FRICTION STIR PROCESSING OF PARTICLE REINFORCED COMPOSITE

A DISSERTATION

Submitted in partial fulfilment of the
requirements for the award of the degree

of

MASTER OF TECHNOLOGY
in

METALLURGICAL AND MATERIALS ENGINEERING
(With Specialization in Industrial Metallurgy)

By

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CANDIDATE' DECLARATION

I hereby declare that the proposed work presented in this dissertation consists title "**Friction stir processing of particle reinforced composites**" for partial fulfilment of the requirements for the award of the degree of Master of Technology in Metallurgical and Materials Engineering with specialization in industrial Metallurgy which is submitted in the Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee is an authentic record of my own work carried out during the period from July 2013 to May 2016 under the supervision of **Dr. Ujjwal Prakash**, Professor, Department of Metallurgy and Material Engineering, Indian Institute of Technology Roorkee, India. The matter presented in this dissertation has not been submitted by me for the award of any other degree.

Dated: May 2016

Place: Roorkee Raghvendra singh

CERTIFICATE

This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

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ABSTRACT

In this study friction stir processing is utilized for microstrucutral modification of metal matrix composite. Friction stir processing is a relatively newer technique for microstrucutral modification. It offers many advantages owing to solid state nature of processing. For preparing metal matrix composite LM13 aluminum alloy and zircon sand of average particle size of 40 µm was used as a particulate reinforcement. Stir casting technique was used for preparing metal matrix composite. Friction stir processing of metal matrix composite resulted in uniform distribution of zircon sand particles with excellent interfacial bonding with the Al matrix. Friction stir processing also led to significant size reduction of zircon sand particles. Microstructural modification of metal matrix composite depends on number of FSP passes. The size reduction of zircon sand particles after four passes was below 10 µm. After four passes of FSP, the metal matrix composite showed improved strength, hardness and wear resistance.

Keywords: Friction stir processing; processing parameters; Mechanical Properties; Microstructure

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CHAPTER 1 INTRODUCTION

1.1 Significance of Friction Stir Process (FSP)

Now days selection for a particular material is key parameter in every industrial application & it become more important when industries belongs to aviation & automotive industries because they needs material with specific properties like high strength, high hardness, low weight & having certain limitation in terms of cost and production time apart from reduction in ductility. So it was necessary requirement that time to develop a technique that would cover all the needs of industrial applicable material related with strength, hardness & ductility in the appropriate rate & time. So, new technique like FSP was developed in addition with advancements in our conventional methods.

Friction stir process (FSP) which is modified form of Friction stir welding (FSW) was invented in 1991 as a solid-state joining technique. Initially it was applied to aluminium alloys [8]. FSP was developed as a general tool for microstructural and mechanical properties modification. It is based on the basic principles of FSW. The basic concept of FSW is remarkable simple in which, a non-consumable rotating tool with a pin and shoulder is plunged into the abutting edges of sheets or plates to be joined and advanced along the line of joint. The heat produced due to the friction between the tool and the work piece and as a result the plastic deformation of the work piece take place, softens the material and a solid state welding is obtained. The FSP is a modified process of FSW, even if applies the same principle which does not join materials but locally modifies the microstructure [9].

FSP having many advantages over the conventional method used worldwide which includes new techniques of material processing like single step process, use of simple and inexpensive tool, less time required for finishing process, use of existing and easily available machine tool technology, suitability for the automation, adaptability for the robotic use, being energy efficient and environmental friendly. Despite the fact that the limitations of FSP are being reduced by concentrated research and development however it has few limitations that include rigid clamping of the work pieces, backing plate requirement, & the keyhole at the end of each pass.

ECAE is also one of the newer materials processing a technique which is used to obtain high strain rate super plasticity at significantly lower temperature.

1.2 Working principle of FSP

For processing a sheet by Friction stir, a non-consumable rotating tool is plunged into the work piece for the localized microstructural modification for enhancing its specific property. Tool has shoulder and pin with its specific dimension and geometry for specific purpose [2]. The tool has to be a small diameter pin with a concentric larger diameter shoulder. A specified rotational speed, traverse speed and certain plunge depth givens to the tool according to the need. Then tool rotates and moves simultaneously into the work piece. A high temp. generates during the process due to rubbing action between the shoulder, pin and the work piece with localized heating. shoulder of the tool & probe-length controls the penetration depth. The probe is typically shorter than the thickness of the metal matrix composite workpiece & its diameter is typically larger than workpiece thickness. The FSP is a solid state process in which solidification structure is absent and problem related to presence of interdendritic & eutectic phase is eliminated. As the pin of tool moves, material is forced to flow around the pin & material undergoes intense plastic deformation at elevated temperature, resulting in generation of defect free & dynamically recrystallized equiaxed fine-grained microstructure. A schematic diagram of FSP has shown in Fig.1.2. Based on the micro structural transformation due to the grain refinement of the materials there are three distinct zones showing : (1) stirred zone (SZ), (2) thermo-mechanical zone (TMAZ) and (3) heat affected zone (HAZ) are shown in tool pin region and nearby region. SZ refers that the zone which is occupied by the tool pin it is fully recrystallized. TMAZ zone refers plastically deformed material and the heat from the process will also have some influence on the material in this zone. Third zone is HAZ where no plastic deformation occurs but the material experiences thermal shocking effect.

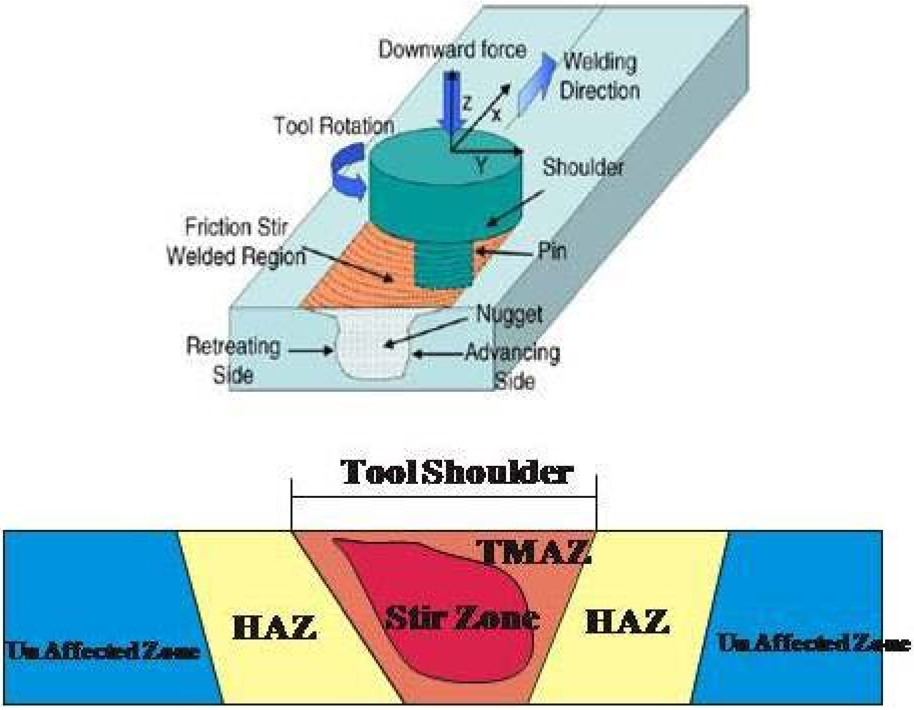

Figure 1.1. Schematic Reprentation of Friction Stir Processing. [1]

1.3 Process parameters in FSP

In order to obtain the desired finer grain size, certain process parameters are to be controlled during FSP (1) tool geometry (2) transverse and rotational speed (3) tilt and plunge depth (4) multi passes [5].

1.4 Surface composites by FSP

Before FSP, Conventional method was used for manufacturing of surface composite like applying a ceramic particles (slurry) layer in a volatile medium but now day’s ceramic particles are reinforced through machined grove in the metal plate [12]. In the process of FSP, Base metal plate is grooved or drilled with holes by a rotating & cutting tool and then these grooved or drilled holes are packed with the reinforcing powders such as SiC, Al_2O_3, TiC or graphite etc. Now FSP is applied on packed powders with optimized process parameters to obtain surface composites material with homogeneous microstructures inside the stirred zone (SZ). Multiple passes are generally required to achieve homogeneous distribution of the powder particles. Due to surfaces composites by FSP, the tribological and mechanical properties like wear, hardness, strength etc. enhanced significantly [21].

