Preparation and characterization of Al-SiC_p nanocomposites developed by stir casting

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CANDIDATE'S DECLARATION

I hereby declare that the proposed work presented in this dissertation entitled **"Preparation and characterization of Al-SiC$_p$ nanocomposites developed by stir casting"** in partial fulfillment of the requirements for the award of the degree of **Master of Technology** in '**Metallurgical and Materials Engineering'** with specialization in **Industrial Metallurgy**, 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 2015 to May 2016, under the supervision of **Dr. Suhrit Mula**, Assistant professor, Department of Metallurgical and Material Engineering, Indian Institute of Technology Roorkee.

The matter presented in this dissertation has not been submitted anywhere, in any form by me from awarding any degree.

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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

Al-6061 alloy is mostly used for automobile parts because of its good corrosion behavior and high stiffness. However, it has poor wear resistance which limits its wide applications. Metal matrix composite (MMC) in which hard ceramic particles such as SiC is dispersed leads to an improvement in wear resistance and stiffness but in the expense of its ductility. Major problem of making the MMC is the uniform distribution of the ceramic particles. In the present study, an attempt has been made to distribute SiC particles uniformly within the matrix by stirring casting and further modified by friction stir processing (FSP) and achieving enhanced property. FSP is a novel technique and can be useful to distribute SiC particles along with the refinement of matrix grains. Thus, mechanical properties could be improved by 2 ways: due to ceramic reinforcement and grain size refinement. In this work, Al 6061 alloy matrix composites (AMCs) reinforced with 2 wt. % SiC particles of (microsize 20-160 µm) and (nanosize 30-80 nm) was prepared using casting via stir casting technique. The FSP was conducted at a speed of 720rpm using a tool of cylindrical pin in two condition first in normal air cooling with traverse speed of 20 mm/min and second in forced air cooling with fan and traverse speed 30 mm/min to lower its processing temperature to the minimum possible value. The microstructure and mechanical properties (hardness, tensile strength, and wear resistance) were compared for the both micro and nanocomposites, with and without FSP. The characterization was carried out using X-ray diffraction (XRD), optical and scanning electron microscopy. After FSP of the composite, it was observed that mechanical properties was improved due to homogeneous distribution of SiC particles and grain refinement of the alloy matrix. Significant improvement in the ductility was also measured after FSP as compared to that of the cast composite. Nano reinforces ceramic gives better property than micro-reinforced after FSP but at the same time nano reinforce composite gives very inferior property before FSP due to the high amount of porosity and inhomogeneity present in the material due to the large surface area to weight ratio of nano particle, which was verified by Archimedes principal. The influence of porosity had been observed in wear rate also wear rate of nanocomposite is found to be more than that of micro reinforcement, but after FSP the significant removal of defects, porosity and homogeneous distribution the strength, ductility and hardness had been recovered. Which shows the effectiveness of the FSP in nanocomposite.

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Chapter 1 INTRODUCTION

1.1 INTRODUCTION

Aluminum (Al) alloys are showing their prominent role in various applications such as: structural applications, aerospace, military, and transportation industries due to their (1) light weight, (2) high stiffness, and (3) corrosion resistant behaviour [1]. However, it has some drawback which inhibits its application and is due their poor wear resistance, manifested by severe adhesive wear [2] [3]. In comparison with the unreinforced Al alloys, Al matrix composites reinforced with ceramic phases exhibit higher strength and stiffness, improves its tribological characteristics, and increases its workability in high temperature applications [4]. Composite refers to the combination of two or more materials having distinct features in which one is reinforcement, distributed in another matrix phase, which in combine gives the distinguishing property than the parent material. On the basis of physical nature this is classified as: Polymer Matrix Composite, Metal Matrix Composite (MMCs) and ceramic composite [5] [6], but this project is specifically concerned with Al-6061 alloy MMC in Al-MMC the Al-6061 is a matrix phase which serves as a binder for reinforcement phase which usually a ceramics such as SiC, Al_2O_3, TiC but in the current emphasis is only on SiC. Moreover, recent research reveals that the MMCs having Nano-reinforcement are showing superior wear resistance, but these composites lacks in thermal conductivity and ductility [7]. However, there are various processes by which the ductility of the composites can be regained without affecting the strength in that queue a very prominent and efficient processing route FSP has been demonstrated [8].

