm. The pin dia. and length of each tool was 2.5 and 5 mm. Different samples were cut for microstructural, hardness and tensile test along the direction of processing. The particles distribution and hardness was found to be good by

threaded pin tool but the square pin tool resulted in maximum tensile strength and elongation. Some secondary phases were also observed in all the specimens which also acted as a strengthening factor. The wear properties are improved upto 34 to 56 % and the ductility of all the specimens were reduced in the range of 22 to 50%. The wear mechanism was dominated by adhesion.

## 2.2 Literature Gap

Previous studies and researches indicates lots of work carried out to study the microstructural, wear and mechanical properties of friction stir welded or processed Al 7075 Aluminium alloy. Researches are also available on fabrication of surface composite of Al7075 with reinforcement particles such as TiN, TiB<sub>2</sub>, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub> and SiC made by friction stir processing. The effect of ZrB<sub>2</sub> particles on Al7075/T651 as surface composite has not been studied yet. Also very less research works are available on study of tensile properties of Al7075 along processing direction.

So this project work is focused on study of effect of ZrB<sub>2</sub> particles on Mechanical and Wear properties of Al7075/T651 as surface composite fabricated by Friction Stir processing.

# CHAPTER 3

## EXPERIMENTAL PROCEDURE

### 3.1 Material Selection

**Matrix Phase:**            **Aluminium Alloy (Al7075/T651)**

This material is chosen as it is widely used in automotive and aerospace application because of its high strength to weight ratio among all kinds of Aluminium alloys. Its chemical composition is given in the table 1 below:

Table No. 1

Chemical composition of Al7075/T651

Zinc	Magnesium	Copper	Manganese	Silicon	Chromium	Titanium	Iron	Aluminium
Zn	Mg	Cu	Mn	Si	Cr	Ti	Fe	Al
5.9	2.4	1.3	0.04	0.05	0.18	0.06	0.21	Balance

**T651** is a designation given to a particular type of heat treatment of Aluminium alloys which includes solution treatment then cold rolling and at last artificial ageing to form second phase precipitates to strengthen the alloy. Some properties of Al7076/T651 are given in Table 2 below:

Table No. 2

Properties of Al7075/T651

UTS	Y.S	Elongation	Melting Point	Density	Hardness	C.T. Expansion
570 MPa	480 MPa	13%	600 °C	2.8 g/cc	86 HV	$23 \times 10^{-6} / ^\circ\text{C}$

**Reinforcement Particles: Zirconium Boride (ZrB<sub>2</sub>)**

Some properties of ZrB<sub>2</sub> are given in Table 3 below:

Table No. 3  
Properties of ZrB<sub>2</sub>

Melting Point	Density	Hardness	Thermal Conductivity	C.T. Expansion
3246 °C	6.08 g/cc	2970 HV	60 W/mk	6.8x10 <sup>-6</sup> /°C

### 3.2 Preparation of Work-Piece

- 1) With the help of hacksaw the plates of AA7075 was cut into 200 x 75 x 8 mm (Fig.4).
- 2) Two grooves of width and depth, 1 x 2 mm and 1 x 4 mm was cut by slitting saw on horizontal milling machine to fill ZrB<sub>2</sub> particles (Fig.5).
- 3) One plate was left without any groove for friction stir processing.
- 4) Plates were cleaned by Acetone properly.
- 5) Grooves (1 x 2 mm) and (1 x 4 mm) were filled with 5 % and 10 % vol. fraction of ZrB<sub>2</sub> Particles respectively.

The volume fraction was calculated by the relation given below [37].

$$\text{Volume Fraction} = \frac{\text{Area of groove}}{\text{Projected area of pin}}$$

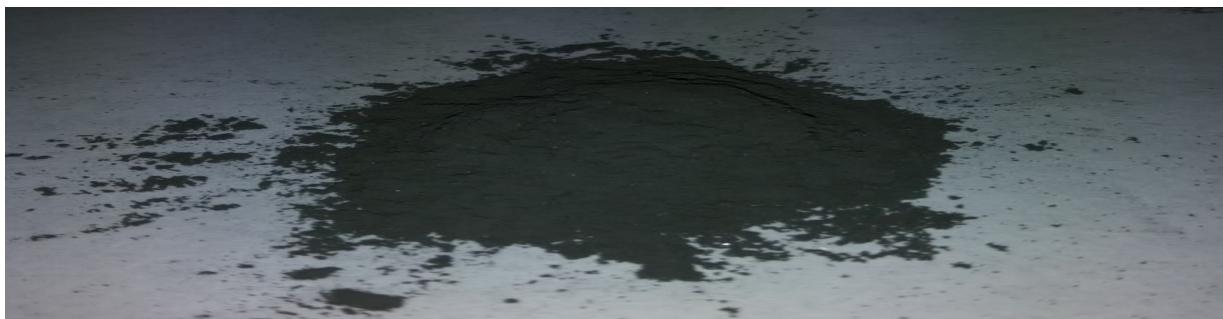


Fig.3. ZrB<sub>2</sub> Particles



Fig.4. Al 7075 Plate

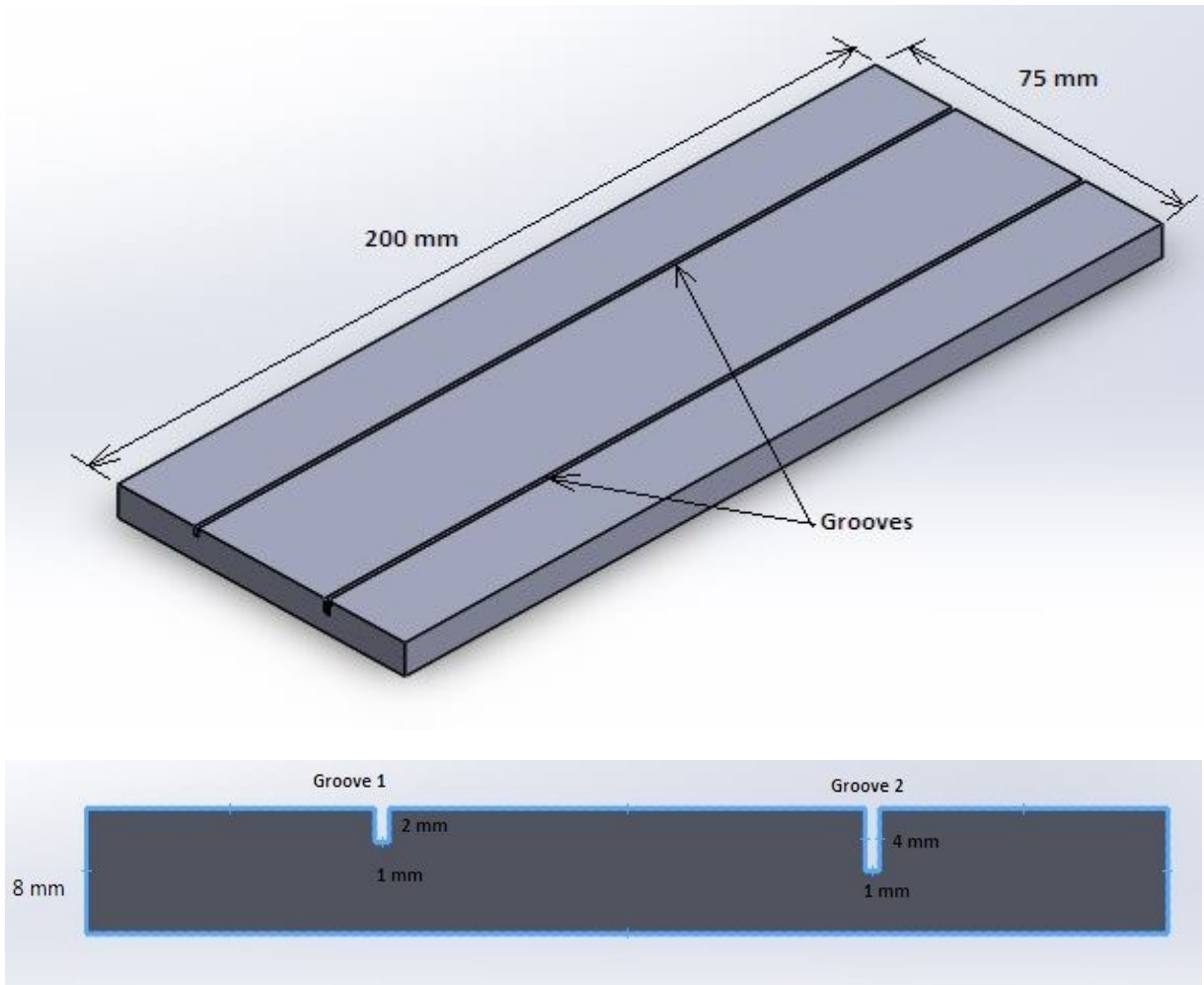


Fig.5. Grooves on plate (CAD Model)



### 3.3 Selection of Tool and Tool Geometry

Tool material selected was H13 with square pin profile. The tool was initially made by turning and grinding operations and then heat treated for getting hardness upto 58 HRC.

The composition of H13 (values in percentage) is given below in Table 4:

Table No. 4

Composition of H13 tool material

Chromium	Molybdenum	Silicon	Vanadium	Manganese	Carbon
4.75-5.50	1.10-1.75	0.80-1.25	0.80-1.20	0.20-0.60	0.32-0.45

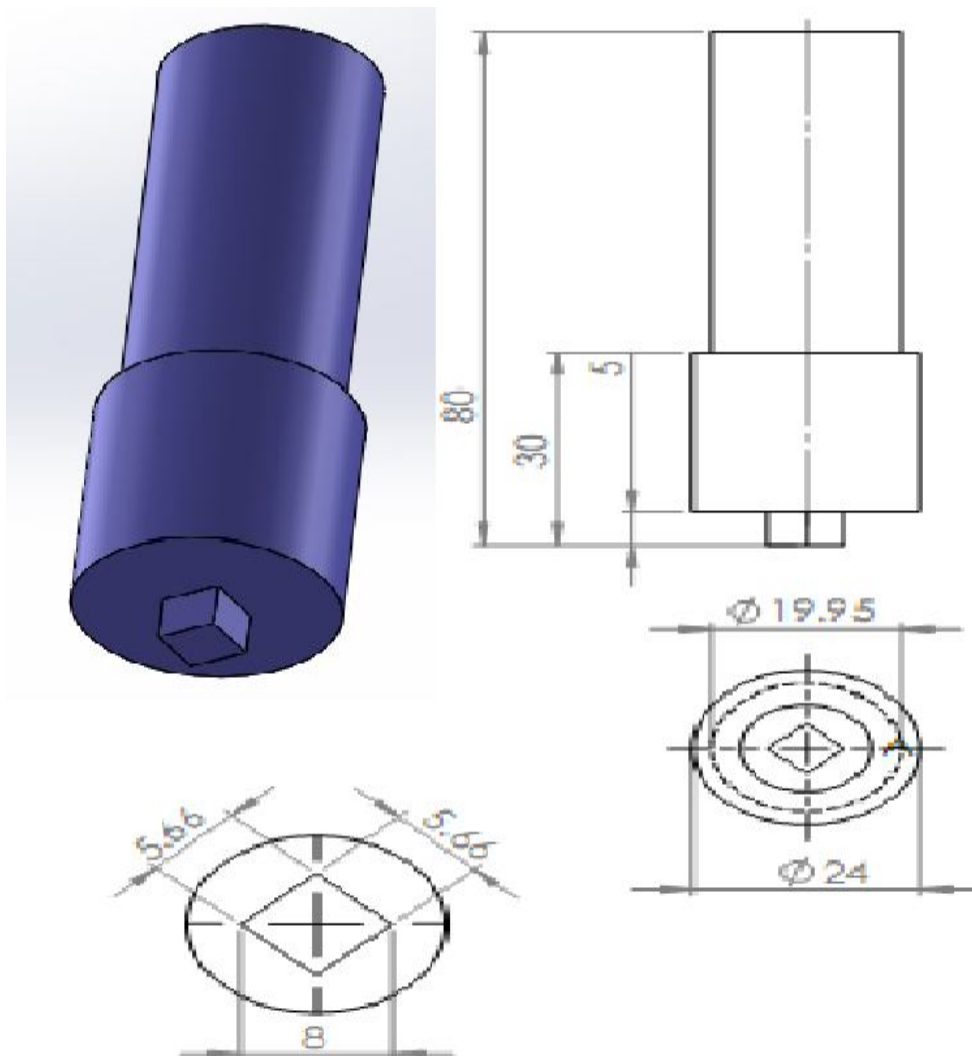


Fig.6. Tool Sketch with dimensions



Fig.7. Actual Picture of the tool

Tool dimensions:

- ❖ Shoulder length 25 mm
- ❖ Shoulder diameter 24 mm
- ❖ Shank diameter 20 mm
- ❖ Shank length 50 mm
- ❖ Pin length 5 mm
- ❖ Pin profile square
- ❖ Pin diagonal 8 mm

### 3.4 Processing parameters

Based on literature review it has been found that processing parameters are key to get good quality of processed region or weld. Improper selection of processing parameters may result in defects like kissing bond, tunnel defect or wormhole etc. It has been found that these parameters depends upon type of work-piece material that is going to be processed and the nature of grains and properties that we want to get. So based on past study and a number trials performed, the final opted processing parameters are given below in Table 5:

Table No. 5

Processing parameters

TOOL ROTATION	TRAVERSE SPEED	NUMBER OF PASS	TILT ANGLE
1200 rpm	50 mm/min	3	0 Deg.

### 3.5 Processing Methodology

- 1) The prepared work-piece plate without groove was fixed with hydraulic clamp on the table of Friction Stir Welding Machine (FSW-4T-HYD, R.V.Machine Tools).
- 2) The H13 tool was inserted and tightened into the spindle.
- 3) The machine was started at speed of 1200 rpm and plunged into the one end of plate.
- 4) The traverse speed of 50 mm/min was given by the table movement.
- 5) After the completion of one pass, the first tool is replaced by second H13 tool of the same dimensions and the second pass was done.
- 6) Similarly third pass was done.
- 7) After completion of three passes, the first plate was unloaded from the table.
- 8) Second plate having grooves of dimension 1x 2 mm and 1 x 4 mm (filled with the ZrB<sub>2</sub> particles) was loaded on the table.
- 9) First, a pin less rotating tool was used to rub over the surface of filled groove to close and compact the particles properly in the groove.
- 10) Now, rest of the above processes were repeated for the processing of second plate also.