Mishra et al. (2003) used FSP in the last decade to fabricate surface composites by applying silicon carbide (Sic) layer on aluminium alloy (AA) 5083 [3]. After then, so many studies have been reported progress in aluminium alloy surface composites. Metallic, ceramic and carbon nano-structures are successfully reinforced in aluminum alloy for surface composite fabrication[8]. It is important to note that the processing of the surface composites is achieved under a solid-state condition. [13]. in the present work, FSP has been done on metal matrix composite. Further we will analysis about the hardness, microstructure, and Tensile strength.

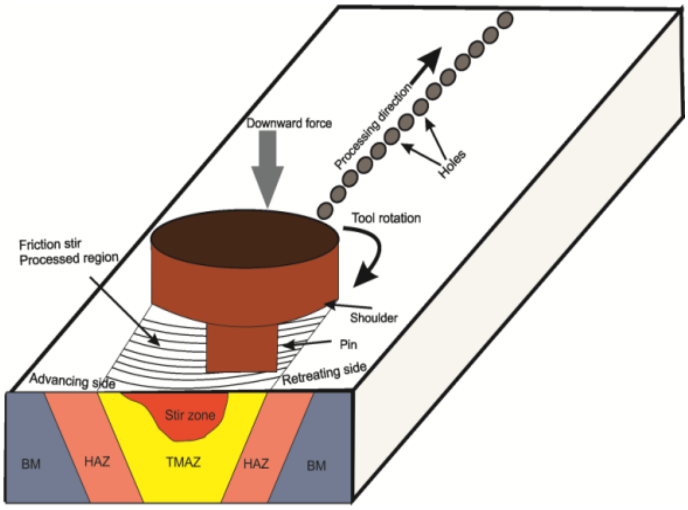

Figure 1.2. Schematic illustration of surface composite fabrication by FSP. [2]

1.5 Objectives of the present work

After understanding the significance of the FSP, our main objective of this thesis is to investigate the effect of the FSP of metal matrix composite material. The following are major objectives:

1. To prepare FSPed surface in metal matrix composite.
2. Applying FSP in metal matrix composite and analysis after each & every pass.
3. To study microstructures of base alloy, FSPed alloy and FSPed composites.
4. To estimate and study of tensile behavior and hardness of base alloy, FSPed alloy and FSPed composites.
5. Attempt to correlate with microstructure & concluding the result received after FSP.

1.6 Thesis Layout

The present thesis is based on five chapters. The first chapter gives a brief description about the introduction of FSP related present work i.e., the significance, working principle of FSP and objectives of the work. The second chapter would provide the detailed literature review on the concept of Friction stir Processing. Third chapter would explain the experimental methodology used for achieving these set objectives. Further, in the fourth chapter results & discussion obtained are presented. Finally in chapter 5 we conclude the work presented in previous chapters and suggest some future work that can be done further to make the study complete.

CHAPTER 2 LITERATURE SURVEY

Friction stir technology was the revolution in the field of welding that time & now days still so many research & developments going on it. This innovative technique produces very fine & equiaxed grains in weld zones. If Friction stir technique could be used as a processing technique, it would replace the existing traditional, complex and expensive processing techniques. FSP can enhance super plasticity property in aluminium alloy.

We can consider FSP as a process in which intense deformation take places to the work piece by the rotating pin and the shoulder. This deformation gives rise to a stir zone (SZ), a thermo-mechanically-affected region (TMAZ) and a heat affected zone (HAZ). The SZ appears to consist of equiaxed and fine grains which size is considerably less than that in the base material. This feature of friction stirred zone resulted in the development of new economical, energy efficient, thermo mechanical material processing technique called FSP. It was performed on aluminium alloys for example 7075 Al and 6061 Al especially to render them super plastic and also was used as a technique to produce aluminium surface metal matrix composite.

FSP being a promising technique, the amount of literature available is less compared to Friction stir welding. Mainly, the effect of process parameters on microstructure and relation between the forces developed and resultant microstructure are not much investigated. There has been wide study on the microstructure of friction stir welded aluminium alloys going on. These studies mostly concentrated on the grain size obtained in the weld zone. Investigations were also made on the effect of rotational speed on microstructure. Tool wear & tear and different optimal tool designs are also being investigated. Mechanical properties like tensile strength and hardness of a friction stir welded joint have been studied.

When we Compared FSP from other metalworking techniques, then we come to know that FSP has distinct advantages. First one is that FSP in a one step processing can achieves microstructural refinement. Second one is that, the properties and microstructural refinement of the processed zone can be controlled by optimizing the process pareamters of FSP.

This section will provide the detailed research that has been done or still going on in the field of Friction Stir technology.

2.1 The microstructure evolution studies during FSP

Main thing which have to be understand about the evolution of microstructure in the dynamically recrystallized region of Friction stirred material and relation of it with the deformation process ,variables of strain, strain rate, temperature and process parameters is very essential. This section would give details about such studies. Friction stir Processing reduces the defects in the cast or sintered material such as porosities, in homogeneity to a very large extent. After the stirring action of the tool, heat is produced due to the friction between the tool pin and the material. Then it makes the material soften due to plastic deformation & recrystallization occours. Due to plastic deformation, material movement takes places & new grains are formed, which results in the high angle grain boundaries in stir zone. As a result the material is perfectly homogenized and consolidated. A fine & equiaxed microstructure also obtained which leads to improved mechanical properties. Fig. 2.1.1 showing the advancing and retreating side in a processed material. The side on which the traverse speed and the tangent to the rotating tool are on the same direction is called the advancing side and retreating side is found at the diametrically opposite end. Based on metal working processes, FSP can be divided into five zones: (1) preheat zone, (2) initial deformation zone, (3) extrusion zone, (4) forging zone, and (5) post heat (cool down) zone which can showing in Fig. 2.1.1 Material flows to the back side of the pin, where it can extrude and forged behind the tool, consolidated and cooled under hydrostatic pressure conditions.

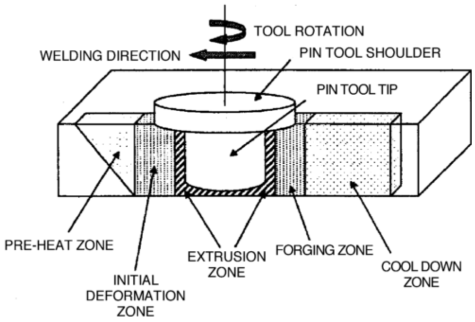

Figure 2.1. Metallurgical processing zones developed during friction stir processing [3]

In a study of the microstructure of cast A356 Aluminum [4], it is observed that at lower rate of rotation speeds, the processed zone (weld nugget) takes basin shape as shown in Fig.3. With increase in speed, the stirring action of the tool is greater and the temperature gradient is higher. Hence, the shape changes into elliptical and after about 800 rpm it acquires onion shape [3, 4]. Further, Adjacent to the nugget zone, thermo mechanically affected zone (TMAZ) is located [3, 4]. It is to be noted that, though in the TMAZ, deformation is observed, but, there is no recrystallization due to insufficient deformation strain. Here, the Si particles are seen in an upward flow pattern. Finally, there is the heat affected zone (HAZ) shown, where there is no any deformation take place Fig. 2.1.2. The interface between the TMAZ and the weld nugget is prominent more in the advancing side. Some band structures (marked as B in Fig.2.1.3a) of coarse Si particles are found in certain combinations of RPM and traverse speed (low rotation rate or low rotation rate to traverse speed ratio). This is because the particles are not well stirred and homogenised at low rotation speeds.Cast A413 aluminium alloy [5] has also been FSPed upon. Si flakes which appear in the cast sample are fragmented to finer particles and dispersed into the matrix. Fluidity of the matrix plays an important role in the evolution of microstructure. At low rotating speed and high traverse speed, the heat input is less; hence cavities appear in the stir zone due to insufficient softening. However, under high heat input condition also, turbulent flow may occur which leads to cavity formation as shown in Fig. 2.1.5 (b). Cavities which appear in the base metal diminish in the stir zone as shown in Fig. 2.1.5 (a). In the FSP of cast or wrought aluminum alloys, matrix grain size of the order of 3- 4 µm is achieved.