The Friction Stir Processing (FSP) is a solid state processing technique which was developed in 1999; by Mishra *et al.* It comes after the invention of friction stir welding (FSW) process which has completely changed the joining process, this was invented by Wayne Thomas at The Welding Institute (TWI Ltd.) In 1991[9]. After that, various researches and successful application has been reported and is used vigorously in metal fabrication industries [10].

FSP uses same thermo-mechanical principal as used by FSW to enhance the microstructural characteristics for property enhancement. In starting FSP was used to produce high grain boundary miss-orientation [11], super plastic Al alloy with ultra-fine grain size [12] and was limited to Al and Mg alloy only because FSP becomes easy for those material which possess low yield strength, low hardness and high ductility, but further the successful research has been carried out on the alloy of copper, steel and titanium [13][14]. Furthermore, research has been carried out successfully to produce surface composite on the Al substrate Property enhancement of cast Al alloy [15]. As well as cast MMCs [14], homogenization and property enhancement of powder metallurgy Al alloy [16]. It also eliminates casting defects Since there has been a lot of successful demonstrations are proposed in very short time, which shows that this technique is growing as a very effective means of solid state fabrication/processing [17].

1.2 MECHANISM OF FSP

This processing technique works on a very simple mechanism, it uses rotating non-consumable tool which consists of accurately designed pin and shoulder arrangement as shown in *Figure*1 the pin is inserted into the monolithic work piece such that the shoulder just touches the workpiece and traverses along the straight line as shown in due to this, heating and material flow takes place. Heat is generated due to friction between shoulder and work piece and partly with a pin and shoulder friction which softens the material and the combined action of translation and rotation moves the material from front to back. In that way stirring is done, which leads to the development of refined microstructure in the processed zone. The main mechanism which works behind the development of fine grains [18].

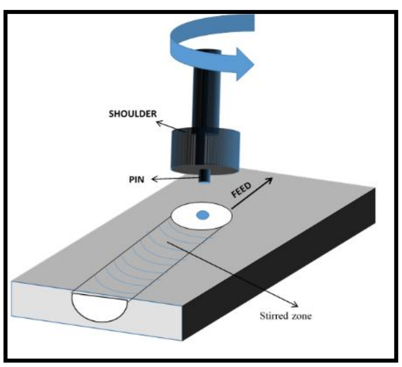

***Figure* 1: Schematic diagram showing Mechanism of FSP.**

Chapter 2	LITERATURE REVIEW

2.1 INTRODUCTION

Al alloys are very promising for structural applications in aerospace, military, and transportation industries due to their light weight, high strength-to-weight ratio, and resistance to corrosion [19]. Heat treatable alloy such as 6000 series has good strength at room temperature and strengthening is due to solid solution strengthening, Hall Petch strengthening and precipitation strengthening [20] but in this the most effective precipitation strengthening is given by the formation of coherent, meta-stable θ″ [3], phase which comes after solutionizing and aging for 1hr at 200℃ beyond this over-aging may starts which reduces the strength and hardness because of formation of θ′ phase which is semi coherent phase, that is why its application is limited at higher temperature because working for few hours at higher temperature results in reduction of strength due to over aging and is beyond recovery it will not regain its strength even if its temperature lowered to room temperature. This is the disadvantage of precipitation hardened alloy over MMCs since the presence of insoluble phase in it can provide strength to the system for longer duration of time, even at high temperature application [21], there is the requirement of composite material. That is the reason why application of these alloys is limited in high temperature applications [23].