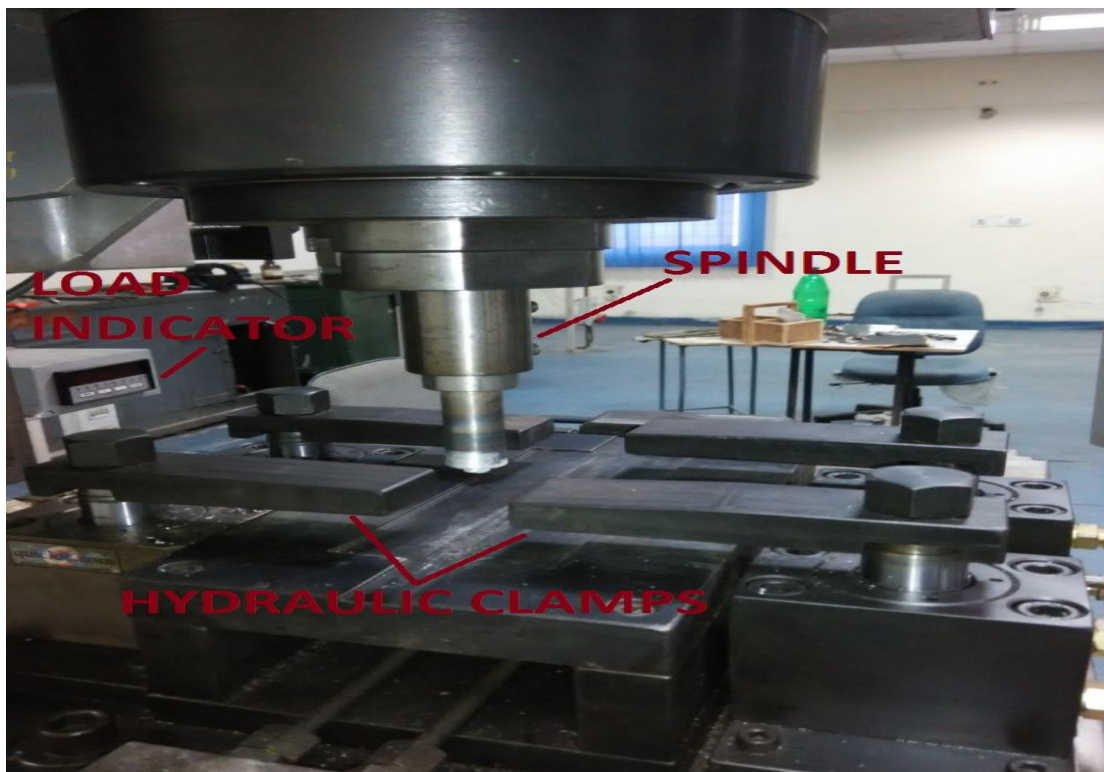


Fig.8. Close view of FSW Machine

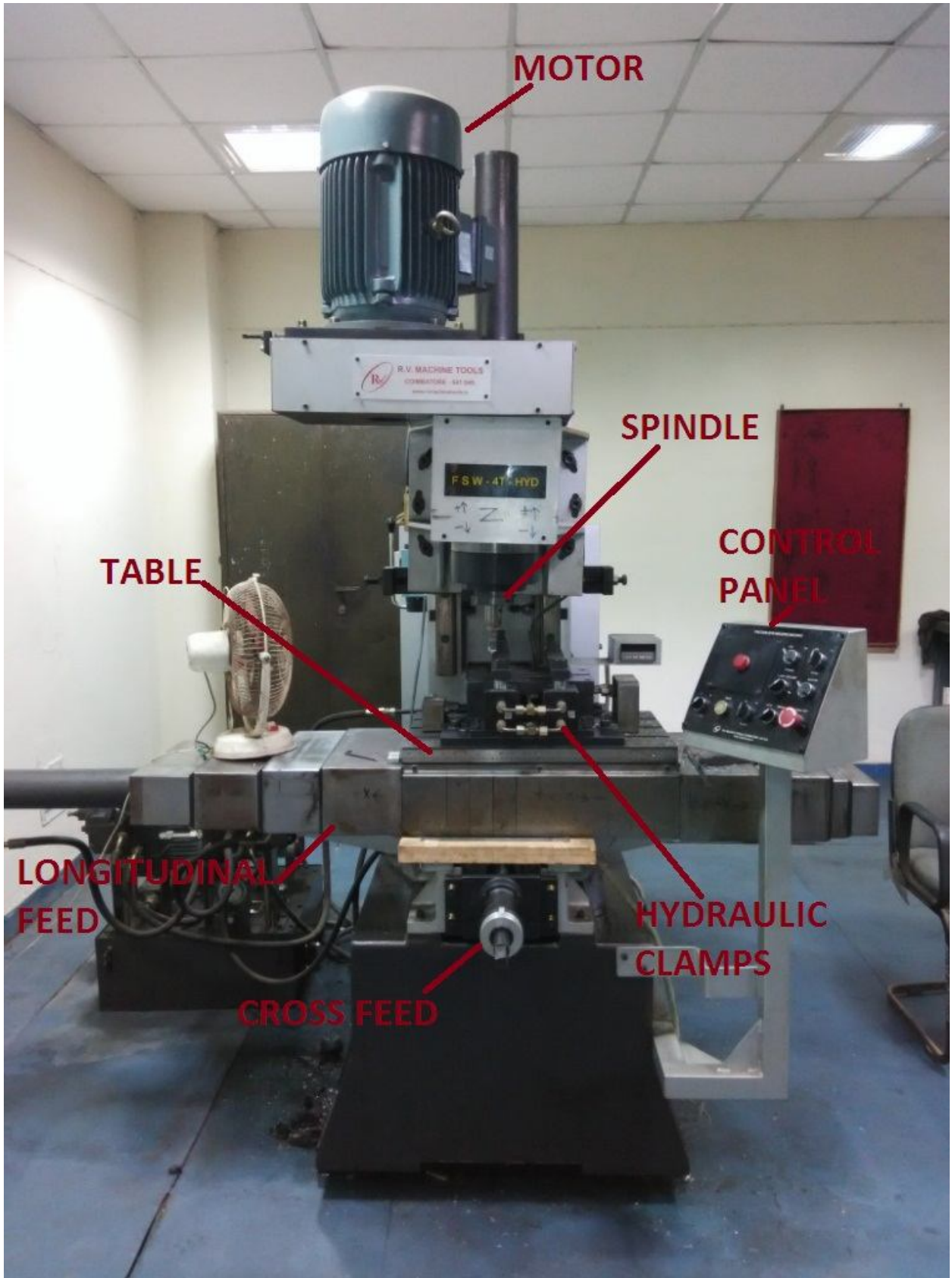


Fig. 9.complete view of FSW Machine





Fig.10. After processing plate (FSPed-Al/ZrB<sub>2</sub> 0 %)



Fig.11. After processing plate (FSPed-Al/ZrB<sub>2</sub> 5 %)



Fig.12. After processing plate (FSPed-Al/ZrB<sub>2</sub> 10 %)

### 3.6 Preparation of Samples for different testing

The samples were cut by Wire-Cut-Electric Discharge Machine from the central region of the processed plates along the longitudinal direction of processing.



Fig.13. After wire cut through EDM-base

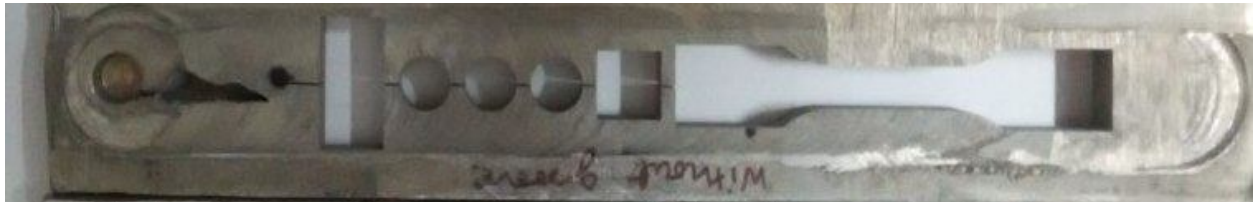


Fig.14. After wire cut through EDM- FSPed-Al/ZrB2 0%



Fig.15. After wire cut through EDM - FSPed-Al/ZrB2 5%



Fig.16. After wire cut through EDM - FSPed-Al/ZrB2 10%

The above figures( Fig.13, Fig. 14, Fig. 15, Fig. 16) shows the plates after cutting of samples for different testings.

Numberings are given to the cut out portions as below:

1-microstructural observation

2, 3 &4 -Wear test

5-Microhardness test

6-Tensile test

### 3.6.1 Sample Preparation for microstructural observation

The samples were cut from the processed region in such a way that it can cover all the regions like heat affected zone (HAZ), thermo mechanically affected zone (TMAZ) and stir zone or nugget (SZ).

#### 3.6.1.1 Making Mold

- ❖ The samples cut by wire electric discharge machine were put in a mold of plastic.
- ❖ The mold was made by mixing a plastic resin with a hardener and the specimen was put in the centre of the mold and left to dry for 3-4 hours.
- ❖ So the samples were prepared in molds to make the handling and polishing easier(Fig.18).

#### 3.6.1.2 Sample Polishing

All the samples were polished by emery papers of different grades in increasing number of grits. Lesser the grit number coarser will be the paper and higher the grit number finer will be the paper. The emery papers used for polishing were coarser to finer at the last (grit number 100, 200, 320, 400, 600, and 1200). At last polishing was done on rotating disc polishing machine having very fine cloth over it(Fig.17). It resulted in mirror like finish.



Fig.17. Polishing Machine



After polishing the surface must be etched by any suitable etchant to reveal the grain structure. Based on literature review, the etchant solution used for Al 7075 was Keller's etchant. It contains 95 ml Distilled Water, 2.5 ml Nitric Acid (HNO<sub>3</sub>), 1.5ml Hydrochloric Acid (HCl), 1 ml Hydrofluoric Acid (HF).The etchant was used on all the samples for the time period of 15-20 seconds. Now the grain structures can be observed through microscope.

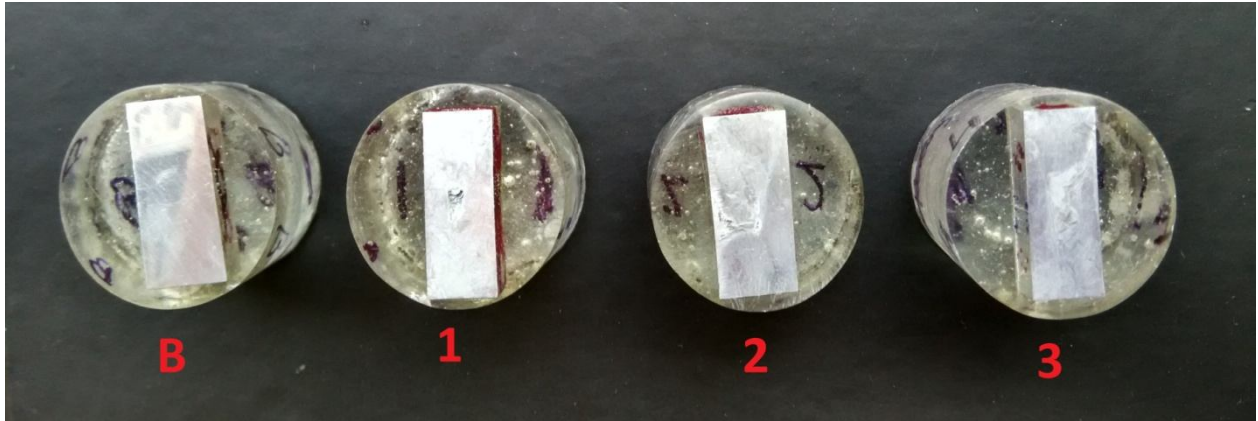


Fig.18.Samples in the mold for microstructural observations (B-base, 1- FSPed-Al/ZrB<sub>2</sub> 0%, 2- FSPed-Al/ZrB<sub>2</sub> 5%, 3-FSPed-Al/ZrB<sub>2</sub> 10%)

### 3.6.1.3 Observations through Optical Microscope

For microstructure observations a optical microscope OLYMPUS MI (Olympus-Opto-Systems India Pvt. Ltd) was used(Fig.19). It was having a software for image analysis and a camera. It was interfaced with the computer to observe and analyze the grain structures and the images at different magnification can be saved easily in the computer storage.



Fig.19. Optical Microscope setup for micro-structural observation



### 3.6.2 Sample Preparation for Microhardness Test

The samples for microhardness were cut in shape of square cross section of side 10 mm by Wire-Cut-Electric Discharge Machine. The samples were fine polished. Then microhardness of the nugget was measured by Vickers Hardness Testing Machine. The load applied was 100 gm for 10 second.

Table No. 6

Specification of Vickers Hardness testing Machine

Make	MITUTOYO
Model	MVK-H1
Hardness range	20-1500 HV
Load range	10 -1000 gm



Fig.20. Vickers Hardness Testing Machine

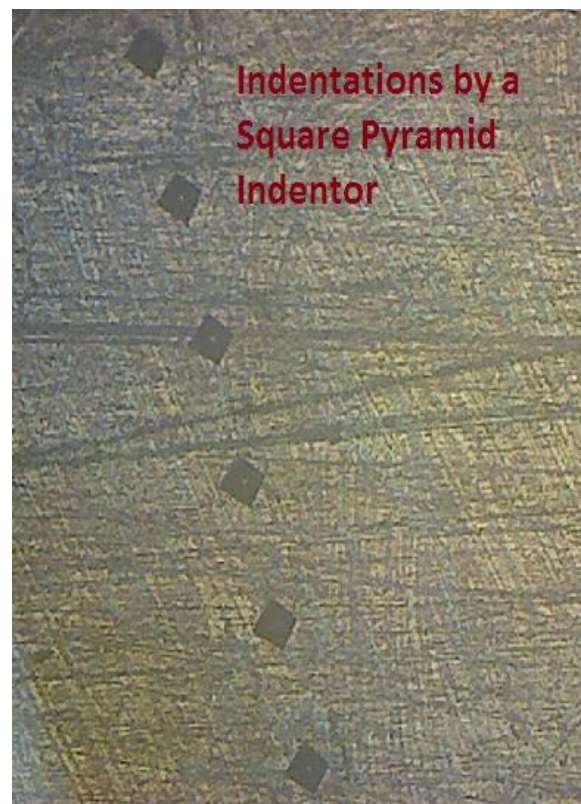


Fig.21. Indentations

### 3.6.3 Sample Preparation for Wear Test

#### 3.6.3.1 Steps for making samples for Wear Test

- ❖ Samples of circular cross section of diameter 8 mm were cut from central region of all the processed plates by Wire-cut Electric Discharge Machine.
- ❖ On the back side of samples (unprocessed region) a hole of 2mm diameter and 4 mm depth was made in each sample by drilling machine.
- ❖ Some stepped pins of mild steel having diameter 8 mm and length 20 -25 mm were made by turning operation on lathe machine.
- ❖ The diameter of the projected portion of the pin was in the range of 1.90-1.98 mm and the length was 4mm.
- ❖ The samples were stuck to the pins by inserting the projected portion of the pin into the hole. In addition, Araldite was used to make the joint stronger.
- ❖ Now the top surface of each sample were made perfectly flat and fine by surface grinding machine.

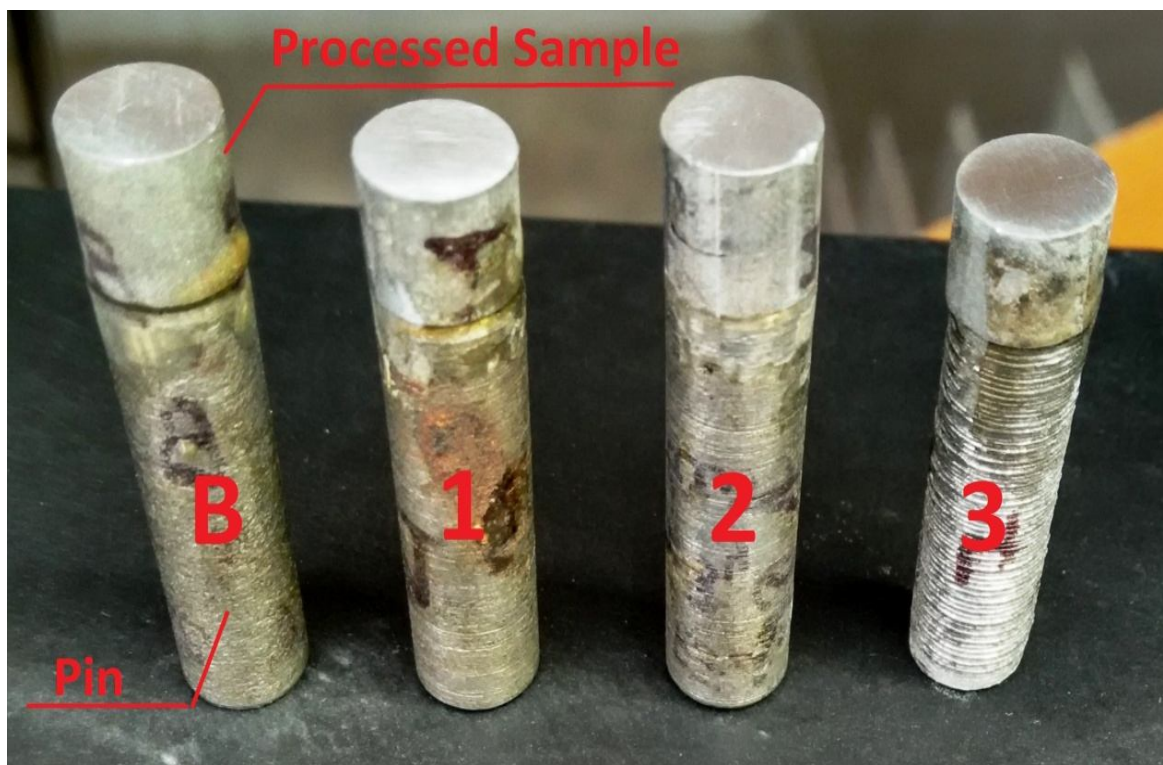


Fig.22. Wear Samples(b-base,1-FSPed-Al/ZrB<sub>2</sub> 0%, 2-FSPed-Al/ZrB<sub>2</sub> 5%, 3-FSPed-Al/ZrB<sub>2</sub> 10%)

### 3.6.3.2 Performing Wear Test

The wear test was done on pin on disc mechanism on the wear testing machine. The model of the machine was TR-20-LE, manufactured by DUCOM , Bangaluru, India.