Another effect of FSP is that, finer particles are found in the left, right and bottom sections of the nugget, than at the top and middle, representing refined structure dominated more of the lower stir zone region [4]. The top portion is subjected to more heat and less deformation. This is pronounced when the depth of the sample is high. Fig. 2.1.6 shows the above mentioned effect in the FSP of 7075 Al alloy [6]. Zhou et al [7] has performed the FSP on pure Al plate and has studied the conversion of low angle grain boundaries ($2\text{-}15^0$) to high angle grain boundaries ($>15^0$) during deformation in the stir zone. Furthermore, deformation and consolidation is better in the advancing side than in the retreating side. The comparison of the fraction of high angle grain boundaries over the transverse section to the direction of processing is shown in Fig. 2.1.7, where d, e and f are in the stir zone, and the rest are in the TMAZ and HAZ.

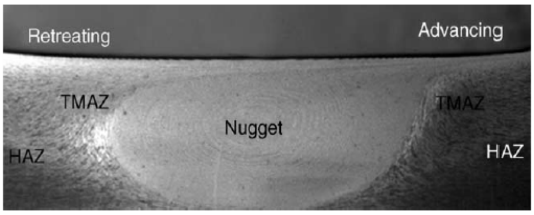

Figure 2.2. A typical macrograph showing various microstructural zones in FSP 7075Al [4]

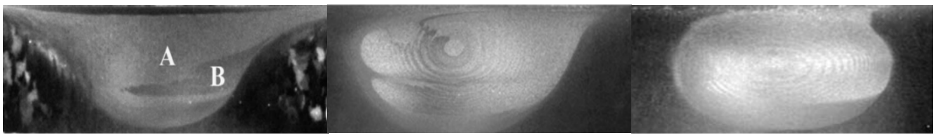

Figure 2.3. Effect of processing parameter on nugget shape in FSP A356: (a) basin shaped, 300 rpm, 51 mm/min and (b) elliptical, 700 rpm, 102 mm/min (c) onion shaped, 900 rpm, 203 mm/ min [5]

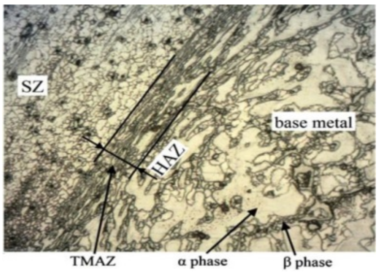

Figure 2.4. Different zones of FSPed specimen AZ91/SiC [6]

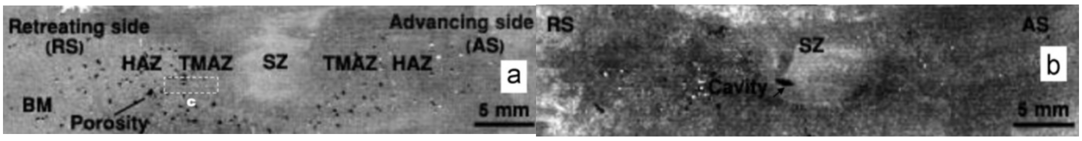

Figure 2.5. Typical macrostructures generated by FSP on Cast A413 aluminum alloy at tool traverse speed of 20 mm/min and tool rotational speed of (a) 1150 rpm and (b) 1400 rpm [7].

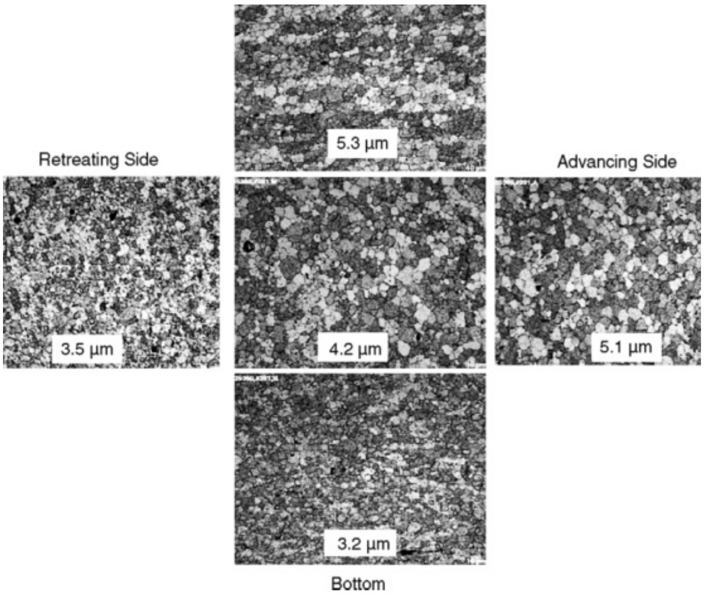

Figure 2.6. Grain size distribution in various locations of 7050Al weld nugget [8].

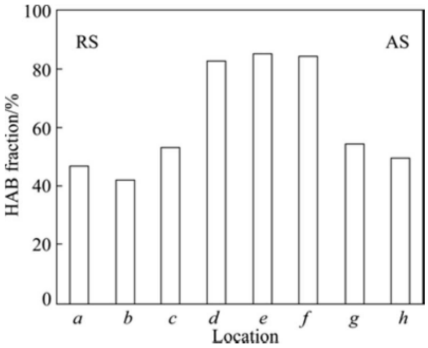

Figure 2.7. Fractions of high-angle boundary for regions across a processed sample [9]

2.2 Process parameters in details

In Friction Stir Processing, there are several processing parameters like tool rotational speed, transverse speed, the tool tilt angle, penetration depth and cooling effect. Classification of process parameters of FSP is shown in Fig. 2.2. The tool rotational speed and transverse speed determine the amount of heat generated in the work piece

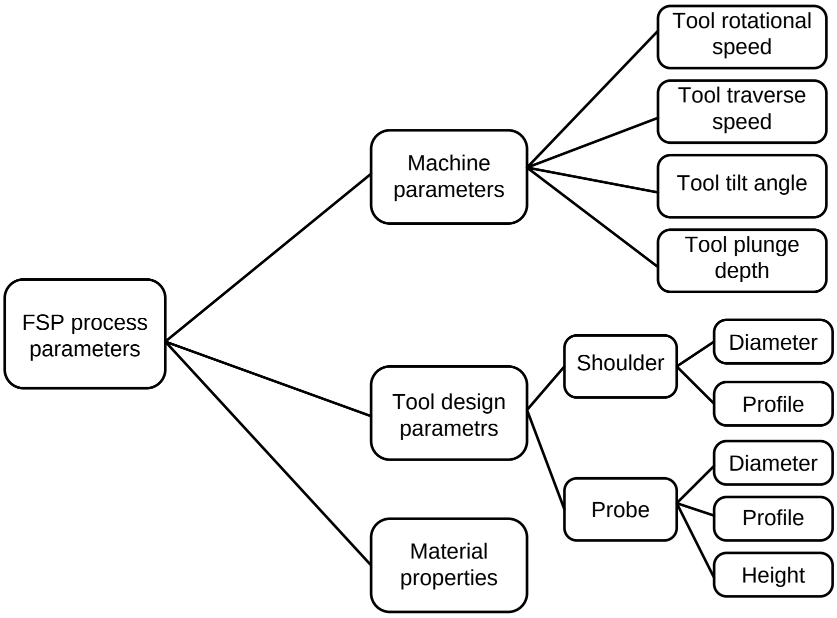

Figure 2.8. Classification of process parameters in FSP [9].

2.2.1 Study on Tool geometry

Tool geometry that is consists of shoulder and pin (probe). The shape and size aspect of shoulder and pin are pivotal and effective features of tool geometry. In General the shoulder of tool having the concave shaped (profile) as it serves as stopper and controller for the escaped softened and plasticized materials of the pin region [3]. The tilt angle of 1-3° is required for the effective processing of material, tilt angle is also required to maintain the material beneath the tool and enables the trailing edge of the shoulder tool for extruding the processed material properly. The tool shoulder is highly responsible for heat generation in the matrix. If we use large diameter shoulder then it leads to high heat generation whereas if we use low diameter shoulder it results formation of defects in metal matrix composite. [1]. The probe also contributes the heat content in the matrix but it is lesser than shoulder portion.