The vigorous investigation on MMCs reinforced with nano particle has been carried out worldwide in recent decades and investigation is still going on [24-27], because of their exceptional and suitable application in the field of transportation, defence, structural and mechanical [19]. Reducing the particle size to nano range introduce an exceptional change in mechanical properties and gives other strengthening effect because of significant interaction of dislocation with particle. The main issue with the Nano reinforcement is there improper incorporation of particles into the matrix phase which is due to large surface area and low wettability [28]. Which makes it difficult to produce it by conventional processes. Powder agglomerates forms clusters which inhibit the homogeneous mixing and its effect can be visualized on mechanical properties and strengthening of material. Several alternatives to overcome this issue had been proposed, broadly the production of MMCs can be divided into two groups in-situ [17] and ex-situ [29]. In the former method the reinforcing particle is introduced into the matrix by external means while in later one the reinforcing phase generates due to chemical reaction involved. Several other techniques also had been employed for the production like powder metallurgy, spray forming, stir casting route etc. Out of this stir casting route is most suitable because of its cost effectiveness and easy set up [30].

2.1.1 Recrystallization mechanism

Grain refinement in the stirred zone is governed by dynamic recrystallization which occurred due to intense plastic deformation at elevated temperature. A different mechanism of dynamic recrystallization had been proposed in the Al alloy such as continuous dynamic recrystallization (CDRX), discontinuous dynamic recrystallization (DDRX), geometric dynamic recrystallization (GDRX) [31]. Because of high stacking fault energy present in Al due to their FCC structure DDRX doesn't happen in them, as they recover very fast [32]. DDRX is indicated by the formation of newly formed grains at high angle boundaries. Whereas sub grain growth, lattice rotation associated with sliding, and lattice rotation associated with slip on the other, hand, shows the mechanism of CDRX. Research done by Jata and Semiatin [33]. Because of continuous rotation of the original low angle boundaries during machining, CDRX is an operative dynamic nucleation mechanism during FSW/FSP.

2.1.2 Strengthening mechanism

a) Grain refinement

MMCs the hard phase provides pinning effect of the grain boundary movement that's why the growth process hindered resulting in relatively finer grains than unreinforced one, in the present study the grain is further refined by the FSP process which provides the additional strength of the material, and is governed by Hall-Petch effect which is given by the Equation--(1)

$$\sigma_{Hall-Petch} = \sigma + \frac{Ky}{\sqrt{d}} \qquad \text{-------- (1)}$$

Since finer the grain will be the mayor is the grain boundary and grain boundaries are an obstacle for the dislocation movement resulting in strengthening of material.

b) Orowan strengthening

This strengthening effect caused by the presence of hard particle in the matrix phase, which restricts the dislocation movement. The effect is more pronounced in nano reinforcement rather than micro reinforcement because of the large size of particle and greater inter particle distance. The cutting or bowing mechanism may happen in the material the *Figure* 2 describing this phenomena. It has been established fact that creep resistance, Fatigue properties and lifespan of Al- MMCs improves with increasing percent of reinforcing phase [34] because orowan bowing is necessary for bypassing the dislocation. The Equation--(2) governs the orowan effect [35][36].

$$\Delta\sigma_{OR} = \frac{0.13bG}{d_p\left(\sqrt[3]{\frac{1}{2}v_p}-1\right)} ln\left(\frac{d_p}{2b}\right) \qquad \text{-------- (2)}$$

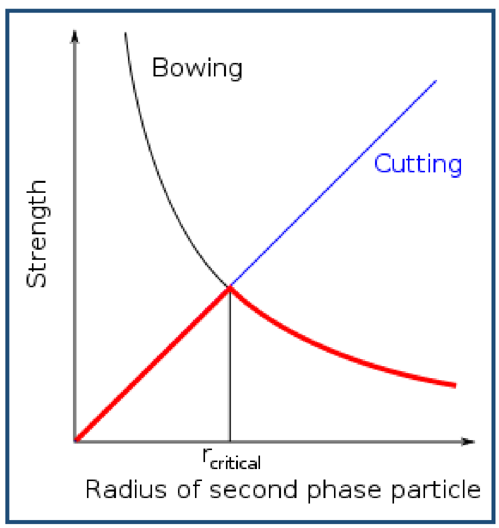

***Figure* 2: Mood of operation of Orowan Mechanism.**

In this *b* is the burgers vector and G is the shear moduluss.