The specification of the machine is given below:

Table No. 7  
Specification of Wear Testing Machine

Size/diameter of the disc it can hold	165mm
Allowable diameter of the pin/sample	4 to 12mm
Thickness of the disc it can hold	8mm
Disc speed range	200 to 2000 rpm
Frictional force recorder	0 to 200 N
Sliding speed range	0.5 m/s to 10 m/s
Normal load range	1 N to 20 N
Wear track radius	25 mm to 70 mm

#### Steps for wear test:

- ❖ The disc of EN-24 material having hardness about 58HRC and radius of 80 mm was placed on the machine.
- ❖ The disc was tightened properly by screw and Allen-key.
- ❖ The sample's bottom surface and disc surface both are cleaned properly by acetone.
- ❖ The sample was inserted in the jaws of the machine and the height of sample was adjusted so that its flat bottom surface touches the disc surface completely.
- ❖ The track radius where the sample touched the disc was 60mm.
- ❖ The sample was clamped and tightened properly by screws given in the jaws.
- ❖ The sample was loaded by 40 N.
- ❖ Now the machine which is interfaced with computer is started and the disc started rotating at a speed of 1.5 m/s (220 rpm).



- ❖ The load, disc speed and track radius were kept constant for each sample.
- ❖ Now the weight loss ,wear rate and coefficient of friction was recorded after each sliding distance covered at 500 m, 1000m, 1500, 2000m, 2500m and 3000m.
- ❖ The samples were weighted properly after cleaning by acetone at each interval of 500 m.
- ❖ The temperature distribution near the contact surface of pin and disc was also recorded by using a thermal imaging camera.

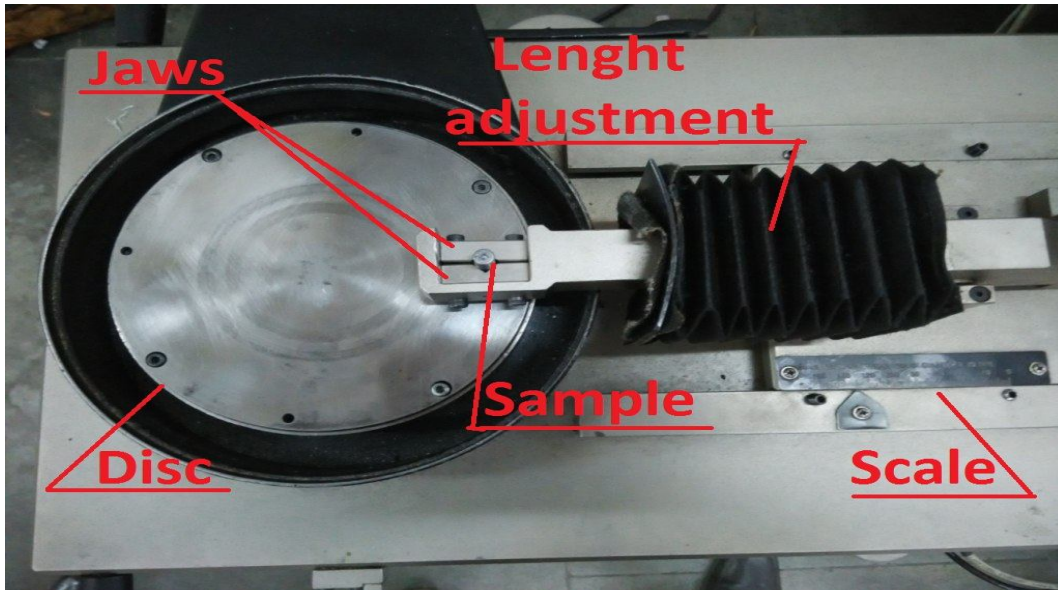


Fig.23. Wear Testing Machine



Fig.24. Computer and Thermal Imaging Camera for data recording

Table 8 shows the parameters taken for wear test:

Table No. 8  
Wear Test Parameters

Disc material	EN-24
Track radius	60 mm
Disc hardness	58 HRC
Disc speed	220 rpm
Sliding velocity	1.5 m/s
Load	40 N
Total distance covered	3000 m

### 3.6.4 Sample preparation for Tensile Test

The samples were cut for tensile test by Wire-cut-Electric discharge machine as per ASTM: E8 sub-standard along the longitudinal direction of the processed region.

The samples were cut in such a way that the gauge length was having only nugget zone .So the tensile strength was tested for the nugget region only.

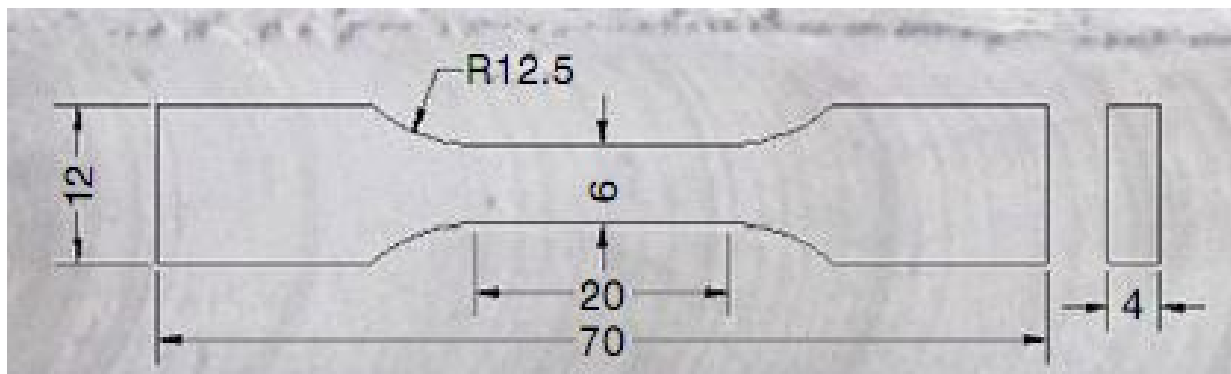


Fig.25. Tensile specimen of substandard size as per ASTM: E8 (dimensions are in mm) [17 ]. The tensile test was carried out on Universal testing machine which was computer controlled at room temperature (25 °C) at a strain rate of 1mm/min. The ultimate strength and elongation was recorded.



Fig.26. Tensile specimens



Fig.27. Computerized Tensile Testing Machine



# CHAPTER 4

## RESULTS AND DISCUSSION

### 4.1 Microstructure Observations



Fig.28. Microstructure of Base material Al7075/T651



Fig.29. Microstructure - FSPed without particles (Al/ZrB2 0%)

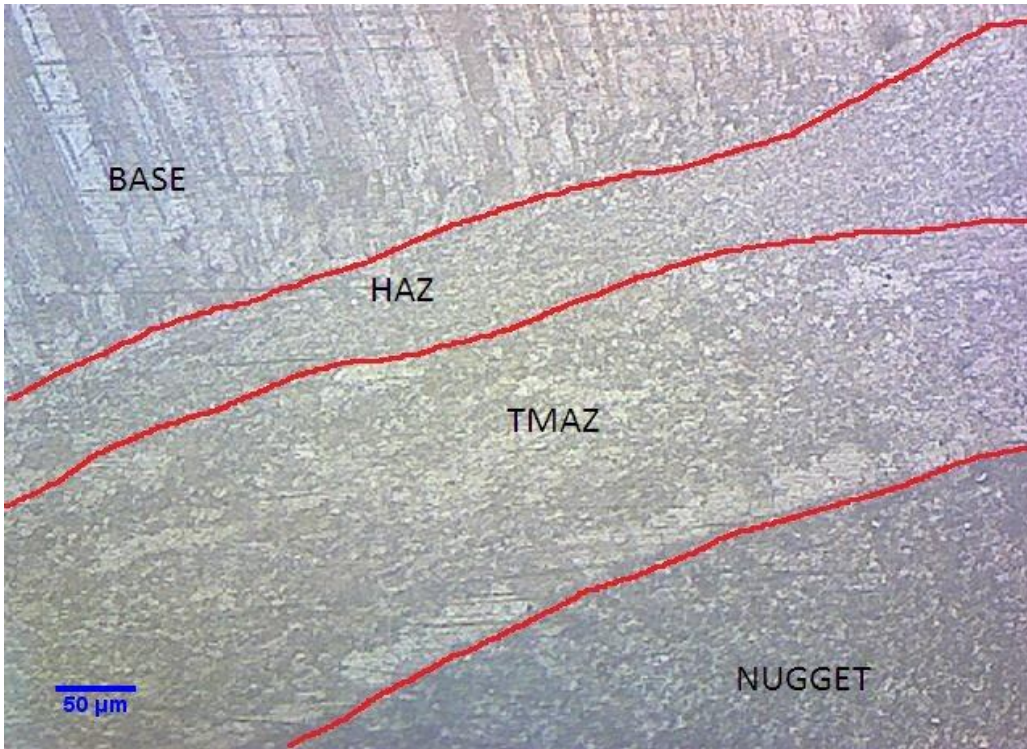


Fig.30. Microstructure - FSPed -(Al/ZrB2 5 %)

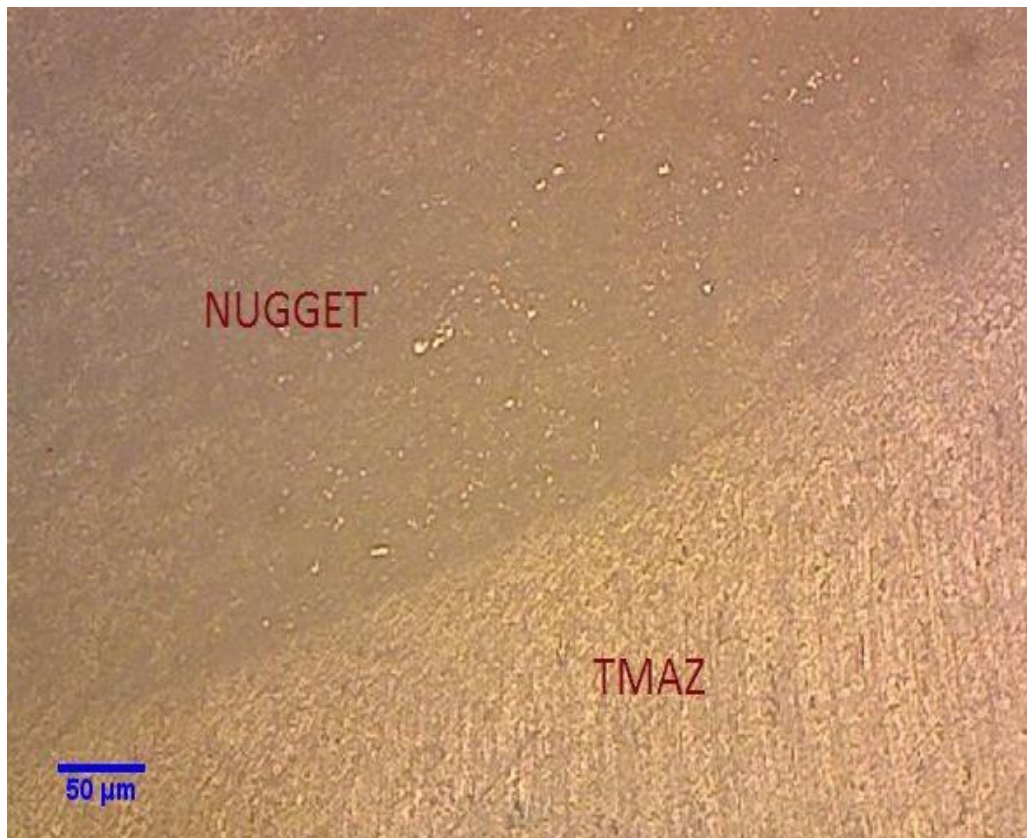


Fig.31. Microstructure -FSPed -(Al/ZrB2 10%)





Fig.32. Microstructure (Nugget Zone) FSPed -(Al/ZrB2 0%)

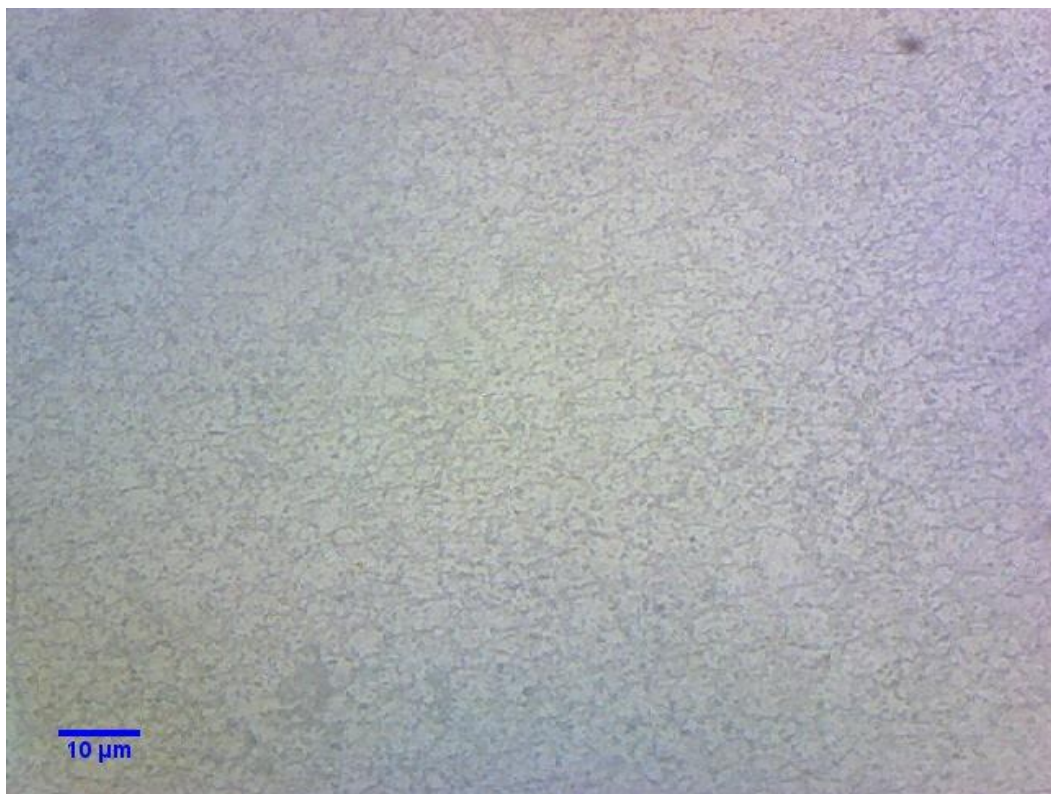


Fig.33. Microstructure (Nugget Zone) FSPed -(Al/ZrB2 0%)



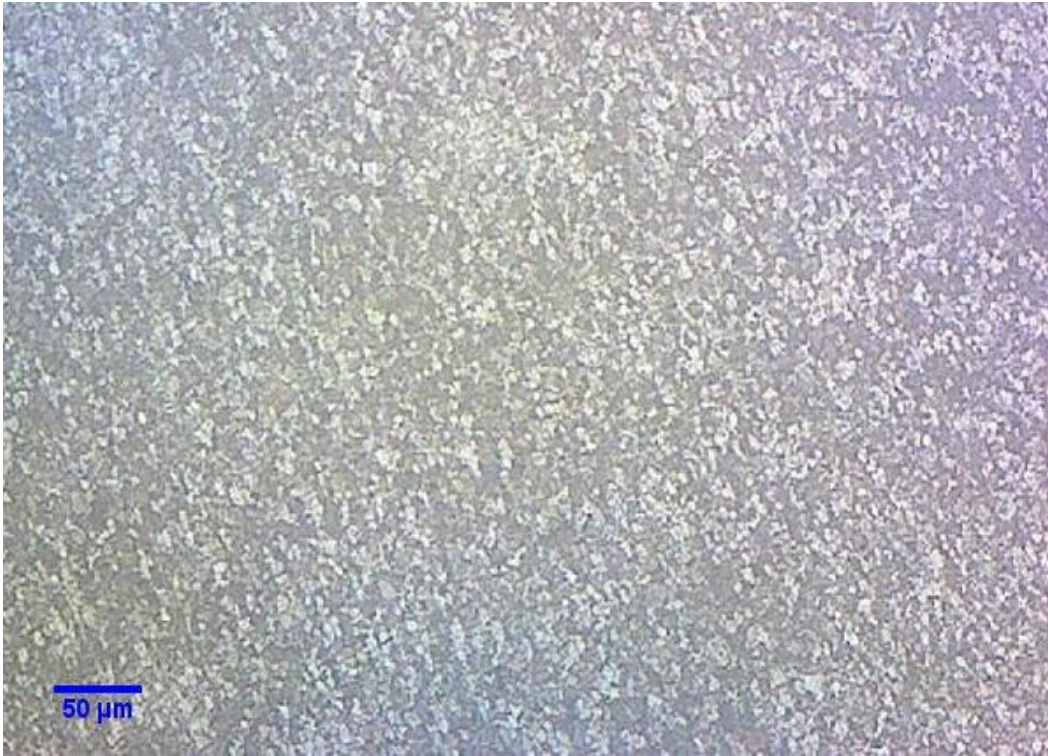


Fig.34. Microstructure (Nugget Zone) FSPed-(Al/ZrB2 5 %)