Tool pin geometries may be taken as triangular, cylindrical, square, threaded, conical etc. for making the surface composites effectively. Some common tool probe is shown in the Fig. 2.2.1 The pin geometry and shape determine the mixing of the plasticized material and strength of the pin during FSP. Threaded cylindrical, square and triangular pin geometries have been used largely in FSP. Rai et.al 2011, suggested that tungsten, molebidinum, iridium also may be used as most suitably tool material as they posses high hardness, high temp strength, low reaction with oxygen [2].

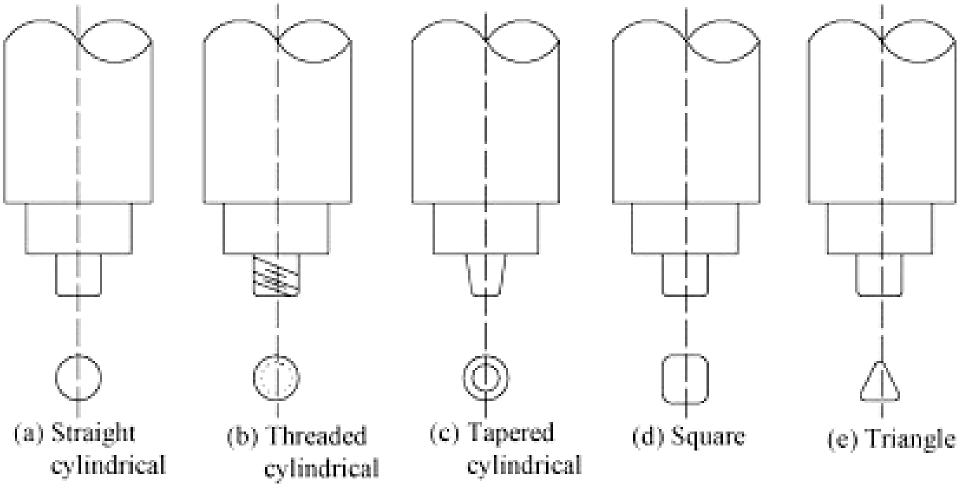

Figure 2.9. Tool probe geometry in FSP for fabrication of surface composite (a) straight cylindrical (b) threaded cylindrical (c) tapered cylindrical (d) square (e) triangular probe [10].

The pin outer surfaces can also have different shapes and features including threads, flats, flutes or combination with these features [14-19].

Thomas et al. suggested that tool geometry in FSP affect and governs the material flow. And also reported that tool with whorl pin profile having better mixing ability than conventional cylindrical pin and it performed better ratio of swept volume which was defined as ratio of material volume displaced by pin to the volume of pin itself. Swept volume for cylindrical pin 1.1:1 and that is for whorl pin 2.6:1[14].

Mahmoud et al. suggested that the surface composite fabrication of SiC on the aluminium material, square probe give more homogenous distribution of SiC particles than the triangular & cylindrical shape tools [15].

Bahrami et al. suggested that 250 nm bands of agglomerated SiC particle were observed with threaded tapered tool while 480 nm bands of agglomerated SiC particle were observed with four flute cylindrical tools [16].

N yuvaraj et al. reported about the effect of threaded tool geometry for fabricating the surface composite of AA5083 with macro (20 µm) and nano size (30-60 nm) particle of B_4C. And found uniform dispersion of B_4C with nano particle size [17].

Sharaeinejad et al. reported about the geometry, profile of probe and number of FSP passes affected the distribution of Al_2O_3 particles in AA5069 to homogenize the matrix uniformly. Then they reported that the threaded pin having cylindrical geometry homogenized the Al matrix uniformly for producing proper plastic deformation, flow & mixing of Al_2O_3 particles' in AA5069 than three flat probe having partial threaded geometry [18].

Vijayavel et al. reported that the ratio of diameter of shoulder (D) to diameter of probe (d) influenced the mechanical and microstructural properties of SiC/stir cast Al composites. Good tensile strength, hardness and proper mixing of SiC particles in matrix were obtained with 2 D/d ratio by severe plastic deformation [19].

2.2.2 Studies about Tool rotational and traverse speed

The rotational speed and traverse speed determine the amount of required heat input in the stir zone, which affects largely the microstructure and mechanical properties of the composite material. Heat input in the stir zone directly influences the grain refinement. More grain refinement occurred by lowering the heat input and vice-versa, but there must be sufficient heat input to plasticize or soften the material. In surface composite fabrication for distribution and breaking up of clusters of reinforcement particles, higher rotational speeds are required. So, the rotational and traverse speeds are carefully optimized to achieve the defect free stir zone and reduced grain size [20-22].

Kurt et al. reported that increasing rotating tool speed (ω) and traverse speed (v) caused uniform distribution of SiC particles in surface composites fabricated by FSP of AA1050 alloy. At very high transverse speed, the surface composite layer was usually weakly bonded with the aluminum alloy substrate. The thickness layer of the composite was significantly high compared with the low travelling speed specimen and high rotational speed. At 15 and 30 mm/min traverse speed, the layer thickness were 150 µm and 75 µm, respectively for SiC reinforced aluminium alloy composite. Increased travelling speeds resulted in significant increment of hardness for all rotational speeds for base alloy [20].

Thangarasu et al. studied the influence of traverse speed on the micro hardness of TiC/AA6082 surface composites via FSP. They revealed that micro hardness of the surface composites increased with increase in traverse speed due to high volume fraction of TiC observed at 80mm/min, while distributions of reinforced particles of TiC was uniform at 40mm/min traverse speed Micro hardness at 40, 60 and 80 mm/min traverse speed were found 112 HV, 125HV and 135HV, respectively [21].

Dolatkhah et al. showed the effect of the rotational speed and traverse speed on the particle distribution of SiC on the surface of Al 5052 alloy. They showed that the most homogenous powder distribution was achieved with a combination of tool rotational speed of 1120 rpm and traverse speed of 80 mm/min [22].

2.2.3 FSP multi-passes

Usually, single pass is not enough to provide proper distribution of reinforced powder particles within base materials. So in FSP, multiple passes play a key role to remove inhomogeneous distribution by breaking the clusters of powders in stir zone(SZ).[23-24]

Liu et al. studied the analysis of reduction of carbon nano tubes (CNTs) in fabricating 2029Al alloy matrix with 4.5 volume percent CNTs by a combination of powder metallurgy and FSP. As the number of FSP passes increased, the fraction of CNT clustering decreased and CNTs were dispersed into the aluminum matrix. The homogeneously distributed CNTs resulted in a finer grain size of the matrix. The strengths of the CNT/2009Al composites were improved significantly after FSP and the maximum strength obtained with three-pass FSP. The CNTs were cut short after FSP due to the shear effect. The CNT length decreased upto 0.24 µm and diameter upto 10.6 nm after five number of FSP passes. The yield strength of the three-pass FSP composite increased by about 64% compared to that of the forged composite after five passes[23].

Izadi et al. studied multi pass effect on the micro hardness of the CNTs/AA5059 composite fabricated by FSP. They found that multi wall carbon nanotubes (MWCNTs) distributed uniformly in Al 5059 alloy with high volume percentage and gave high micro hardness of 243HV after 2 passes, micro hardness of 372 HV after 3 passes that were higher than the micro hardness (90 HV) for base AA5059. The micro hardness increased due to the direct strengthening and grain refinement [24].

2.3 Mechanical properties

Devraju et al. reported that tensile properties such as ultimate tensile strength (UTS), yield strength (YS), and percentage elongation (%EL) and microhardness were decreased with increasing rotational speed and constant traverse speed (40mm/min) with reinforcement of SiC, Al_2O_3 and Graphite (Gr) in AA6061 matrix. This was attributed to the hindrance to grain boundary sliding, dislocation movement and weak interfacial bonding of reinforced particles with matrix [25].

Zohoor et al. reported that microhardness and tensile properties such as UTS, YS of nanocomposite of Cu in AA5083 matrix enhanced when compared against FSPed alloy and base alloy at higher rotational speed (1900 rpm). This was due to increasing dislocation density and intense grain boundary pinning effect [26].