Since current work is performed on 6061 Al-alloy which is a precipitation hard enable alloy as it contains Mg and Si the precipitate forms into the material also provide strength by orowan strengthening mechanism.

2.1.3 Other strengthening

a) Load transfer effect

In the composite presence of reinforcing phase, which is hard and stiff takes a part of load into it providing strength to the base material. To calculate the contribution in strengthening Nardone and Prewo [20], proposed a modified shear lag model can be used. So the equation for the load transfer to the material can be given by Equation (3)

$$\Delta\sigma = \sigma_{Matrix} \times V_{particles}\left[\frac{(l+t)A}{4l}\right] \qquad \text{-------- (3)}$$

Where “l” and “t” are the lengths along the load and perpendicular to the load, A is the cross sectional area $V_{particles}$ is the volume fraction of the particle.

b) Coefficient of thermal expansion mismatch

Due to the difference in coefficient of thermal expansion there developed a strain field around the particle or there is a very high dislocation density around particle which, when interact with dislocation hinders its movement resulting in greater strength of material [37].

However the contributions of different strengthening mechanism are different and varied according to particle size and volume fraction it can be understood from the *Figure* 3 a research done on Al/Al_2O_3 by Casati et al. [38]. Reveals the effect of strengthening effect and their contribution.

As can be seen from the above *Figure* 3 that there is very high contribution of orowan strengthening and coefficient of thermal expansion mismatch when particle size is very small and the contribution of load bearing phase effect is least.

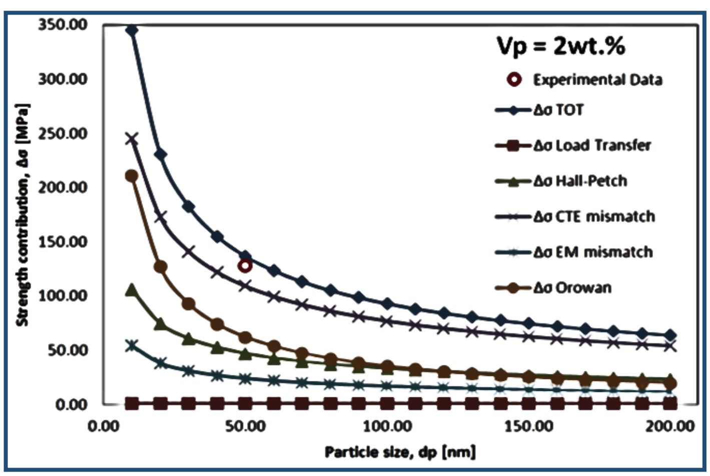

***Figure* 3: Effect of variation of various strengthening mechanism with Particle size.**

2.2 PRESENT STATUS OF THE WORK

A variety of research is currently going on in this field by taking various grades of alloys and reinforcing phase like SiC [39], Al_2O_3 [24], TiB_2 [40] etc. In different shapes and sizes and fabrication of MMCs using these two components are done in various processing routes such as stir casting [41], spark plasma [42], squeeze casting [43], powder metallurgy [44] and ultrasonic spray forming. There are various challenges associated with the type and size of particles as well as with fabrication route. So this section will discuss about the current and past working/research status, which are going on worldwide in the perspective of Al MMCs their processing technique challenges and opportunity in the various fields such as aerospace, automobiles and metal fabrication [19].