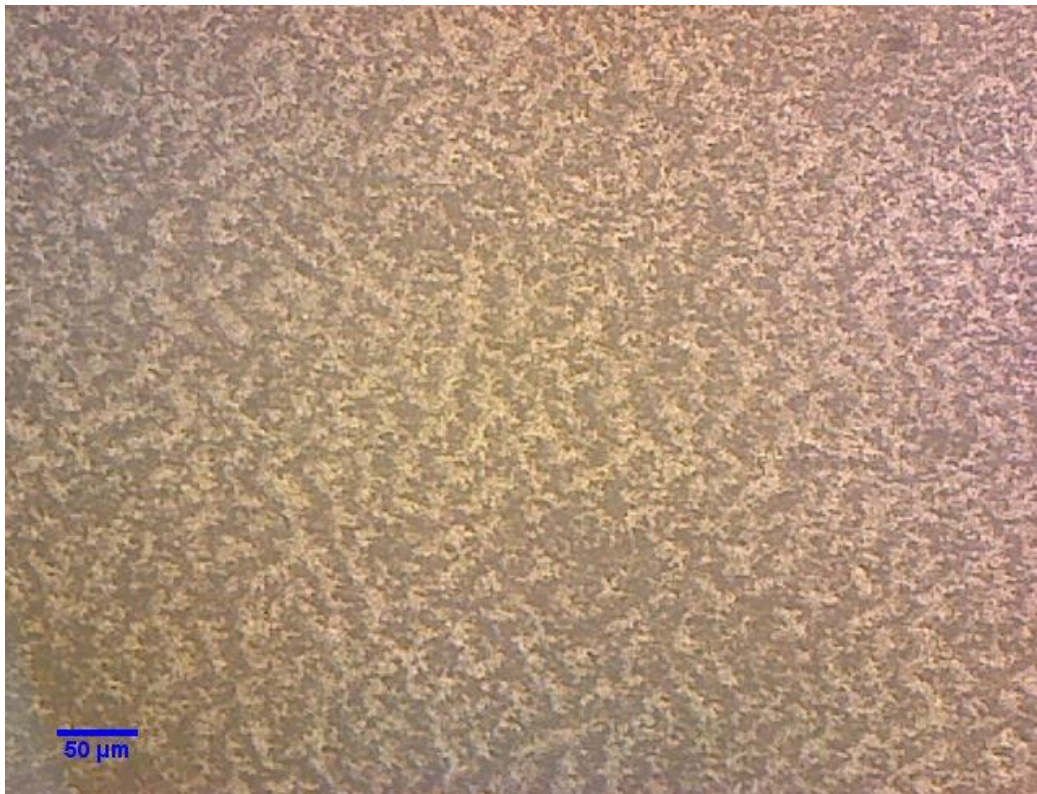


Fig.35. Microstructure (Nugget Zone) FSPed -(Al/ZrB2 10%)

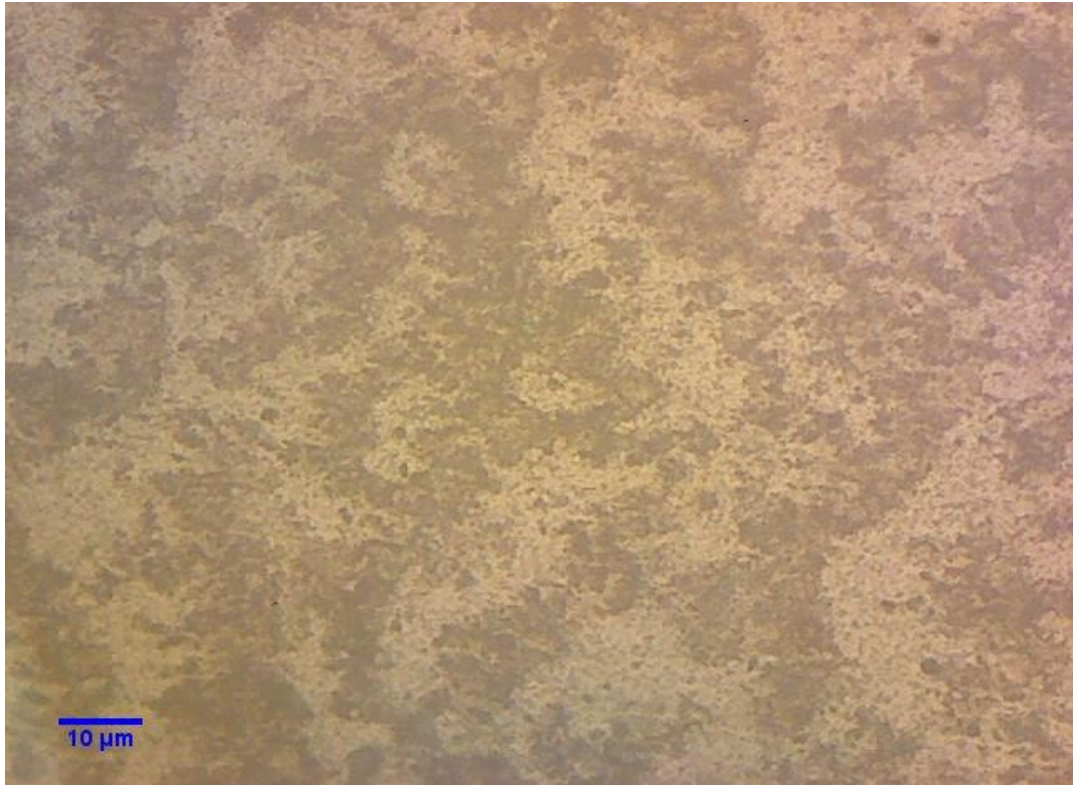


Fig.36.Microstructure(Nugget Zone) FSPed -(Al/ZrB<sub>2</sub> 10%)

The Fig.28 shows the grains of the base material Al 7075 which are elongated in the rolling direction at initial stage. In the Fig.29,30,31 the different zones of processed samples with different density of ZrB<sub>2</sub> particles at 0%,5% and 10% vol. are clearly visible which indicates how the size of grains goes on changing at different zones.Fig.32 and Fig.33 shows the recrystallised fine grains of nugget zone of processed sample without particles at low and high magnification. The refinement and great reduction in grain size can be seen in these figures. In the Fig.34 uniform distribution of particles and grains at nugget zone of the sample processed with ZrB<sub>2</sub> 5% vol. can be seen.Fig.35and 36 shows the little bit clustering of ZrB<sub>2</sub> particles in Aluminium matrix of the sample processed with ZrB<sub>2</sub> 10% vol. But it can be observed that these clustering are very small and are uniformly distributed in the matrix.

## 4.2 Microhardness Observations

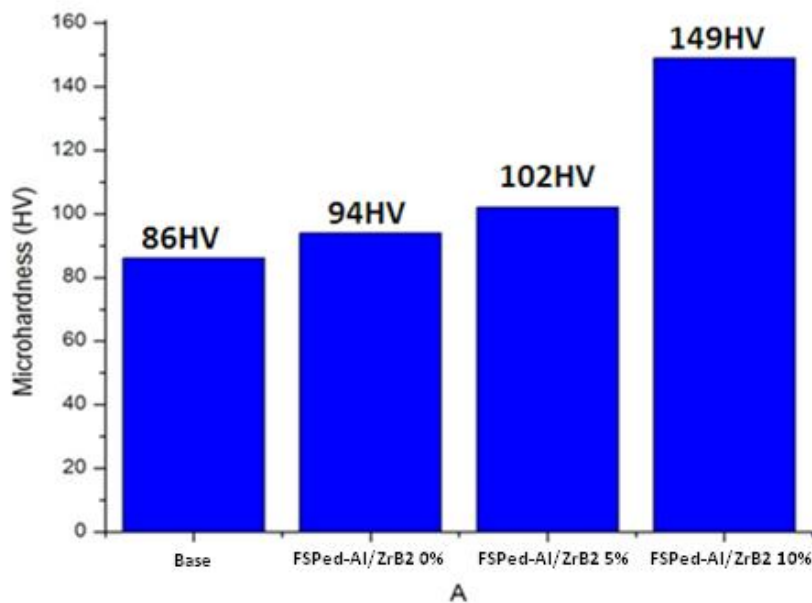


Fig.37. Avg. Microhardness

The base metal is having hardness 86 HV. Fig.37 shows that the hardness is increased after friction stir processing. The processing without particles resulted in increase in hardness upto 94 HV and this is because of grain refinement only according to the Hall Patch relationship:

$$\sigma_y = \sigma_0 + kd^{-1/2}$$

Where the  $\sigma_y$  is the increased strength,  $\sigma_0$  is stress required to move dislocations,  $k$  is material constant and  $d$  is the grain size.

Clearly we can see that the movement of dislocations caused by any external indent or force is inversely proportional to the size of the grain. Addition of ZrB<sub>2</sub> particles further increased the hardness because the dispersion of ZrB<sub>2</sub> particles leads to the further reduction in grain growth and limits the size of grain. And also the hardness of particles partially gets added because the load is shared by particles along with Aluminium matrix. In the Fig.39 we can see that increasing the ZrB<sub>2</sub> by 5% vol. resulted in small increase in hardness but when this particles are doubled upto 10% the hardness is sharply increased upto 149 HV.



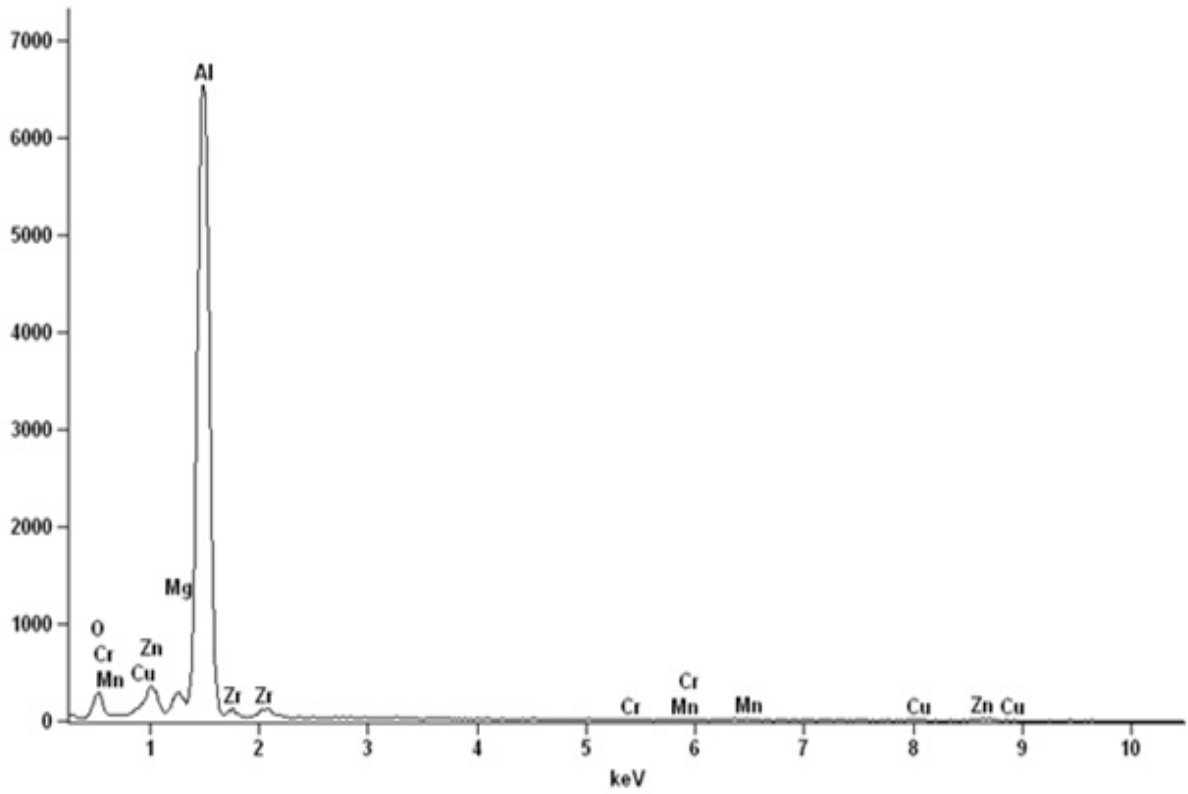


Fig.38. EDS of FSPed-Al/ZrB<sub>2</sub> 5%

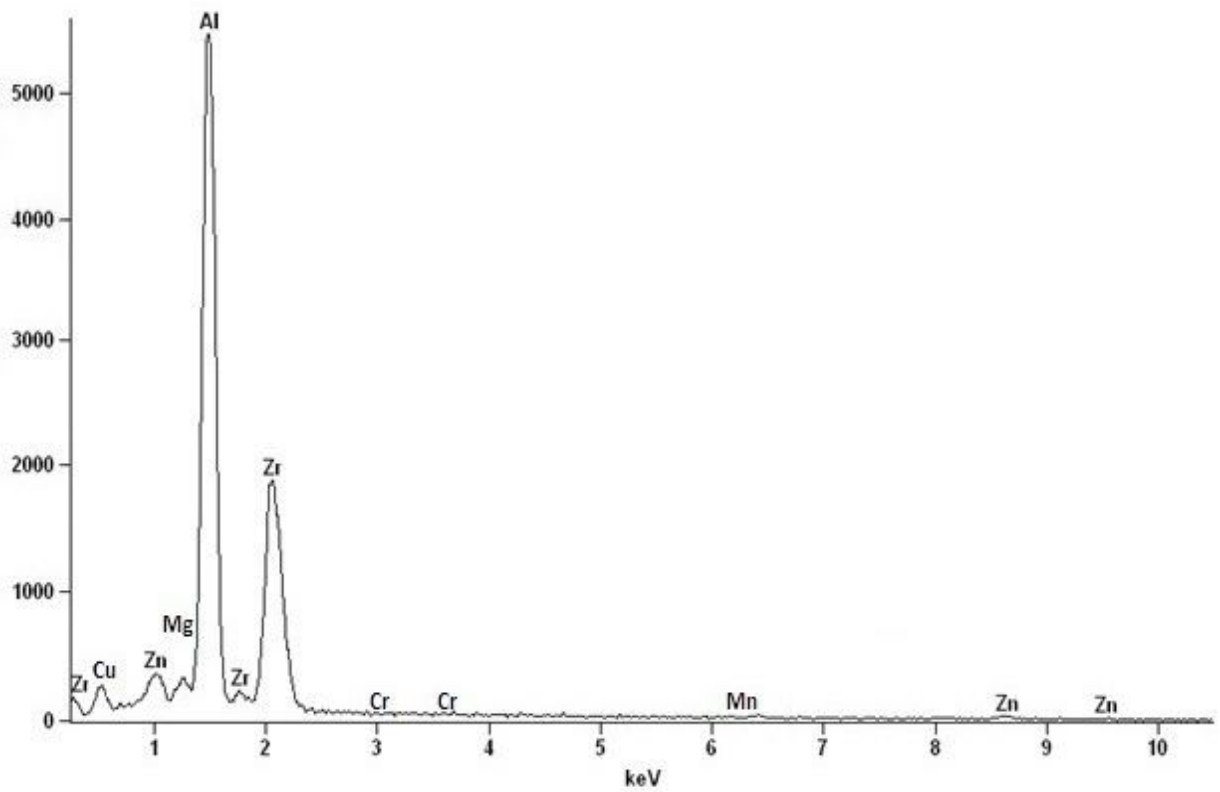


Fig.39. EDS of FSPed-Al/ZrB<sub>2</sub> 10%

EDS of processed samples are shown in the Fig.38 and Fig.39 .The figures clearly shows the presence and distribution of ZrB<sub>2</sub> particles. Also in the EDS figure of FSPed-Al/ZrB<sub>2</sub>-5% the peaks of Zr are somewhat higher than in the Fig.38 which indicates the increase in density of ZrB<sub>2</sub> particles.