Dollatkhah et al. also observed that multi pass FSP increased microhardness of SiC/AA5052 nano surface composites with 5μm SiC particle size. The increased pinning action to the grain boundary and grain refinement were accounted for the increase in micro hardness [22].

Bauri et al. utilized FSP to homogenize the particle distribution in TiC/Al in situ bulk composite, which was fabricated by melt stirring. A single pass of FSP was not enough to break the particle segregation from the grain boundaries and two passes of FSP resulted in homogenization and elimination of casting defects. The strength and hardness of the composite improved substantially after FSP without compromising the loss in ductility [27].

Chen et al. studied aluminum alloy in situ composites from powder mixtures of aluminum and cerium oxide. The in situ reinforcing phases are observed in composites after sintering and these phases homogenize after FSP. The reinforcing phases $Al_{11}Ce_3$ of 1.4-3.5 μm size and δ-Al_2O_3 of 10 nm size were found uniformly distributed in the aluminum matrix. The composite possessed enhanced modulus and strength as well as moderate ductility. The high strength of the composite increased due to sub micrometer grain structure of aluminum matrix in the SZ and the Orowan strengthening caused by the fine dispersion of nanometer sized Al_2O_3 particles inside Al matrix [28].

Magdy et al. observed that microhardness values were decreased from SZ to HAZ and found constant in the SZ in AA7010. The highest hardness observed in the SZ was due to the existence of fine-grained microstructure and severe plastic deformation. The TMAZ zone was slightly softer than the SZ due to partial recrystallization [29].

Mazheri et al. reported that in the Al_2O_3/A356 surface composites fabricated by the combination of FSP and high velocity oxy-fuel spraying with micro and nano particles size. In case of FSPed composites, the hardness decreased relative to base A356 due to dissolution of precipitates in the matrix and maximum hardness achieved in case of nano size Al_2O_3/A356 surface composites due to grain refinement and Orowan strengthening [30].

Balasubramanium et al. investigated that increase in volume percentage (as 5%,10% and 15%) of SiC particles of 17-20 µm enhanced the hardness (from 55.8 kgf/mm^2 to 86.07 kgf/mm^2). But tensile strength decreased from 178 MPa at 10% volume fraction to 165 MPa at 15% volume fraction producing brittle characteristics in AA6063. With the increment of volume fraction of the SiC, the tensile sample failure occurred due to propagation of microvoids (ductile failure) and transgranular cleavage (brittle failure) near SiC and AA boundaries. They reported that ductile failure dominated the tensile fracture [31].

2.4 Fine grained structure through FSP

When we used FSP, its generates significant frictional heating & intense plastic deformation, thereby resulting in the occurrence of dynamic recrystallization in the stirred zone (SZ). In this case, fine and equiaxed recrystallized grains of quite uniform size were produced in the Stir zone. Figure 2.4 shows a typical microstructure of FSP 7075AlT651. In which a fine-grained microstructure of ~7.5 micrometer was produced at a tool rotation rate of 400 rpm and a traverse speed of 102 mm/min. even though there is still a controversy about the grain-refinement mechanism in the Stir zone. It is generally believed that the grain refinement is due to dynamic recrystallization. Therefore, the factors influencing the nucleation and growth of the dynamic recrystallization will determine the resultant grain microstructure in the Stir zone. It has been demonstrated that the FSP parameters, tool geometry, material chemistry, work piece temperature, vertical pressure, and active cooling exert a significant effect o size of the recrystallized grains in the Stir zone.

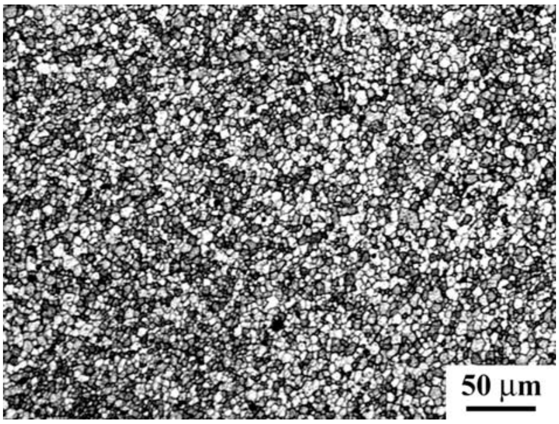

Figure 2.10. Optical micrograph showing fine and equiaxed grains in FSP 7075Al-T651, at processing parameters of 400 rpm and 102 mm/min[11].

2.5 Tribological Behaviour

Alidokht et al. studied the wear behavior of A356 Al alloy based hybrid composite of SiC and MoS_2 produced by FSP. They demonstrated that reinforcement of SiC particles (SiCp) in A356 Al alloy increased wear resistance of the composites but the addition of solid lubricant MoS_2 in SiCp /A356 composite as hybrid reinforcement further enhanced the wear resistance of the composites by producing the stable and MoS_2 rich mechanically mixed layer (MML), which prevented the metal to metal contact. They found that composite has lesser worn surfaces and high microhardness than FSPed A356 due to microstructural refinement of Al matrix by breaking up of Si particles and Al dendrites, and making uniform distribution of Si particles in Al matrix.

Mahmoud et al. [15] studied the wear characteristics of Al base hybrid composites reinforced with a mixture of SiC and Al_2O_3, fabricated on Al plate by FSP in their relative weight ratio. They found that SiC, Al_2O_3 or their mixture uniformly distributed over nugget zone by FSP having only some small void defects. They found that these micro voids were mainly found around the Al_2O_3 particles while it was less noticeable around SiC particles. At 2N normal load, wear resistance decreased with increment in particle ratio of Al_2O_3 in reinforcement. The wear resistance was independent of relative proportion of SiC and Al_2O_3 at 10N normal load but in the case of 5N, 20% SiC and 80% Al_2O_3 in reinforcement performed superior wear resistance [32].

Zahmatkesh et al. investigated the microstructural and tribological behaviour in AA2024 alloy via FSP with constant tool rotating speed of 800 rpm and traverse speed of 25 mm/min. A very fine equiaxed grains were achieved with 4µm in nugget zone due to dynamic recrystallization. The severe plastic deformation reduced the grain size. The heat-affected zone (HAZ) experienced a thermal cycle, but does not undergo any plastic deformation. The variation of friction coefficient with sliding distance for base metal (BM) and NZ samples are found due to the continuous deposition and elimination of wear debris on the track and repeated band structure in the tool traveling direction resulted from the tool pitch [33].

3.1 Experimental Methods:

In this work, metal matrix composites (MMCs) were prepared by melt stirring or stir casting technique. Stir casting set-up is shown in Fig. 3.1. Stir casting is the one of the extensively used and economical synthesis technique for fabricating the particulate reinforced aluminum reinforced composite. It is simpler as compared to other available techniques and flexibility in tailoring the desired properties in the composite.

Figure 3.1. Stir casting set-up [12]

For preparing metal matrix composites LM13 alloy and zircon sand ($ZrSiO4$) are used. LM 13 alloy has been chosen for matrix alloy as it is used as commercial piston alloy and contains around 1% Mg which is necessary for wetting of dispersoid in the matrix. The composition of LM13 alloy in weight percentage is provided in table-1.

Table 3.1. LM13 alloy composition in wt.%

Si	Cu	Fe	Mg	Mn	Zn	Ti	Ni	Al
11.7	1.2	0.28	0.9	0.4	0.2	0.02	0.9	Balance

Following are the main advantage of using zircon Sand as reinforcement:

(1) High hardness,
(2) High modulus of elasticity
(3) High temperature resistance (melting point of 2500 °C)
(4) Acid corrosion resistance
(5) Thermal stability

Main properties of zircon sand is given in Table-2

Table 3.2. Properties of zircon sand

Properties	Values
Melting Point (0C)	2500
Limit of applications(0C)	1850-1880
Hardness(Mohr’s Scale)	7.5
Density(g/cm3)	4.25
Linear coeff. of expansion(10-6 K)	4.5
Fracture toughness (MPa- m1/2)	5
Crystal structure	Tetragonal

The process for fabrication of metal-matrix composites by stir casting consist of melting LM13 alloy in a crucible. After melting of alloy, the reinforcement particles of zircon sand were mixed in the melt by the rotating graphite stirrer. Stirring of melt was carried out for 10 minutes to ensure proper mixing of reinforcement particles in the melt. The melt is poured in a metallic mould and allowed to solidify at room temperature. The process parameters for fabrication of MMCs are presented in table-3.