The research done by Mohsen Hossein-Zadeh et.al [45]; Semnan University Iran ,in which they have taken Al_2O_3 powder which was milled in a high-energy ball mill for 20 hr, at the same time 1 wt. % of Al_2O_3 was introduced into the melt. Various mechanical properties such as tensile test and wear and microstructural characterization of this fabricated composite had been carried out. A nano particle in the matrix had been revealed by peak broadening in X-Ray diffraction (XRD). Moreover the distribution of reinforcing phase was quite inhomogeneous. Because of the presence of nano hard particle grain size reduces as it acts as a pinning effect to the growing grains, it act as a heterogeneous nucleation site for the grain nucleation and due to the refinement of the grain hardness, yield strength and wear resistance of Al–Al_2O_3 has been raised significantly. Furthermore, there is always a well-known problem for metallurgist which is optimization of strength and ductility as they are mutually exclusive to each other was present in this case, but the research done by Gaohui *Wu et al.* [46], they show that adding distinctive elements (Sc, Zr) refining the heterogeneous nucleation cites to nano level and due to this adding high volume fraction of reinforcement during the fabrication had been achieved with adequate strength and ductility and this process represents a very important tool for achieving outstanding combination of strength and ductility in high volume fraction SiCp/Al composites. Even further research has been carried out on a cast A356 alloy reinforced with nano-SiC Ali Mazahery et al. [27], and with Al_2O_3. Mazahery *et al.* [24], P. Rama Murty Raju *et al.* [47]. nanocomposites and also on Al/A_2O_6. Tahamtan *et al*. [48], and Al/2024 Hai Su *et al*. [49], with Al_2O_3 using stir casting routes which shows almost uniform distribution of reinforcing phase enhancement in ultimate tensile strength and ductility exhibited by nano- Al_2O_3 reinforced Al and is attributed by uniform distribution and grain refinement. Although stir casting is widely used due to its cost effectiveness and easy fabrication method S.A. Sajjadi *et al*. [50] shows in his work in which they compare composites in which one is fabricated by stir casting and another by compo-casting processes, they found that using compo-

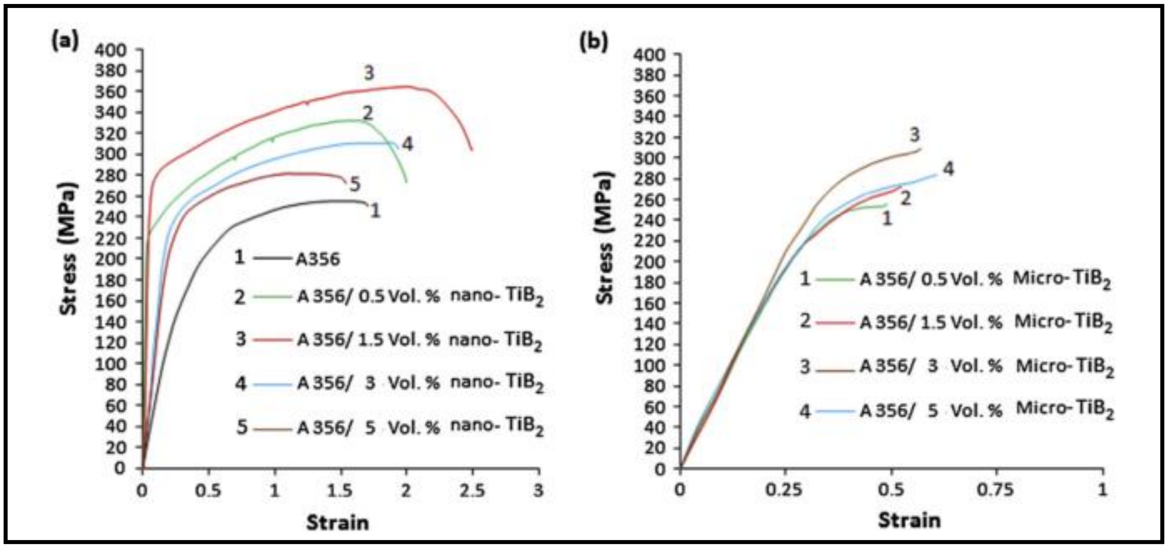

***Figure* 4: The tensile curve of (a) nano and (b) microcomposite at 800℃**

casting process gives best mechanical properties in turn. There is an effect of casting temperature on mechanical properties which was demonstrated by M. Karbalaei Akbari *et al*. [40]. In which TiB2 particle as a reinforcement in A356 Al alloy using casting route they conclude that the effect of casting temperature on its increment reduces the mechanical property of material as more porosity likely to form at high temperature. Their stress-strain relationship is given in the *Figure* 4.