The Fig.40 shows the XRD of FSPed-Al/ZrB<sub>2</sub> 10% sample.It was observed that some phases of intermettalic compound of Magnisium-Zinc (MgZn<sub>2</sub>) were present in the matrix of nugget zone.

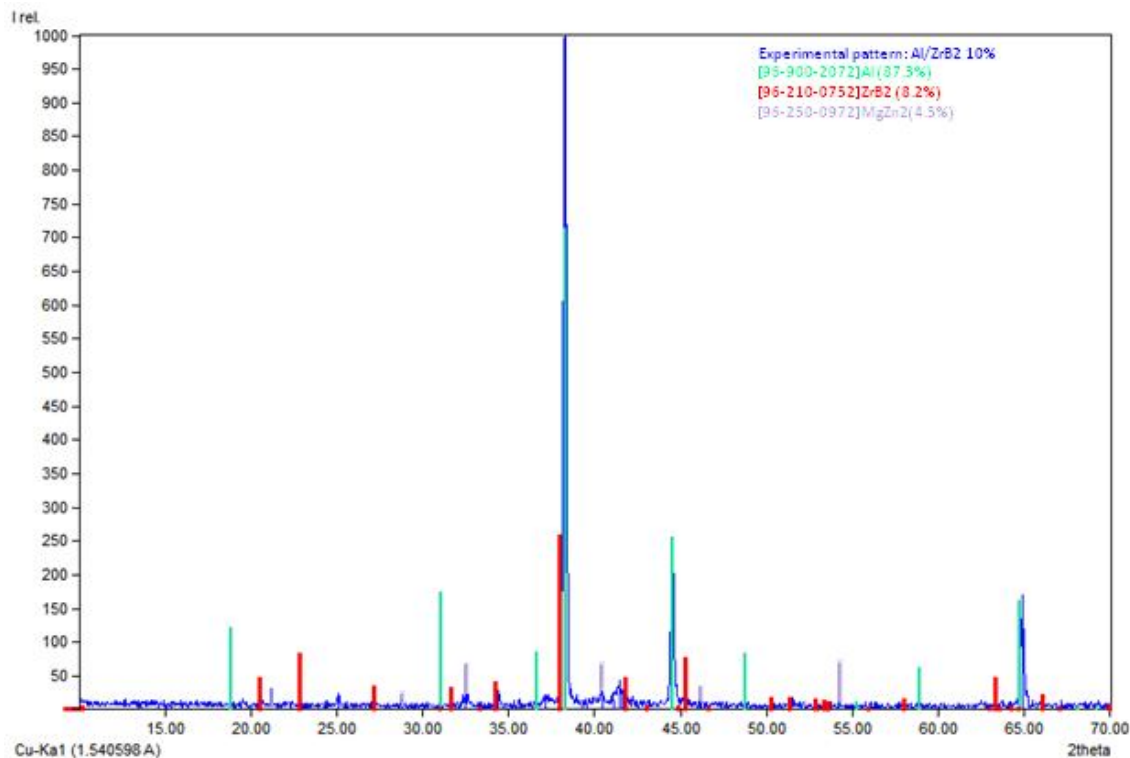


Fig.40. XRD of sample **FSPed Al/ZrB<sub>2</sub>-10%**

As Al 7075 is T651 heat treated and age hardened to form some precipitates of intermettalic compounds of Al, Mn, Cu and Zn. But the presence of Zn and Mg in Al 7075 is more .So formation of MgZn<sub>2</sub> in the matrix dominates over other precipitates. These precipitates causes hardening and strengthening of Al 7075. After Friction Stir Processing presence of MgZn<sub>2</sub> indicates that some precipitates of MgZn<sub>2</sub> did not dissolved or may be formed again. In overall, these precipitates strengthen and harden the Aluminium composite along with ZrB<sub>2</sub> particles.

## SEM of samples showing presence of ZrB<sub>2</sub> Particles

The presence and distribution of ZrB<sub>2</sub> particles can also be seen by Scanning Electron Microscope on the surface of the samples. The Fig.42 and Fig.43 Shows the absence of particles but in the Fig.44 and Fig.45 the particles distribution can be seen.

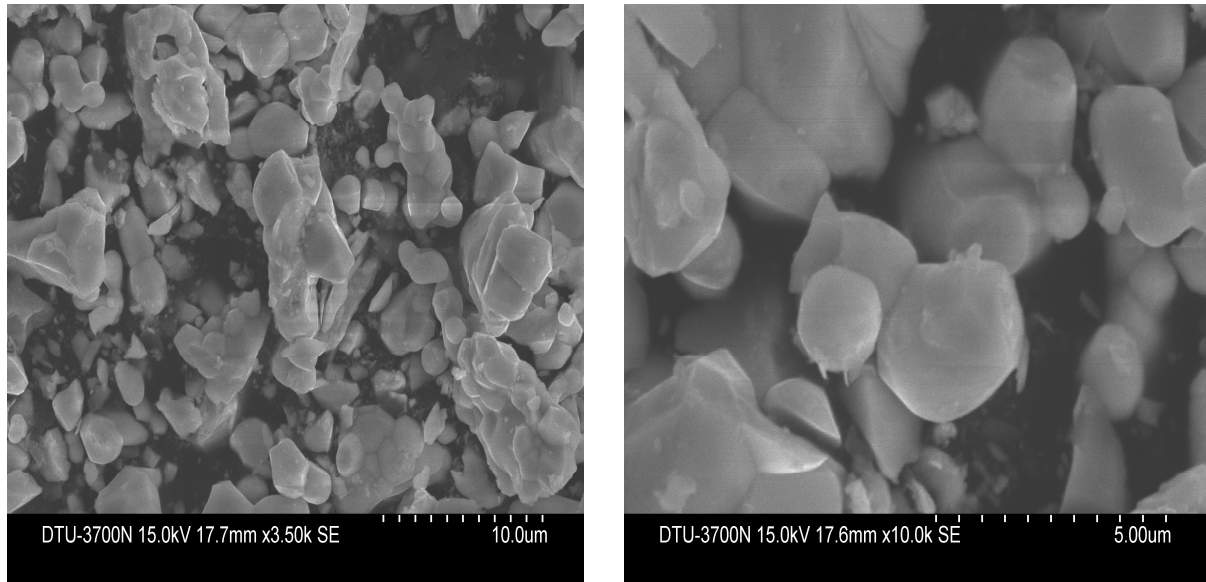


Fig.41. SEM of **ZrB<sub>2</sub>** particles with low and high magnification

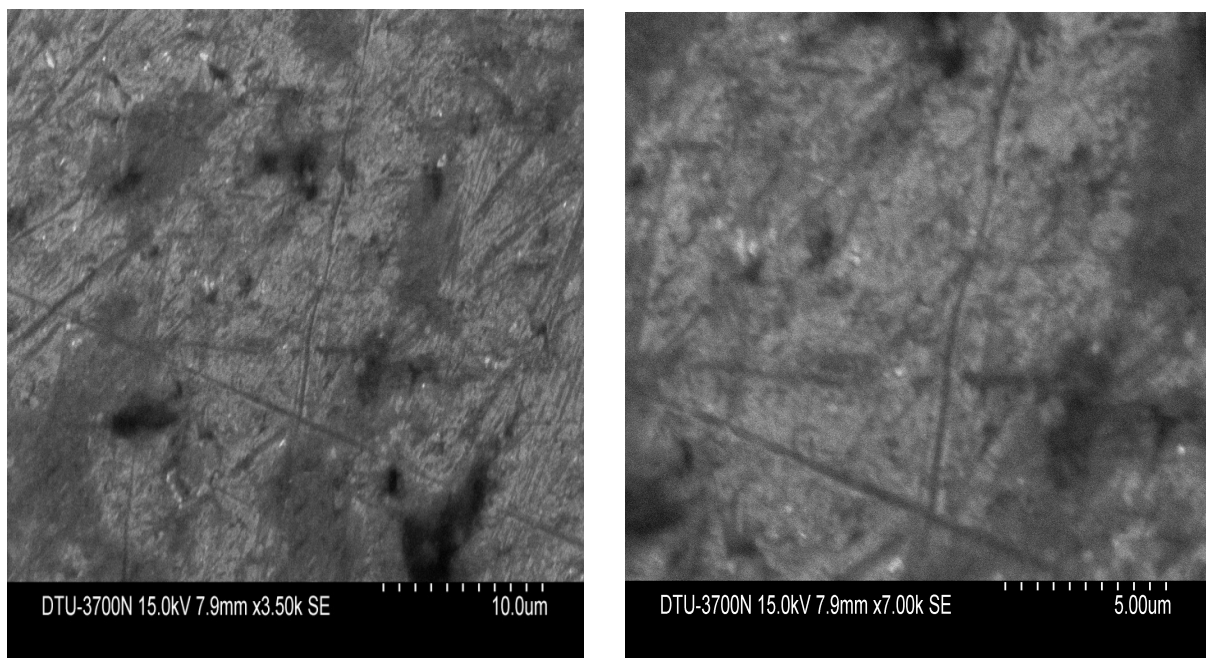


Fig.42. SEM of **Base** with low and high magnification

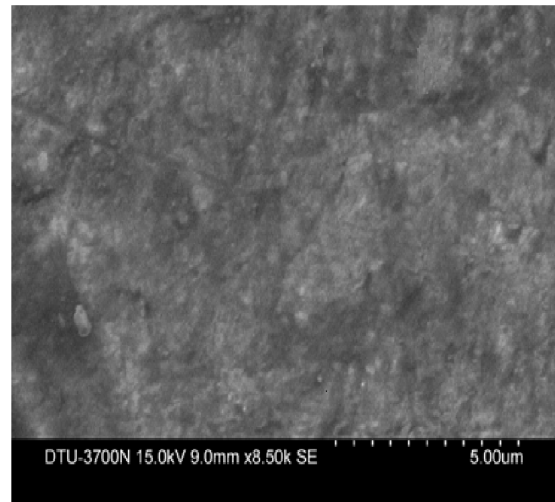
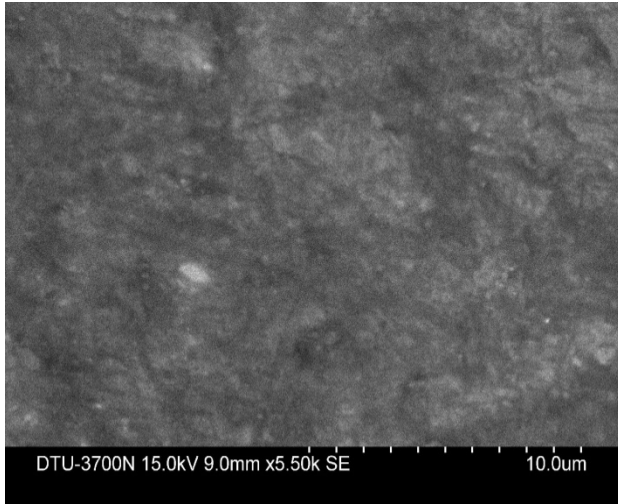


Fig.43 SEM of **FSPed-Al/ZrB2 0%** with low and high magnification

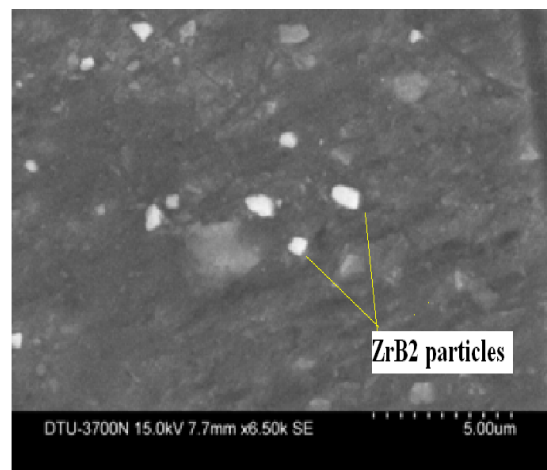
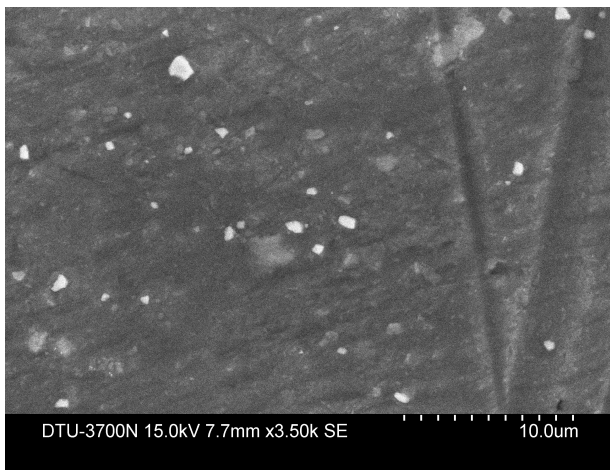


Fig.44 SEM of **FSPed-Al/ZrB2 5%** with low high magnification

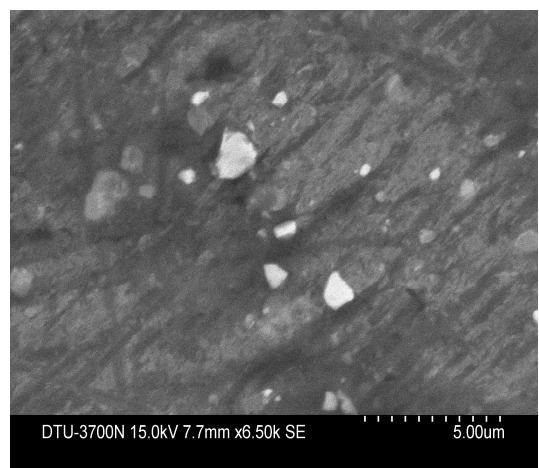
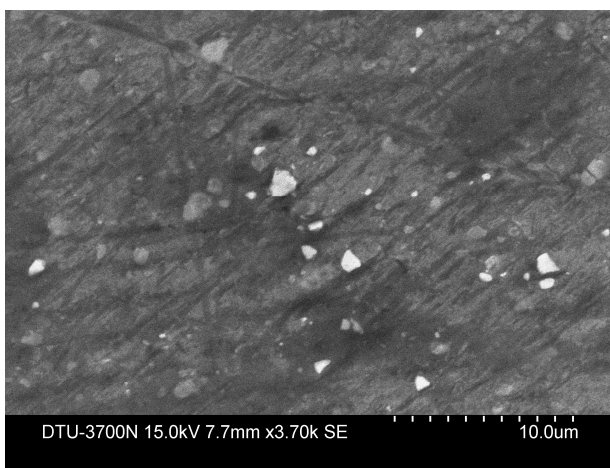


Fig.45 SEM of **FSPed-Al/ZrB2 10%** with low and high magnification



### 4.3 Tensile Test Observations

#### 4.3.1 Ultimate Tensile Strength and Elongation



Fig.46. Tensile test before and after

Table No. 9  
UTS and Elongation

Sample	UTS (MPa)	Elongation (%)
Base	570	13
FSPed-Al/ZrB <sub>2</sub> 0%	582	17
FSPed-Al/ZrB <sub>2</sub> 5%	620	11
FSPed-Al/ZrB <sub>2</sub> 10%	628	6