Table 3.3. Process parameters for fabricating MMCs

Melting temperature	750 °C
Stirring time	10 minutes
Position of stirrer	up to 2/3 depth in the melt
Reinforcement wt.%	15 %
Reinforcement size	32-50 µm

After solidification, the MMC was cut in required size of 120 × 60 × 6 mm for conducting FSP. Schematic of FSP is presented in Fig. 3.2.

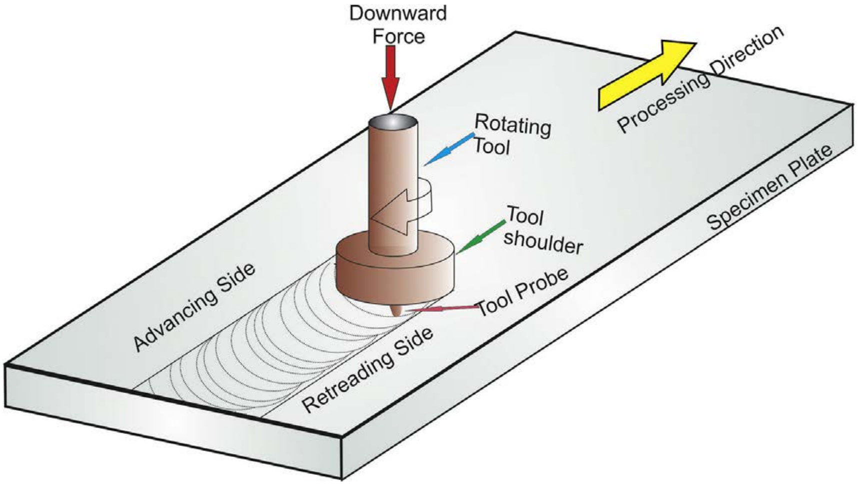

Figure 3.2. Schematic of FSP technique[13]

The MMC plate was fixed on the vertical milling machine for conducting FSP. Experimental setup of FSP used in this study is shown in Fig. 3.3. A hot die steel tool (60 HRC) with a shoulder of 20 mm diameter and cylindrical pin of 4 mm length with 4 mm diameter was used to process the MMCs (Fig. 3.4). One pass, two passes and four passes of FSP was applied. In two and four passes of FSP, 100% overlapping of previous pass and changing the direction of tool rotation after each pass. The FSP of the plates was carried out at the tool rotational and traverse speed of 1400 rpm and 63 mm/min, respectively.

Figure 3.3. Vertical milling machine set-up for conducting FSP[14]

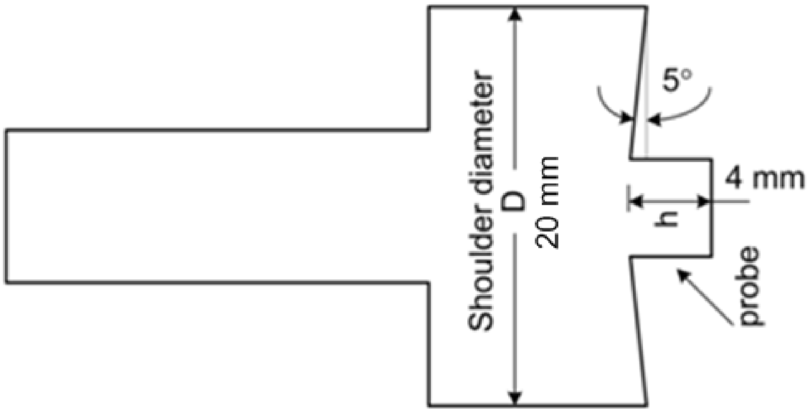

Figure 3.4. FSP tool dimensions [15]

3.2 Microstructural characterization

For the characterization of the MMC a thin slices were cut from the as-cast and stir zone of the plate. Basically microscopy was carried out using LEICA DMI 5000M optical microscope. Samples for metallographic investigation were prepared using emery papers of increasing grades (starting from 320 grades & up to 1500 grit size) and polished by velvet cloth with alumina powder. Etching is done by modified Poulton's reagent (40 mL HNO_3, 30 mL HCl, 12 g CrO_3 , 2.5mL HF, 42.5 mL H_2O) for 10 seconds.

Figure 3.5. Polishing Machine for polishing & grinding purpose

Then after microstructure is inspected in Leica DMI 5000 M optical microscope attached with digital camera (Fig 3.6). We know that etched surfaces of friction stir processed sample were observed by using optical microscope to reveal the samples structure.

Figure 3.6. Lieca Optical micrpscope

3.3 Hardness Testing

The hardness of the samples was measured by Vickers’s micro hardness tester (Omnitech MVH II, India). A 200 gf load was applied for a dwell time of 15 seconds. Total 10 measurements at same load were taken for each sample (i.e base material , 1 pass, 2 pass & 4 pass material) and then average value with standard deviation was reported. The microhardness tester is shown in Fig. 3.7.

Figure 3.7. Vickers’s micro hardness tester

3.4 Tensile Test

The tensile tests were conducted on universal testing machine (UTM) (TINIUS OLSEN, MODEL: H25K-S, 25 KN, UK). The flat shape tensile specimens were cut from the processed zone and tested with a cross head speed of 1 mm/min. All specimen tested at same room temperature & Hardness values were measured using the Computerized Vickers Hardness Tester. The tensile specimen dimensions are shown in Fig. 3.8.

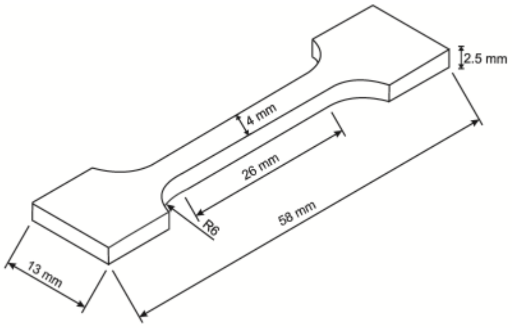

Figure 3.8. Dimensions of tensile test specimen

Figure 3.9. Tensile testing machine (UTS)

3.5 Sliding wear

Sliding wear tests at 10N and 20 N were performed using a ball-on-disc tribometer (TR-201E-M2,DucomInstruments, Bangalore, India). A photograph of tribometer is shown in Fig.3.10. Sliding wear tests were done in laboratory conditions of 30 ± 5% RH and 35 ± 5^{o}C.Slinding wear tested samples are shown in Fig. 3.11. The friction force was measured by a transducer and the coefficient of friction was recorded on line.

Figure 3.10. Tribometer

Figure 3.11. Sliding wear tested samples

For each load three samples are tested and their average value of COF is reported. A profilometer (Mitutoyo SJ-400, South Korea) was used to measure wear track width and depth, and the wear volume is calculated as per the following formula:

Wear volume = $2\pi r\Delta r\Delta h$ ……………………………………………………………(1)

where r is wear track diameter, Δr is average track width and Δh is average track depth.

4.1 Microstructural analysis:

The micrograph of zircon sand reinforced MMC is shown in Figs. 4.1 (a-d). Distribution of zircon sand was not uniform and particle-matrix interface was also observed (Fig. 4.1a). This interface consists of brittle phases which further deteriorate the mechanical properties. After one pass of FSP break up of zircon sand particles and acicular silicon were observed (Fig. 4.2b). Particles agglomeration and casting defects were eliminated in a one pass of FSP. However, some zircon sand particles having their original size. Particle-matrix interface also showed the presence of brittle phases. Thus one pass of FSP can not effectively refine the zircon sand particle size.