Fracture surface reveals the particle segregation at grain boundaries along with fine dimples and nano particle in it. The results show an evidence of Si cracking and de-bonding, which is one of the main characteristics of fracture mechanism of Al–Si alloys which was detected on the fracture surface of composite matrix, which is reinforced with TiB2 Nano and micro particles *Figure* 5

Moreover, for the improvement of mechanical properties hybridization technique is implemented using stir casting and ultrasonic vibration impairment during the introduction

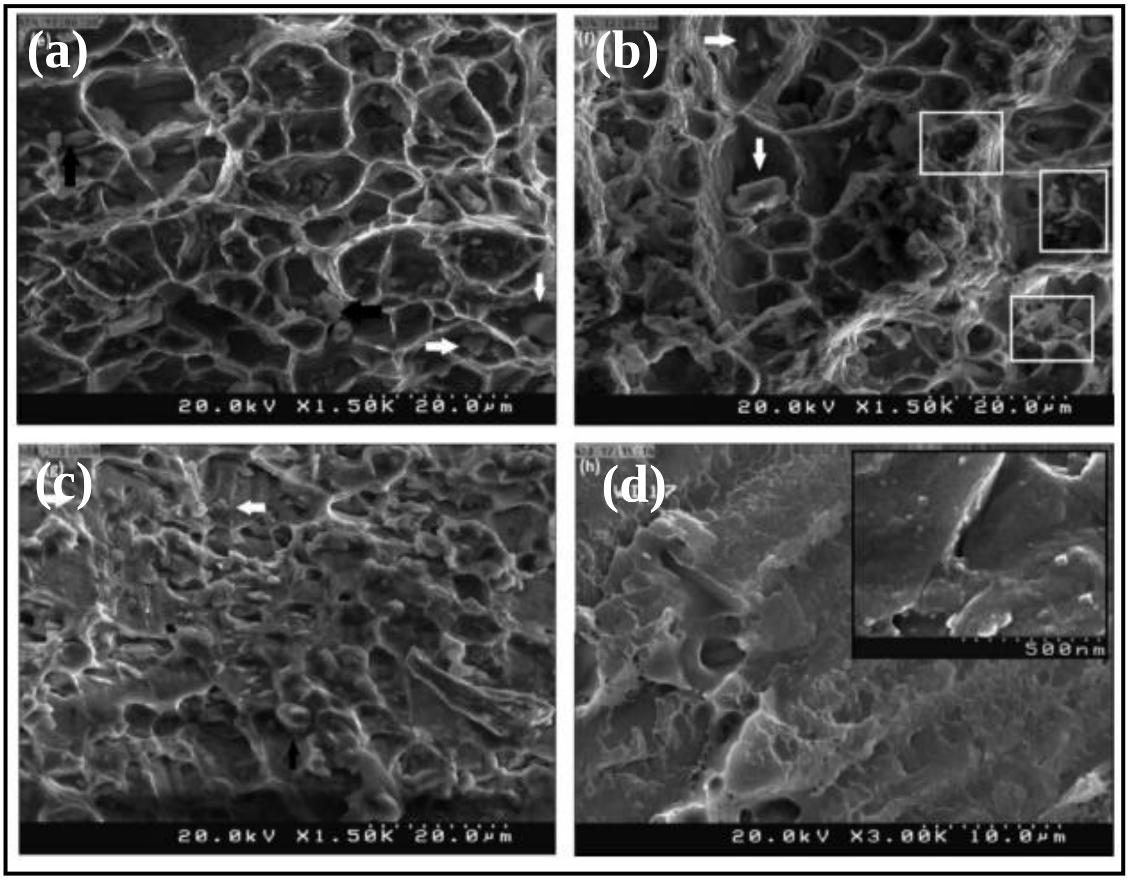

***Figure* 5: The fracture surface (a) shrinkage void (b) fine dimples with smooth surface in nanocomposite (c) ultrafine dimples with rough surface in nanocomposite (d) imbedded nano particles in dimples.**