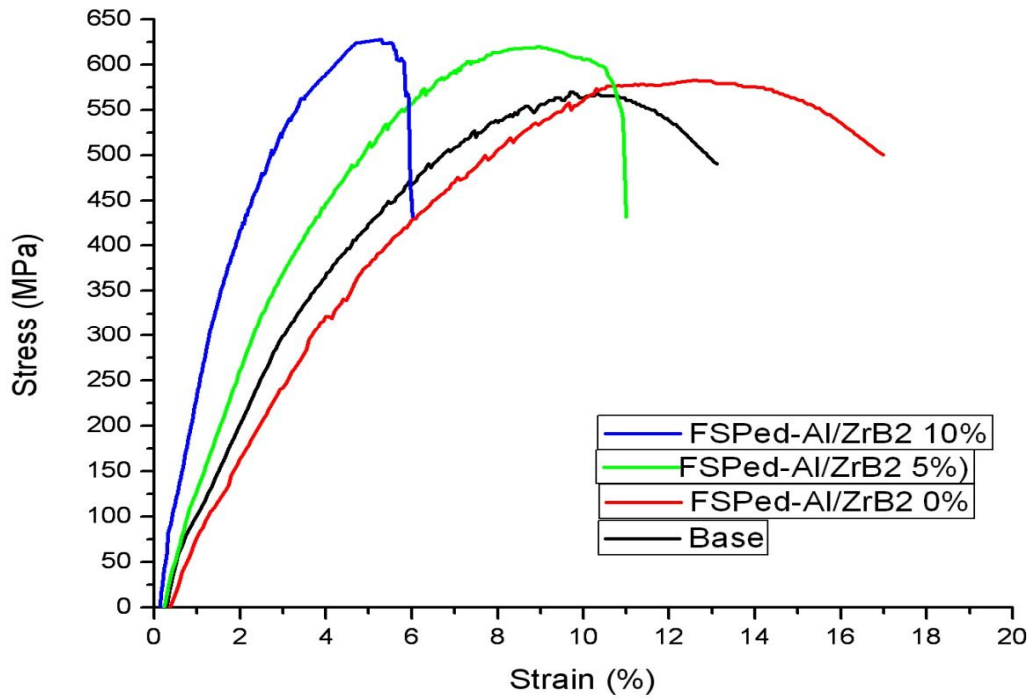


Fig.47. Stress-Strain curves of samples

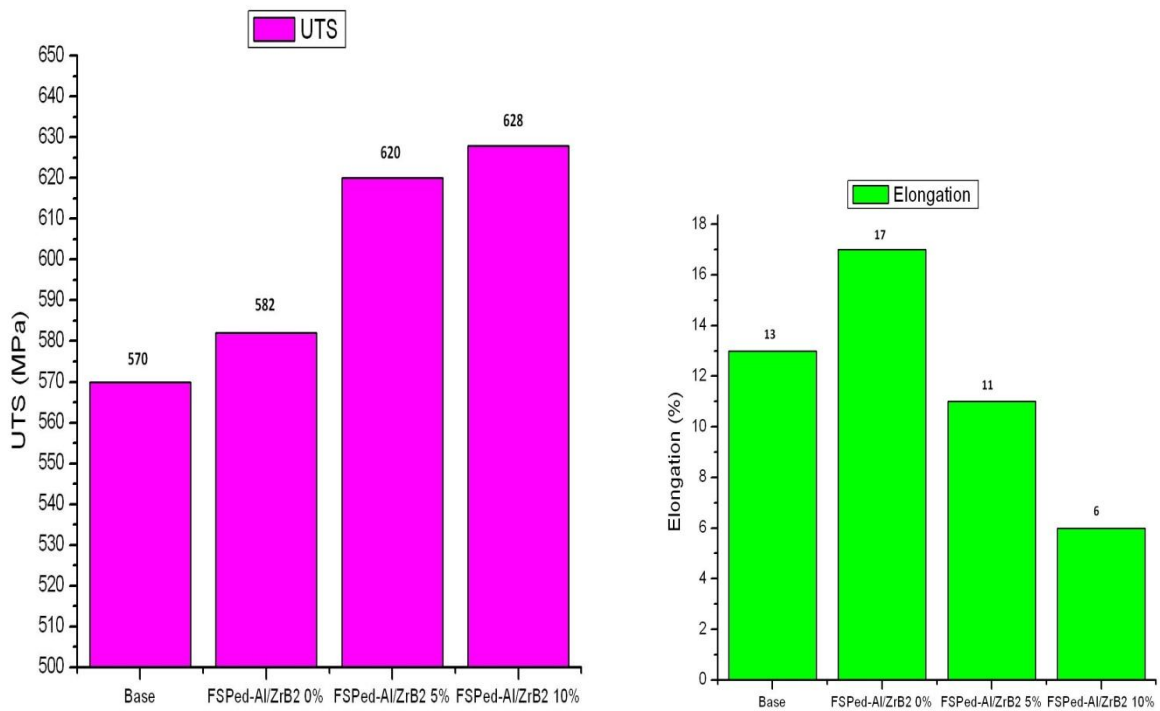


Fig.48.UTS and Elongation

Tensile properties of base Al7075 and composite with ZrB<sub>2</sub> were evaluated at room temperature. It was observed that there is improvement in UTS after processing. Stress-Strain diagram is given in the Fig.47 which also indicates the improvement in yield strength. Processing without particle resulted in increase in both UTS and elongation but after processing with ZrB<sub>2</sub> 5% vol. resulted in sharp increase in UTS. Further increase in particles (ZrB<sub>2</sub> 10% vol.), a small increase in UTS was observed but it resulted in great reduction in elongation (only 6%).

The increase in UTS can be attributed to grain refinement, insertion of ZrB<sub>2</sub>, difference between coefficient of thermal expansion of Al7075 and ZrB<sub>2</sub>. The strength of any material is directly proportional to the surface area of grains and length of grain boundaries. When big size grains are refined to small sized grains, resultant surface area and grain boundary length is increased. This results in more resistance to flow of cracks.

The ZrB<sub>2</sub> particles also reduce the grain growth and limit the size which causes further refinement of grains. Also the applied load is shared by the particles and reduces the load on soft matrix. The ZrB<sub>2</sub> particles also act as barriers to the flow of dislocation and cracks.

The coefficient of thermal expansion of ZrB<sub>2</sub> is  $6.8 \times 10^{-6}/^{\circ}\text{C}$  and that of Al7075 is  $23 \times 10^{-6}/^{\circ}\text{C}$ . This big difference results in development of strain fields during cooling around the particles and in the matrix. These strain fields cause further increase in dislocation density. These dislocations are randomly oriented in the matrix and oppose the sliding and flow of each other.

The reduction in elongation is caused by increase in ZrB<sub>2</sub> particles. These particles resist the movement of dislocations and hence plastic flow is decreased.

More addition of ZrB<sub>2</sub> particles may lead to reduction of matrix volume. The formation of clustering will be there. These clusterings weaken the material as the bond between them is very weak. Also due to less volume of matrix the bond between particle and aluminum decreases. This causes reduction in strength. We can see in the Fig.48, the UTS of Al/ ZrB<sub>2</sub> 10% is not so much improved as compared to Al/ ZrB<sub>2</sub> 5% but great reduction in elongation. This may be due to the clustering and formation of weak bonds. This indicates that further increase in ZrB<sub>2</sub> particles may lead to reduction in both UTS and elongation. It was observed that Al/ ZrB<sub>2</sub> 5% have increase in UTS without much loss in ductility.

### 4.3.2 Fractography of Tensile Tested Samples

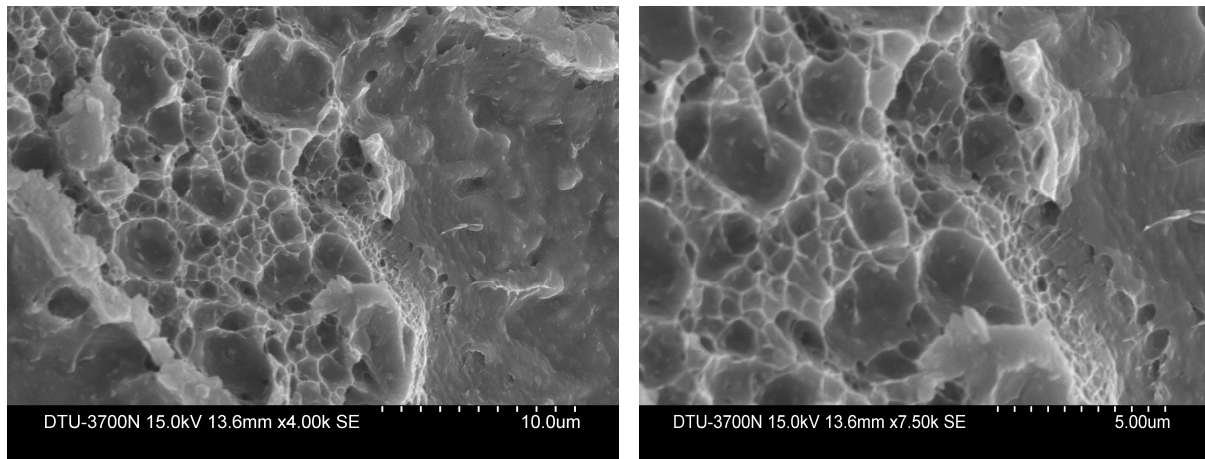


Fig.49. SEM of tensile sample after fracture **base** at low and high magnification

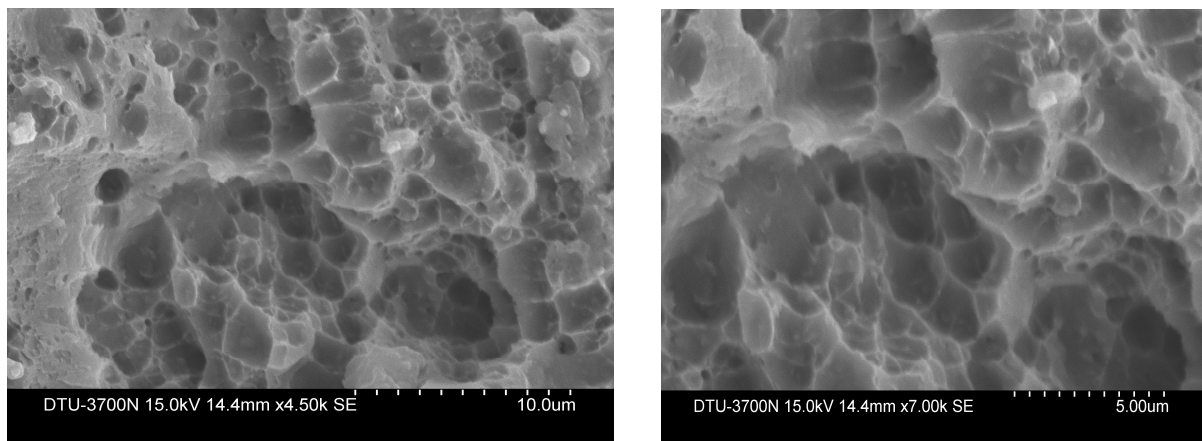


Fig.50. SEM of tensile sample after fracture **FSPed-Al/ZrB2 0%** at low and high magnification

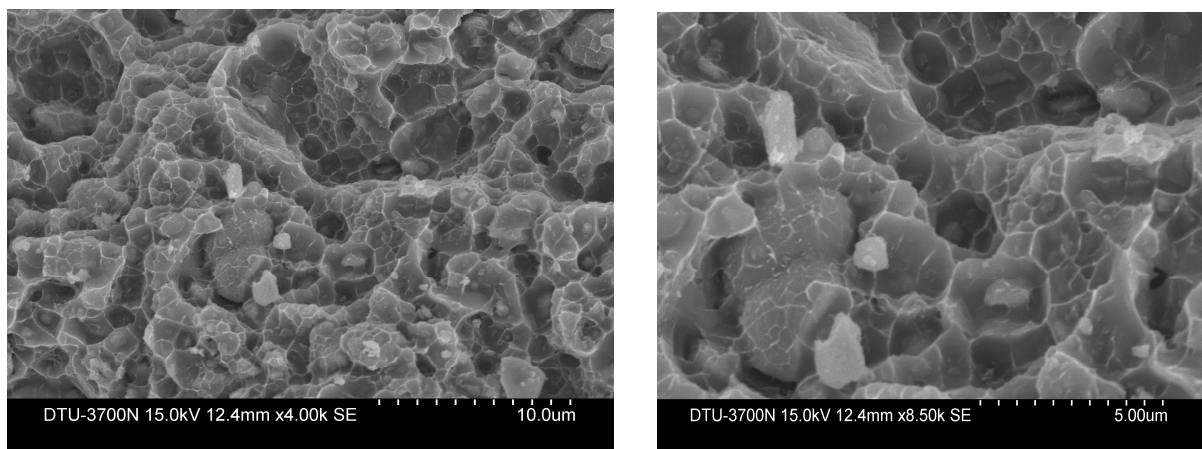


Fig.51. SEM of tensile sample after fracture **FSPed-Al/ZrB2 5%** at low and high magnification



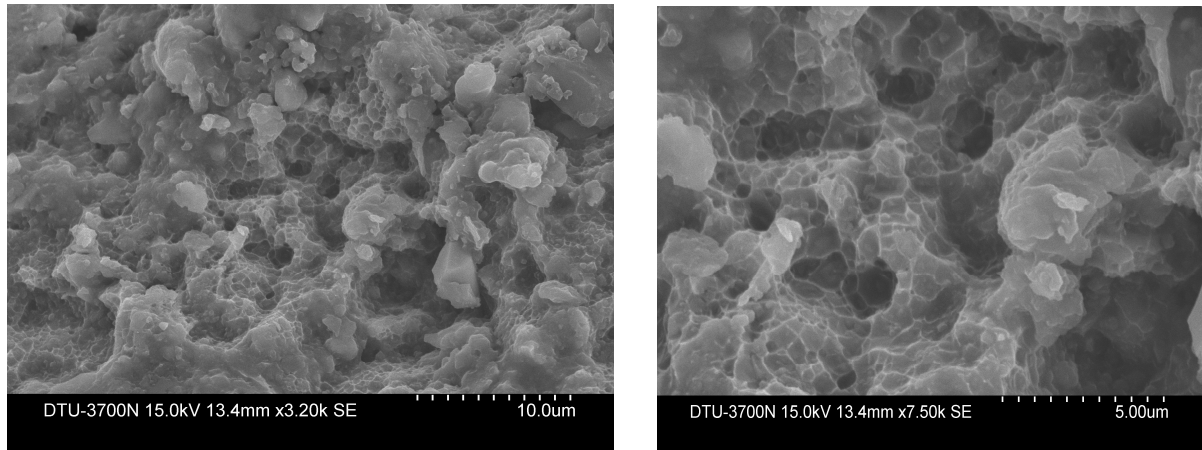


Fig.52.SEM of tensile sample after fracture **FSPed-Al/ZrB2 10%** at low and high magnification

Large and deep dimple size indicates that the material has undergone large amount of plastic deformation before breaking or failure. In the Fig.49 of base material, the deep and big dimples can be seen. In the Fig.50 the uniform and deeper dimples can be seen which indicates increase in elongation after processing. In Fig.51 , after addition of ZrB2 particles to 5% vol. still there are dimples of comparable size to that of base material which indicates very less loss of ductility. In Fig.52, the dimples are very less and almost flat which indicates brittle failure of composite. Some clusters of ZrB2 are also visible in Fig.52. The particles reduces plastic deformation which leads to reduction in dimple depth and the mechanism of failure of the material goes on changing from ductile to brittle.