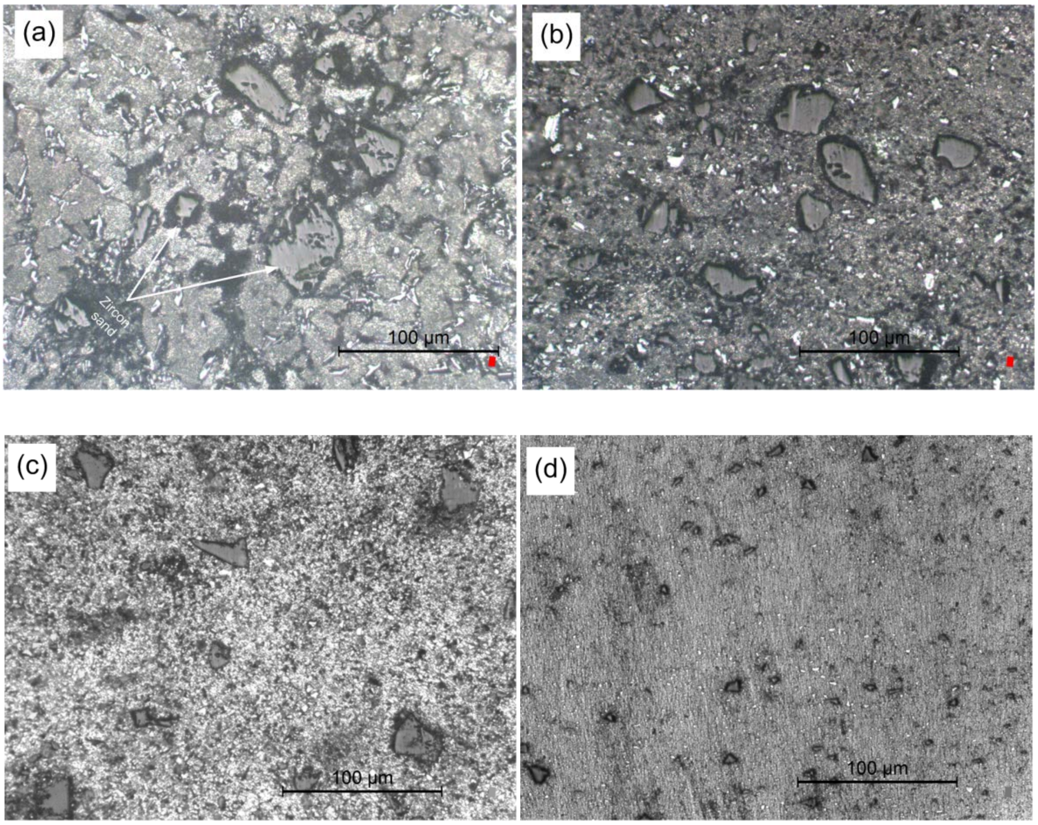

Figure 4.1. Optical micrographs of MMC and FSP specimens (a) zircon sand/LM13 MMC(b) stir zone after one pass of FSP (c) stir zone after two passes of FSP (d) stir zone after four passes of FSP

After two passes of FSP, the zircon sand particles size was decreased to below 30 µmas that of 50 µm in the MMC (Fig. 4c). The silicon was also refined to a greater extent in the processed zone. Repeated deformation occurred in second pass of FSP resulted in break up of zircon sand and silicon. More significant change in zircon sand particles was observed after four passes of FSP as shown in Fig. 4.1 (d). The zircon sand particles size was decreased to less than 10 µm and silicon size was decreased to 1-2 µm. Moreover, the distribution of zircon sand particles was also uniform as compared to one and two pass of FSP. Four passes of FSP imposes intense plastic deformation which reduces the size of zircon sand particles.

4.2 Hardness:

Microhardness of MMC and FSPedMMC is shown in Fig. 4.2. The microhardness of MMCincreases after one pass of FSP. Microstructural modification and uniform distribution of zircon sand resulted in increase in hardness. After two passes of FSP hardness improved due to size reduction of zircon sand particles. Four passes of FSP causes further improvement in hardness due to break up of silicon and size reduction of zircon sand particles. Moreover, uniform distribution of zircon sand particles after four passes of FSP also contribute to increase in hardness. It was concluded that hardness increases with increase in number of passes of FSP.

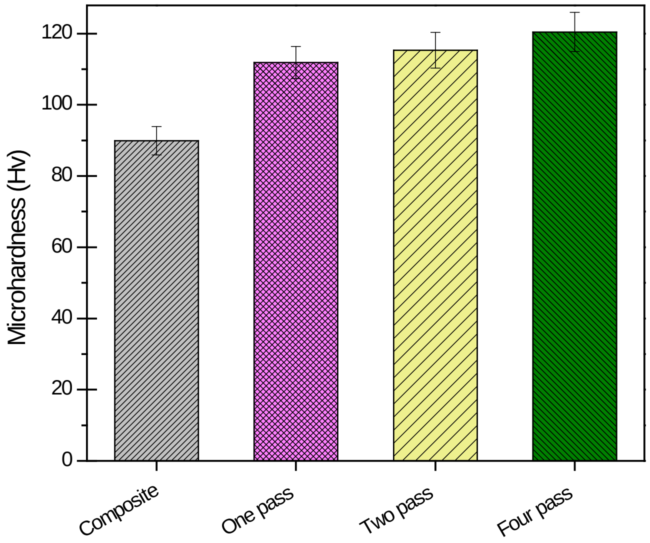

Figure 4.2. Microhardness of MMC and FSP specimens

4.3 Tensile strength:

Tensile strength of the MMC and FSPed MMC is shown in Fig. 4.3. The MMC exhibits low tensile strength and ductility. The tensile strength of MMC was found to be 60 MPa and elongation was below 1%. The lower strength of MMC was attributed to the coarse zircon sand particles which can easily initiate void formation and subsequently fracture. After one pass of FSP the strength improves and elongation was increased above 1%. Microstrucutral modification causes the improvement in strength and elongation. Strength further improves after two passes of FSP due to size reduction of zircon sand particles. After four passes of FSP, the strength increase to 116 MPa and four times improvement in elongation as compared to non-processed MMC. Size reduction of zircon sand, grain refinement and break up of silicon are main contributor to the improved strength. It was concluded that after four passes of FSP the tensile strength was increased to nearly two times as compared to un-processed composite.

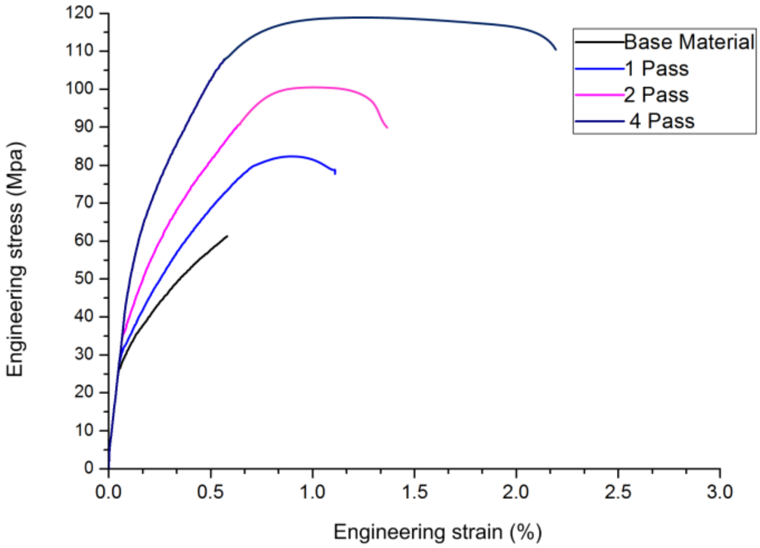

Figure 4.3. Engineering stress-strain curve of MMC and FSP specimens

4.4 Tribological characteristics:

4.4.1 Coefficient of friction:

The average coefficient of friction (COF) of MMC and friction stir processed specimens is shown in Fig. 4.4. For the MMC the variation is observed from 0.29 to 0.3 with increase load from 10N to 20N. It is no significant difference in higher load than small load which shown in Fig.. But the variation occurs 0.29 to 0.25 from MMC to 4 pass MMC at 10N and 0.3 to 0.26 from MMC to 4 pass MMC at 20N. But it is still not significant difference from MMC to 4 pass FSPed MMC.