of reinforcing phase S. Tahamtan *et al.* [51] proposed a technique in which they enhance tensile properties of a network-structure Al-A206matrix composite by hybridizing microsized composites with Al_2O_3 Nano particulates, and furthermore this hybridizing technique had also been used by Weiping Chen *et al.* [46] By squeeze casting route in which (SiCp-Ti)/7075 Al hybrid composites with added minor Ti particles were fabricated by squeeze casting. This way the feature comes out on the turn in which the ductility was unaffected and significant improvement of strength compared with Ti particle one. This may provide a new method of fabricating composites with excellent mechanical properties. More on squeeze casting technique, Al–Si base composites reinforced with different mixtures of Ni and nano-Al_2O_3 particles have been fabricated by squeeze casting and their metallurgical and mechanical characterization has been done by Hashem *et al.* [43]. In which they found that the ductility of the fabricated composites is significantly improved by increasing the added Ni particles. The composite material, reinforced with 5 wt. % Ni and 2 wt. % nano-Al_2O_3 particles showed superior ultimate tensile strength and good ductility compared with any other added particles in their investigation. But in various cases there has been a decrease in strength when working under elevated temperature, but the research done on nano-SiCp/Al2014composites by Long-Jiang Zhang *et al.* [52], using stir casting combined with hot extrusion shows high strength and good ductility even at elevated temperature. Besides the major reinforcing phase like SiC and Al_2O_3composite had also been fabricated by nano MgO particles A. Ansary Yar *et al.* [41]. In which, Al alloy (A356) matrix composites reinforced with nanoparticle of nanoparticles were fabricated via stir casting method. Hardness and compression tests were carried out in order to identify mechanical properties. The results reveal that the composites containing 1.5 vol. % reinforcement particle fabricated at 850°C has homogenous microstructure as well as improved mechanical properties.

The major challenge in stir casting is wettability of reinforcing phase where poor wettability hinders the fabrication of good casting by agglomeration and inhomogeneous distribution. One of the remedy for this issue is pre-coating of particle is a good way of increasing irritability. In the study by M. Heydarzadeh Sohi [53], on AA8011and Al_2O_3, a mixture obtained by mechanical alloying of 40 wt. % Al and 60 wt. % of Al_2O_3 shows good distribution and mechanical property.

Beside stir casting ultrasonic cavitation method used by S. Gopalakannan, and T. Senthilvelan [54] is also in great use which shows uniform distribution of, SiC nanoparticles in the MMC. Moreover spark plasma sintering method was also applied for the purpose of fabricating composite the work presented by D. Garbiec *et al*. [42] Shows the fabrication and characterization of Al–Al_2O_3 composite materials taking 5%, 10%, 15% and 20% volume fraction of Al_2O_3. The spark plasma sintering method was applied for the purpose of fabricating these materials. The obtained Al–Al_2O_3 composites were characterized with porosity from 1.27% to 5.07%. It was proven that as vol. Fraction of reinforcing phase increase, its density, hardness, and compression as well as tensile strength also increases.

Out of all above discussed method homogeneous distribution of particle is very difficult and require lots of prior effort to achieving sound casting with good wettability and homogenization. But the FSP comes with a very good and easy method of post fabrication and modification method. Various attempts had been done in improvising the property of micro and nano MMCs.

For achieving homogeneous distribution in the matrix phase refinement of grains and dendrites and to improve metallurgical and mechanical properties, various research has been proposed to demonstrate the effect of FSP on the Al/alloy-SiCp composite.

2.3 CASTING OF MMCs

The suitable and economical method for composite fabrication is stir casting route. The main effect was observed in the mould preheat temperature as the temperature increases the porosity level goes down, whereas but inhomogeneous mixing dominates on the counterpart. From this literature is that the mould temperature should be optimized in order to get minimum porosity and agglomeration and SL route should be practiced while casting [55].

Previously a research has been carried out to find appropriate ways of manufacturing composite by minimizing the flaws produced in compo-casting Composites based on two Al alloys (A536 and 6061) reinforced with 10% or 20% volume fraction of SiC particles were produced by gravity casting and a novel two-step mixing method was applied successfully to improve the wettability and distribution of the particles [39]. Property of the composite can be easily altered by varying composition of reinforcing phase and as well size of the reinforcing phase incorporation of Nano particle leads to the improvement in various properties only being the problem is.