#### 4.4 Wear Test Results

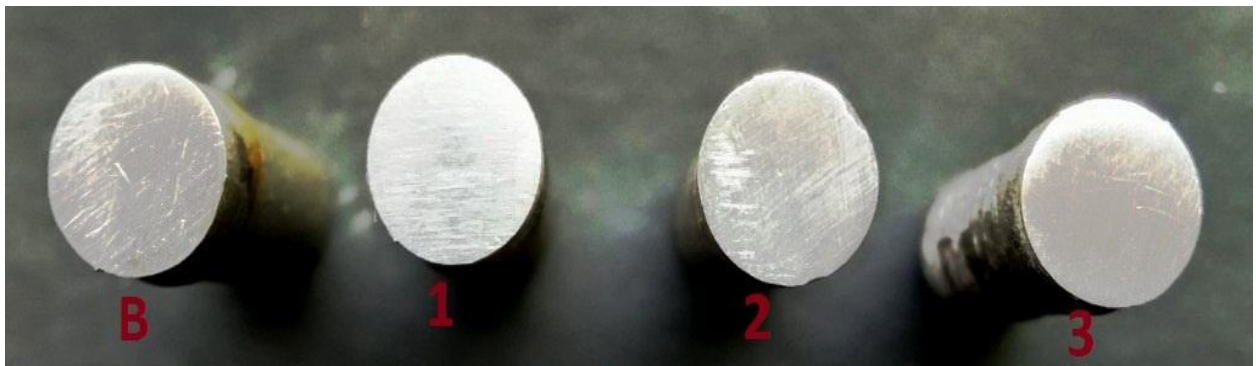


Fig.53. Samples before wear test

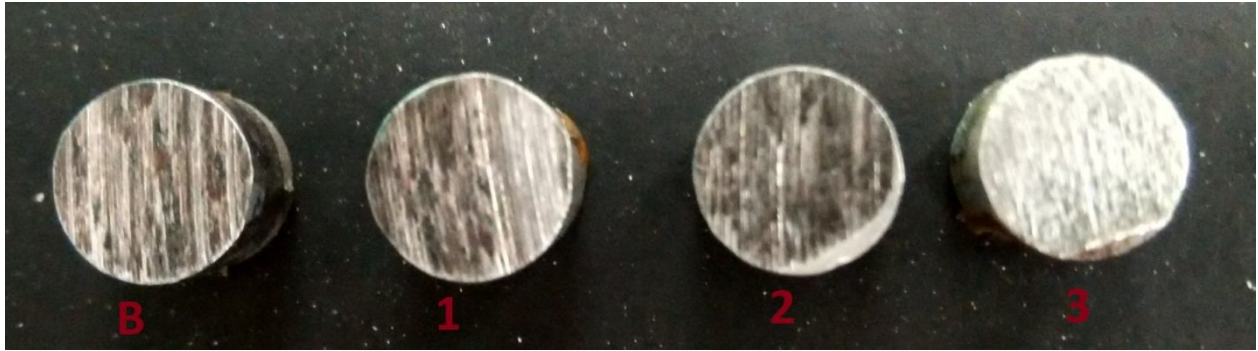


Fig.54. Samples after wear test

#### 4.4.1 Weight loss and Wear rate

Table No. 10

Weight loss and wear rate at each interval of 500 m (base material)

Sliding Distance (m)	Wt. loss (mg) (cumulative)	Wear Rate (mg/m) X10 <sup>-3</sup>
500	3.1985	6.2397
1000	6.2904	6.2904
1500	9.4980	6.3320
2000	12.7496	6.3748
2500	16.0717	6.4287
3000	19.3824	6.4608
<b>Coefficient Of Friction</b>	<b>0.62</b>	<b>Avg. Wear rate 6.3544</b>

Table No. 11

Weight loss and wear rate at each interval of 500 m (FSPed-Al/ZrB<sub>2</sub> 0%)

Sliding Distance (m)	Wt. loss (mg) (cumulative)	Wear Rate (mg/m) X10 <sup>-3</sup>
500	2.6822	5.3644
1000	5.3700	5.3700
1500	8.0860	5.3907
2000	10.8002	5.4001
2500	13.4860	5.3944
3000	16.2831	5.4277
<b>Coefficient Of Friction</b>	<b>0.58</b>	<b>Avg. Wear rate 5.391</b>

Table No. 12

Weight loss and wear rate at each interval of 500 m (FSPed-Al/ZrB<sub>2</sub> 5%)

Sliding Distance (m)	Wt. loss (mg) (cumulative)	Wear Rate (mg/m) X10 <sup>-3</sup>
500	1.8252	3.6505
1000	3.6621	3.6621
1500	5.5213	3.6809
2000	7.3796	3.6898
2500	9.9261	3.7045
3000	11.4063	3.8021
<b>Coefficient Of Friction</b>	<b>0.51</b>	<b>Avg. Wear rate 3.6983</b>

Table No. 13

Weight loss and wear rate at each interval of 500 m (FSPed-Al/ZrB<sub>2</sub> 10%)

Sliding Distance (m)	Wt. loss (mg) (cumulative)	Wear Rate (mg/m) X10 <sup>-3</sup>
500	0.6701	1.3402
1000	1.3540	1.3540
1500	2.0611	1.3741
2000	2.7310	1.3655
2500	3.4512	1.3805
3000	4.1703	<b>1.3901</b>
<b>Coefficient Of Friction</b>	<b>0.43</b>	<b>Avg. Wear rate 1.3674</b>

The weight loss increased as the sliding distance is increased because of more rubbing. The continuous rubbing resulted in increase in temperature which lowers the bonding between the particles and the matrix. Increasing the ZrB<sub>2</sub> reduces the wear because it is highly refractory material. It can bear excess heat generated during rubbing. It is hard than Al matrix and minimizes the contact area of the Al matrix to the counter mating surface. It directly transfers the acting load to the counter mating surface thereby saving the Al matrix .Fig.55 and Fig .56 shows the insertion of particles greatly reduces the weight loss and wear rate.

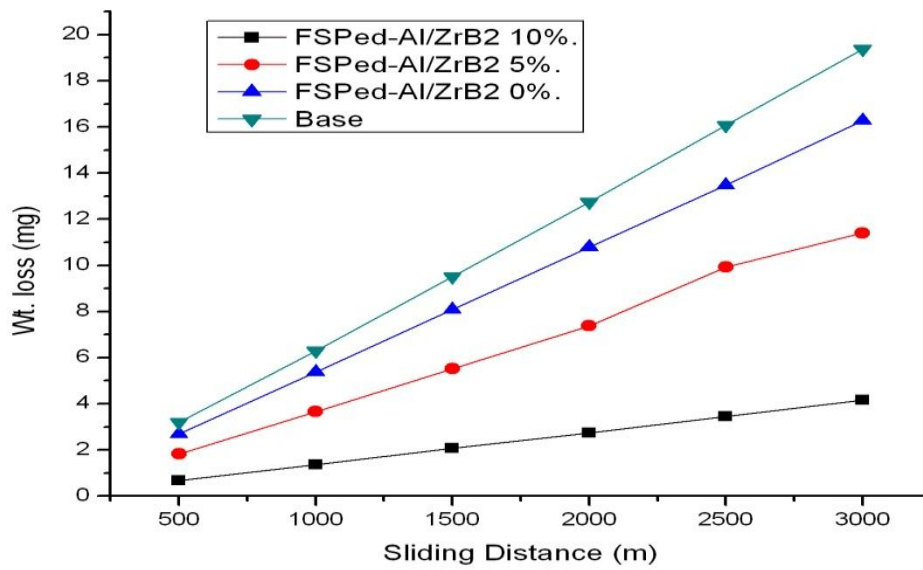


Fig.55. Weight Loss v/s Sliding Distance

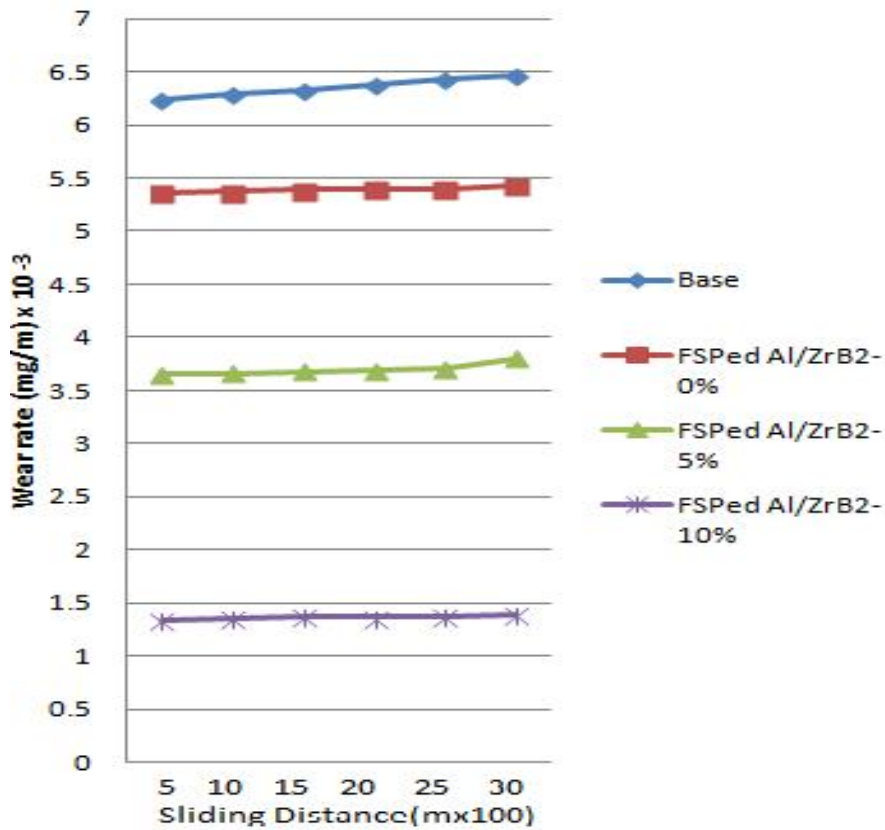


Fig.56. Wear Rate v/s Sliding Distance



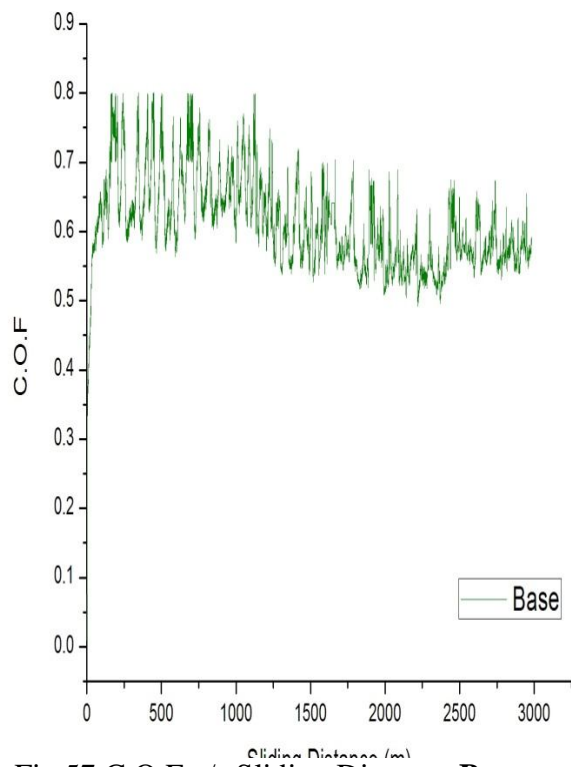


Fig.57.C.O.F v/s Sliding Distance-Base

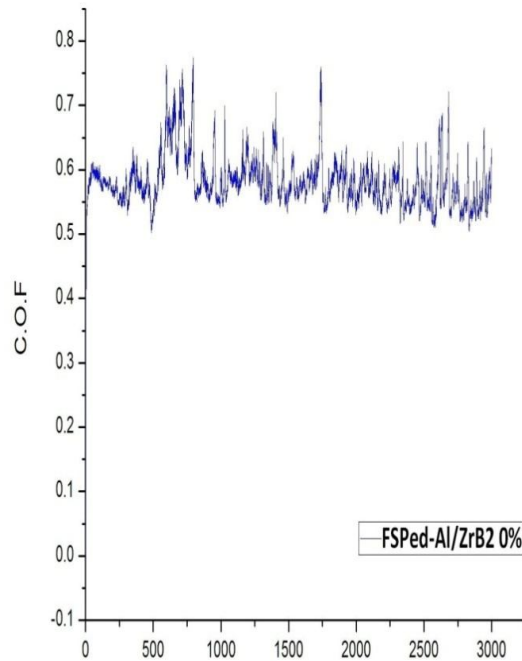


Fig.58.C.O.F v/s Sliding Distance FSPed-Al/ZrB2 0%

0%

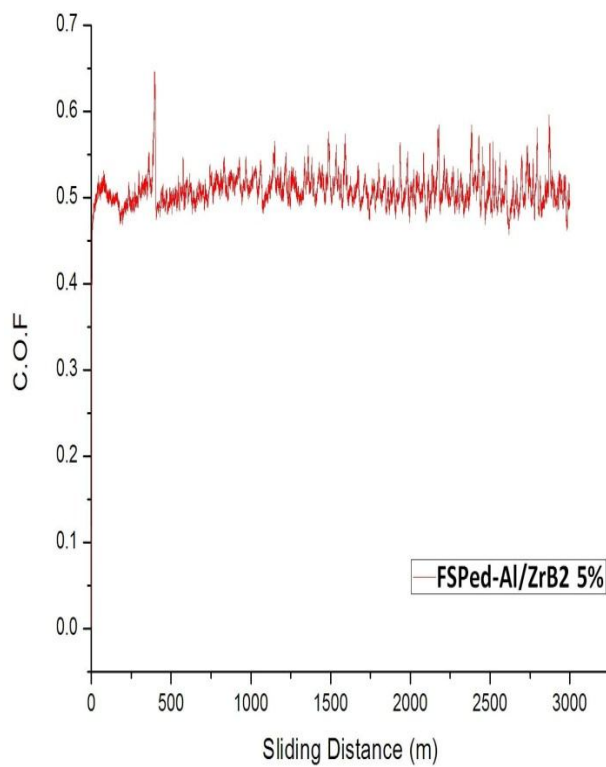


Fig.59. C.O.F v/s Sliding Distance- FSPed-Al/ZrB2 5%

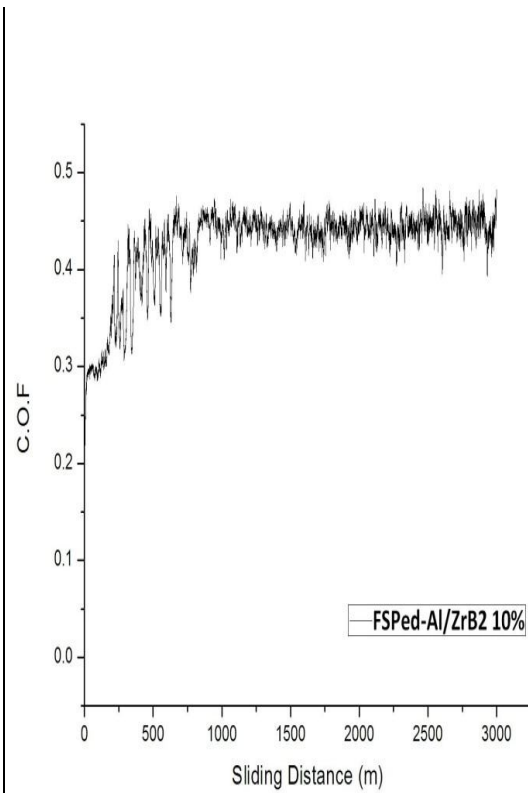


Fig.60.C.O.F v/s Sliding Distance FSPed-Al/ZrB2 10%

10%

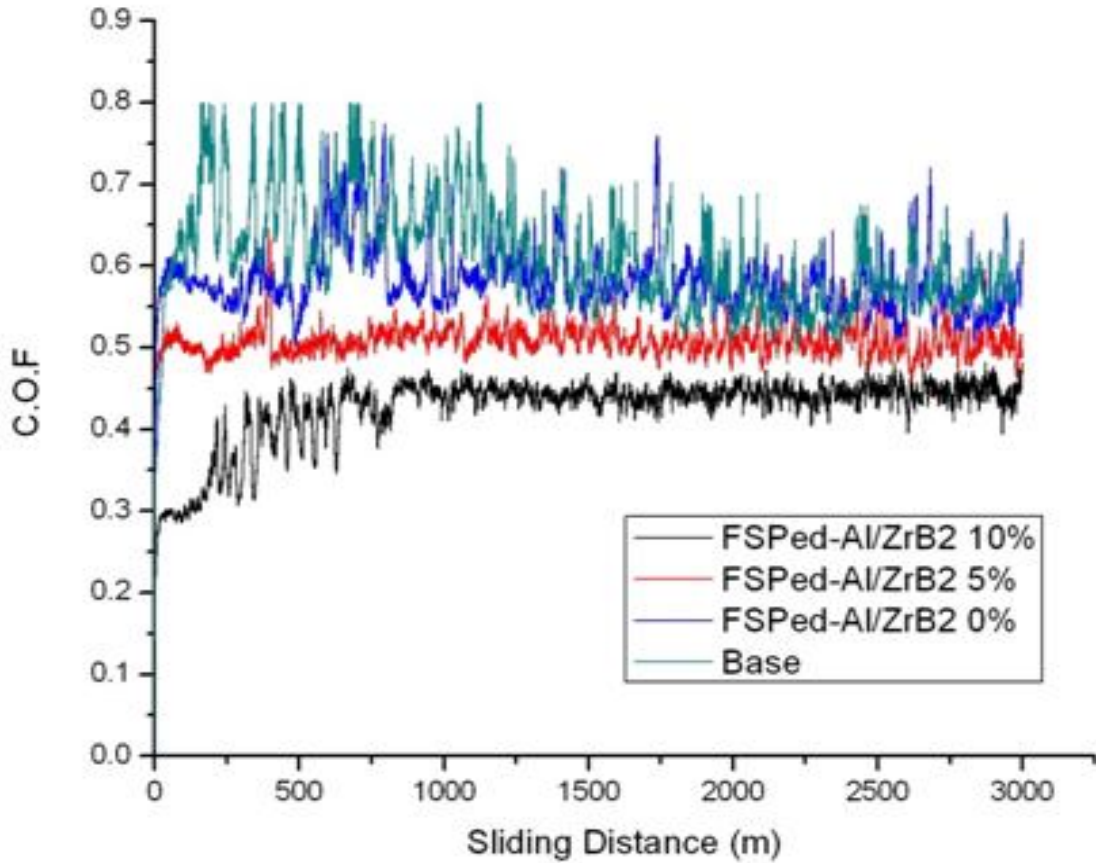


Fig.61. Comparison of C.O.F of the samples

The above figures shows that there is reduction in coefficient of friction after processing .In the base material, when the load is acting on it, the Al matrix directly comes in contact to the counter surface and the whole load is carried by it. Also the soft Al increases the frictional force due to rubbing which results in high coefficient of friction .After insertion of ZrB<sub>2</sub> particles the load is shared by the particles and it transfers the whole load to the counter surface easily because of its high hardness with less rubbing. The average coefficient of friction for base, FSPed-Al/ZrB<sub>2</sub> 0%, FSPed-Al/ZrB<sub>2</sub> 5%,and FSPed-Al/ZrB<sub>2</sub> 10% are 0.62,0.58,0.51and 0.43 respectively. Clearly we can see that the average coefficient of friction goes on decreasing as the hardness is increased and this is due to dynamically recrystallised fine grains and distribution of the ZrB<sub>2</sub> Particles in the Aluminium matrix. A combined comparison of coefficient of friction of all the samples is given in the Fig.61.