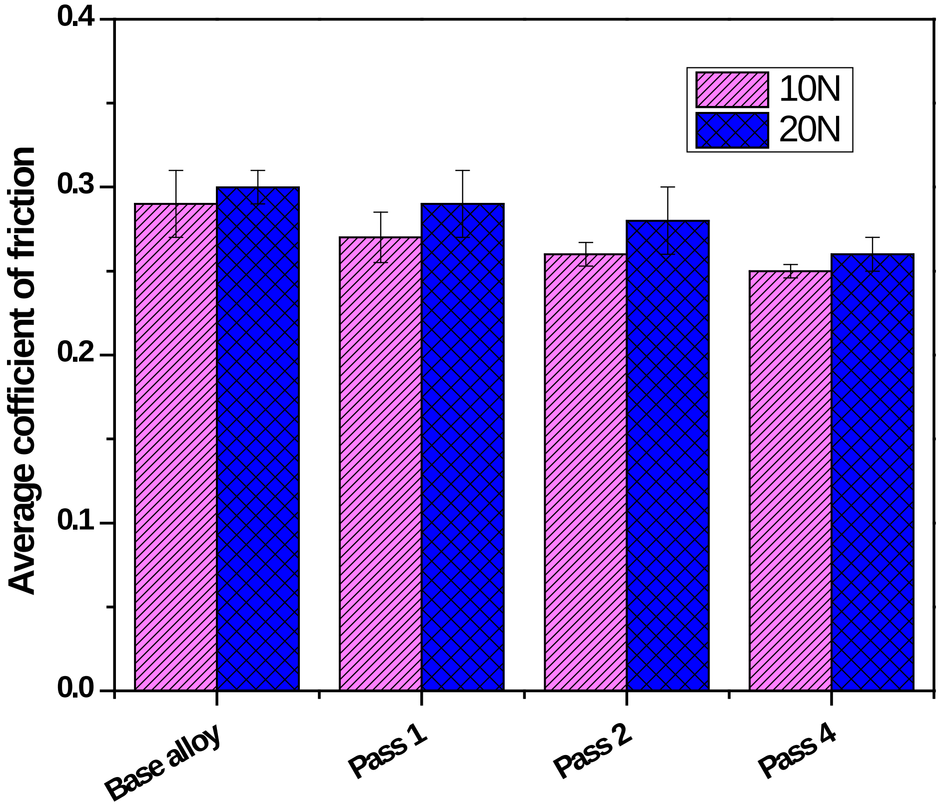

Figure 4.4. Average coefficient of friction of MMC and FSPedMMC at 10 and 20N.

4.4.2 Wear volume:

The wear volume of MMC and friction stir processed specimens is shown in Fig. (and). The calculated wear volume of MMC and FSPedMMCat 10 N and 20N load is shown in Fig. A continuous decrease in wear volume is observed from MMC to 4 pass MMC. The higher

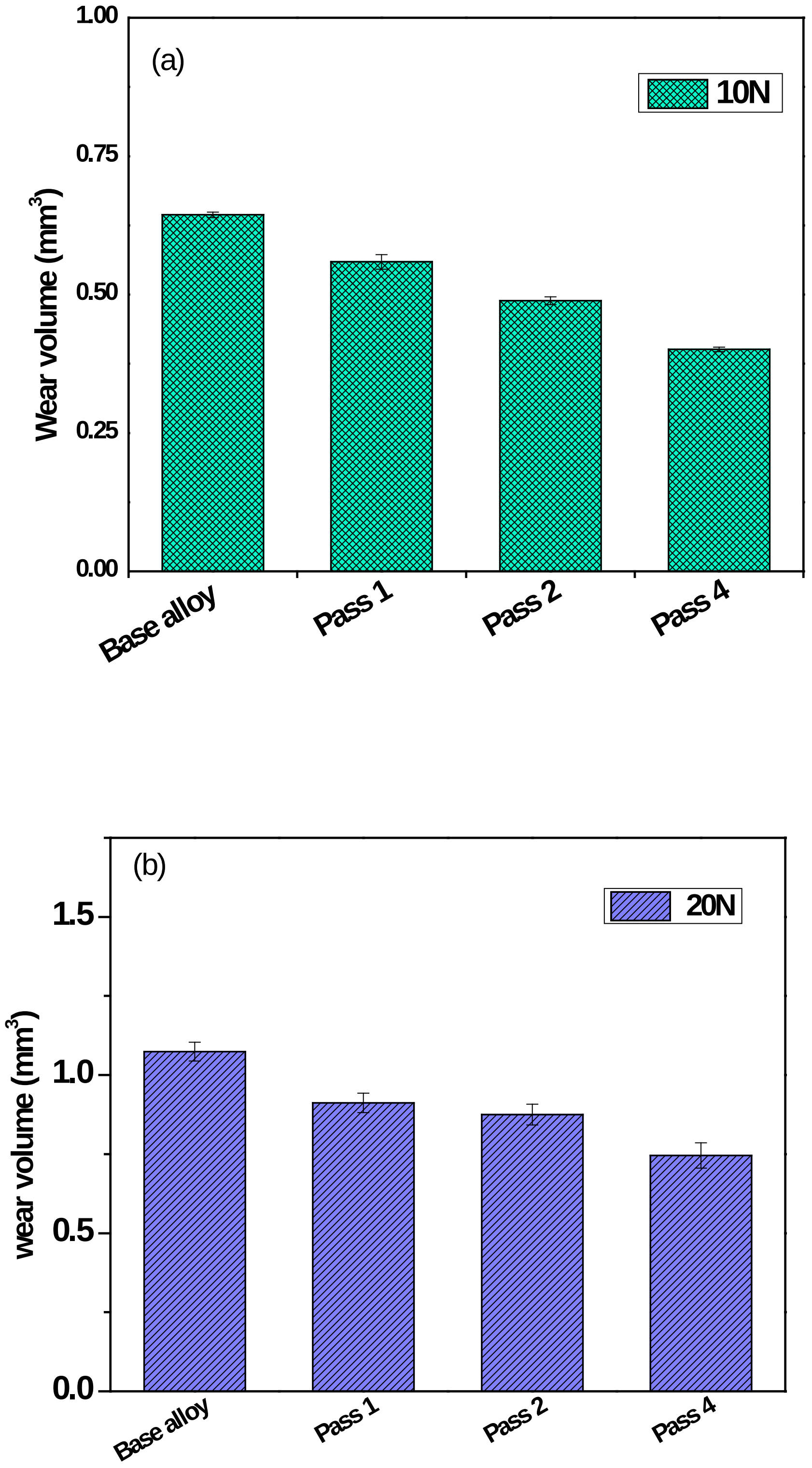

Figure 4.5. Wear volume of MMC and FSPed MMC at (a)10 and (b) 20N load.

difference in wear volume is observed between MMC and 4 pass FSPed MMC. At 10N load, the obtained wear volume is 0.644 to 0.401 mm^3 from MMC to 4 pass FSPed MMC and at 20N load, the obtained wear volume is 1.074 to 0.746 $mm^3$from MMC to 4 pass FSPed MMC particle size reduction of zircon sand and grain refinement in LM13 matrix occurred due to effect of 4 pass which increased the hardness and strength. The increase in hardness and strength of MMC increased the wear resistance against steel ball in sliding wear test therefore less wear volume is obtained in 4 pass MMC. Bauri et al. [34] Zahoor et al.[35] and Mahmoud et al.[36] have reported decrease in wear volume due to enhancement of hardness and strength in aluminium alloy matrix.

CHAPTER 5 CONCLUSION

Microstructural modification zircon sand reinforced LM13 alloy composite was conducted by friction stir processing. The Microstructural modification is correlated with number of friction stir processing passes. The following are main conclusion of the present study:

1. Microstructural modification of composite depends on number of passes. As number of passes increases zircon sand particle size decreases.
2. After four passesfriction stir processing nearly a five times size reduction of zircon sand occurred from 50µm to 10µm.
3. Mechanical properties and tribological properties also depend on number of passes. Strength, hardness and wear resistance increase after four passes of friction stir processing.

CHAPTER 6 SCOPE FOR FUTURE WORK

1. Effect of cooling on microstructural modification of composites can be studied in term of grain refinement.
2. This study can be further extended to study of corrosion behaviour of friction stir processed composites.
3. In microstructural modification of metal matrix, tool wear can be systematically investigated.
4. Effect of process parameters like rotational and transverse speeds on microstructural modification can be investigated.
5. Size reduction of reinforced particles during friction stir processing passes enables the usage of coarser reinforced particles.

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