#### 4.4.2 SEM of Worn Out Surface of Wear Samples

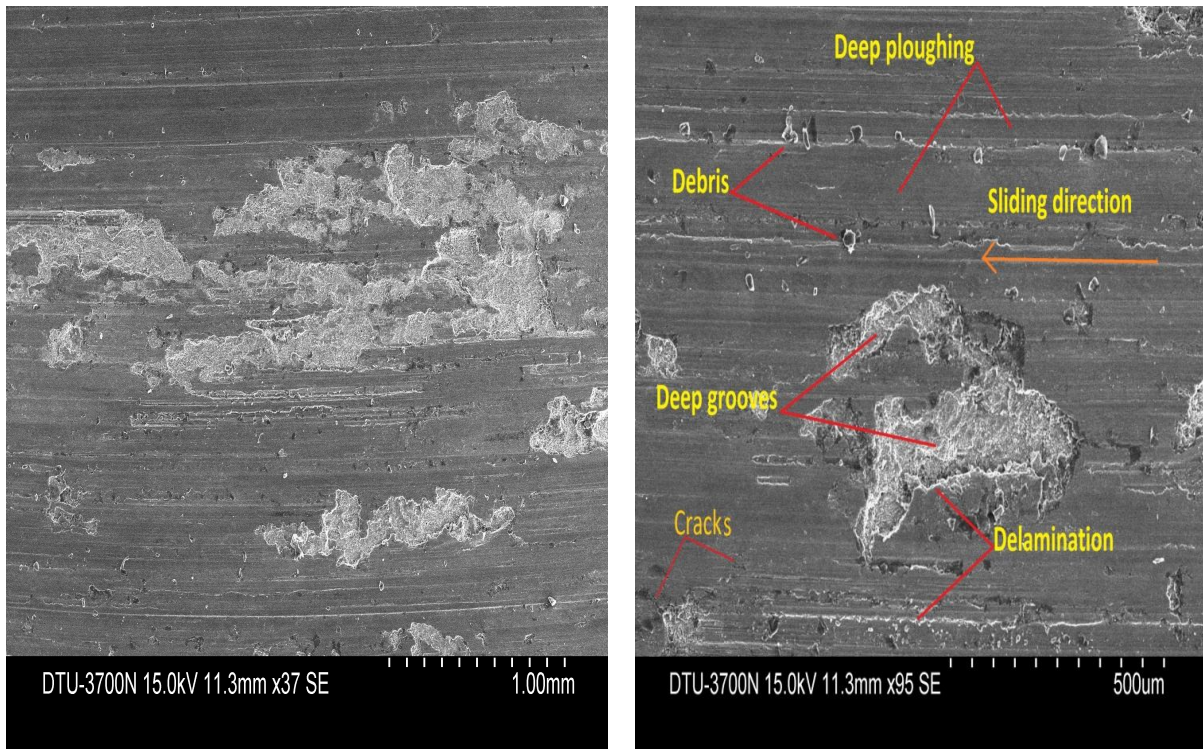


Fig.62.SEM of worn out surface of **Base** at low and high magnification

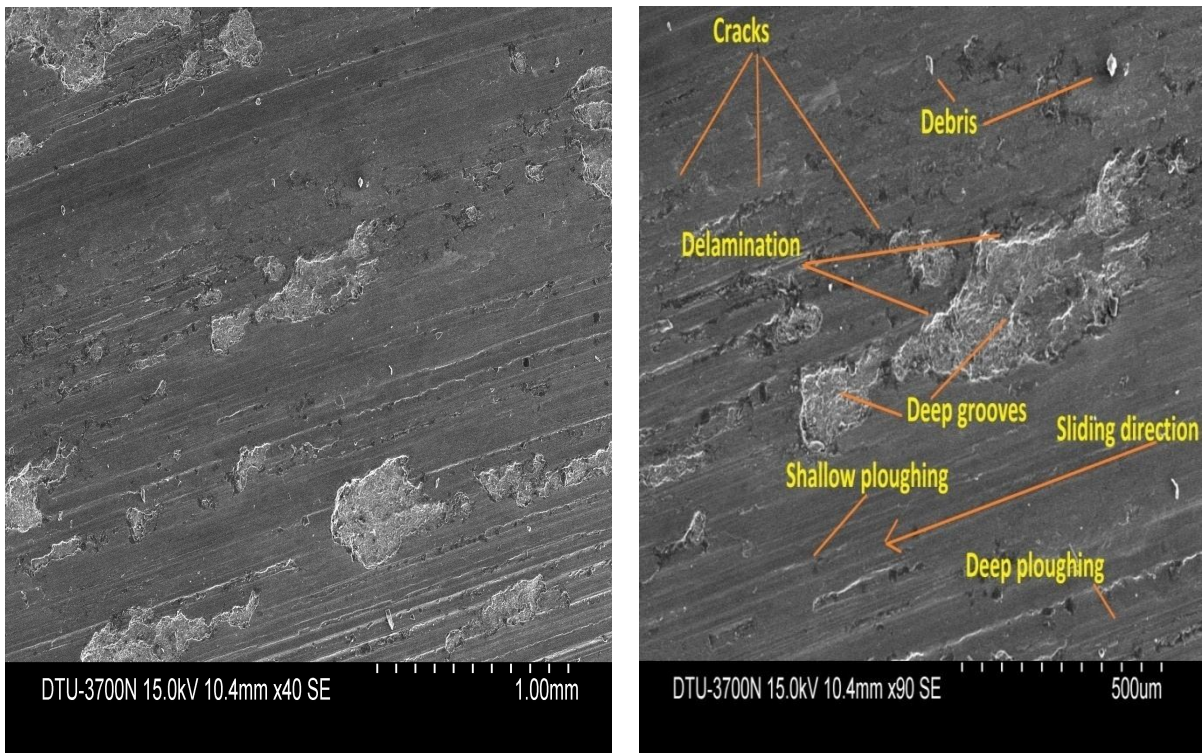


Fig.63.SEM-worn out surface of **FSPed-Al/ZrB2 0%** at low and high magnification



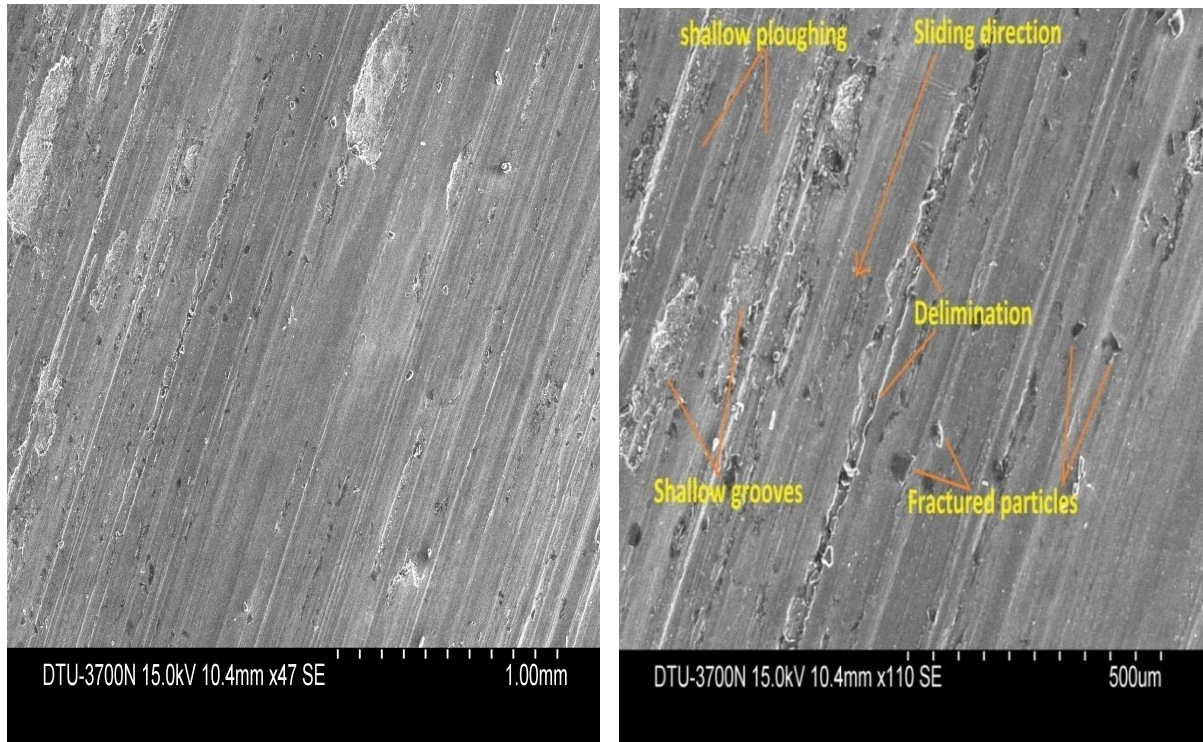


Fig.64.SEM -worn out surface of **FSPed-Al/ZrB2 5%** at low and high magnification

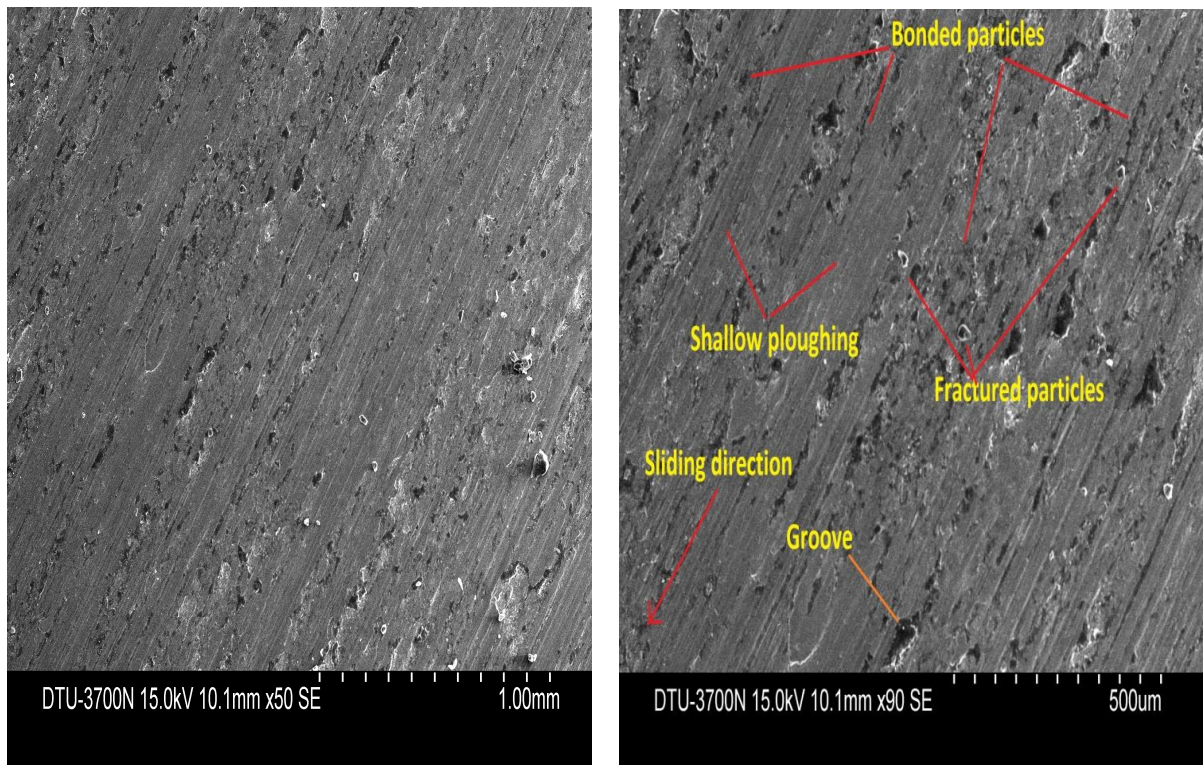


Fig.65.SEM -worn out surface of **FSPed-Al/ZrB2 10%** at low and high magnification

Wearing of any surface is consists of some mechanism like abrasion, adhesion, delamination, cracks, ploughing etc. From the above figures it was observes that at initial stage of wearing, the material from the matrix is removed by abrasion and cutting action of the counter mating surface and in the composite surface the fragmentation of particles takes place.

As the frictional heat goes on increasing with increase in sliding distance, softening of matrix phase and plastic deformation of matrix takes place. These plastically deformed matrix easily taken away by counter mating surface and the wear mechanism changes from abrasion to adhesion. Also because of softening of Aluminium matrix the penetration depth of the counter surface is increased which also increases the wear rate .Further increase in sliding distance the surface becomes irregular and full of cracks and debris which further causes increase in wear.

In the Fig.62 we can see that the surface of the base is severely damaged due to excessive wear. Because of soft matrix, formation of ploughs and grooves are very deep. The cyclic stresses have also resulted in formation delamination. Due to cyclic stresses some layers starts forming and then easily removed away by counter surface. In Fig.64 and Fig.65, increase in ZrB<sub>2</sub> particles, the ploughs and grooves become shallow and the penetration of the counter surface is decreased thereby decrease in wear rate . This is can be attributed to increase in hardness of the composite.

# CHAPTER 5

## CONCLUSIONS

In this research work, surface composite of Al7075/ZrB<sub>2</sub> successfully fabricated by Friction Stir Processing. The conclusions from this study are as given below:

1. After processing the grain size becomes very fine (3 microns) due to dynamically recrystallization.
2. The hardness of base material (86 HV) was increased after processing without ZrB<sub>2</sub> particles to 94 HV. After processing with 5% and 10% vol. fraction of ZrB<sub>2</sub> particles, the hardness is further increased to 102HV and 149 HV.
3. The UTS of base (570 MPa) improved to 582 MPa after processing without ZrB<sub>2</sub> particles and processing with 5%, 10% vol. fraction of particles the UTS increased to 620 and 628 MPa.
4. The elongation of base (13%) is increased to 17% after processing without ZrB<sub>2</sub> particles. Processed sample with 5% vol. fraction of particles indicated increase in UTS without much loss of ductility (11%).
5. Processed sample with 10% vol. fraction of ZrB<sub>2</sub> particles indicated somewhat brittle fracture with only 6% elongation.
6. The coefficient of friction was decreased from 0.62 of base material to 0.43 of processed samples with particles because of increase in hardness.
7. There is reduction in wear rate from 6.3544(base material) to 1.3674(mg/m)  $\times 10^{-3}$  (with ZrB<sub>2</sub>) because of increase in hardness, uniform distribution of ZrB<sub>2</sub> particles and minimization of contact area of soft matrix to the counter surface.
8. The wear mechanism changes from abrasion to adhesion as the sliding distance goes on increasing.

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