A research has been carried taking various reinforcing phase B4C, SiC and Al_2O_3 (0–20 vol. %) and properties, interfacial reactions were analyzed deeply[56]. The stir-casting route followed by hot extrusion had been adopted. Clear interfacial reaction product/layer was found at Al–SiC interface for composites held for a relatively long processing time (>30 min). No reaction product was observed at Al–B_4C and Al– Al_2O_3 two secondary phases (alumina and another phase containing Al, boron and carbon) were found in the Al matrix away from the interface in Al– B_4C composites [57].

For the last two decades composite material is showing their benchmarks in various fields of engineering, which attracts researcher to produce various composite by easy and economical route. Manufacturing of Al/Al-alloy-SiC particle can be done in various ways, such as stir casting, powder metallurgy and spray casting routes.

Shriyash S. Shinde *et al.* [58], Demonstrated how stir casting route is an economical and effective way to produce composite in which a dispersed phase (ceramic particles, short fibres) is mixed with a molten matrix metal by means of mechanical stirring. The liquid composite material is then cast by conventional casting methods. In preparing MMCs by stir casting method some of the factors that need considerable attention are as follows:-

- To achieve uniform distribution of the reinforcement material.
- To achieve wettability between the two main substances.
- To minimize porosity in the cast MMCs.

2.4 FRICTION STIR PROCESSING (FSP)

2.4.1 Microstructural and mechanical properties in FSP

This section thoroughly discusses about the parameters or variables and their effect on the microstructure and other mechanical properties in processed zone.

Grain size is influenced by the operating temperature of FSP as the temperature of machining increases grain becomes coarse. These variables in the FSP are optimized in such a way to obtain required microstructure such as tool rotation speed increases the heat

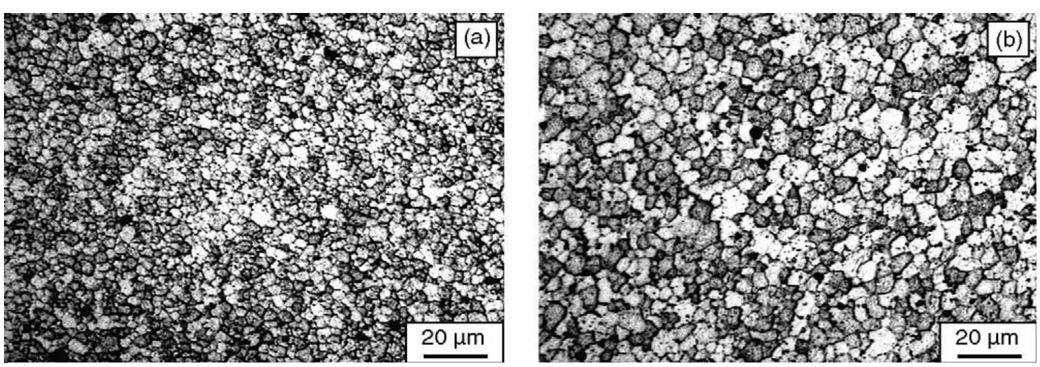

***Figure* 6: Effect of FSP parameters on nugget grain size in FSP 7075Al-T7651 at processing parameter of: (a) 350 rpm, 152 mm/min and (b) 400 rpm, 102 mm/min.**

generation is more and the consequence of this relatively coarser grains are formed. Which leads to the lesser strength and high ductility. There is an another effect of the tool traverse speed as the tool traverse speed increases less heat is generated and finer grains are produced. In combination the effect of grain size and correspondingly strength and ductility is influenced by the ratios of the rotation speed and tool traverse speed as this ratio increases coarser grains are formed as seen in *Figure* 6.

In stirred zone the temp distribution from advancing side to the retreating side changes and it also changes from top to bottom this thing also affects the grain size throughout the nugget zone *Figure* 7.

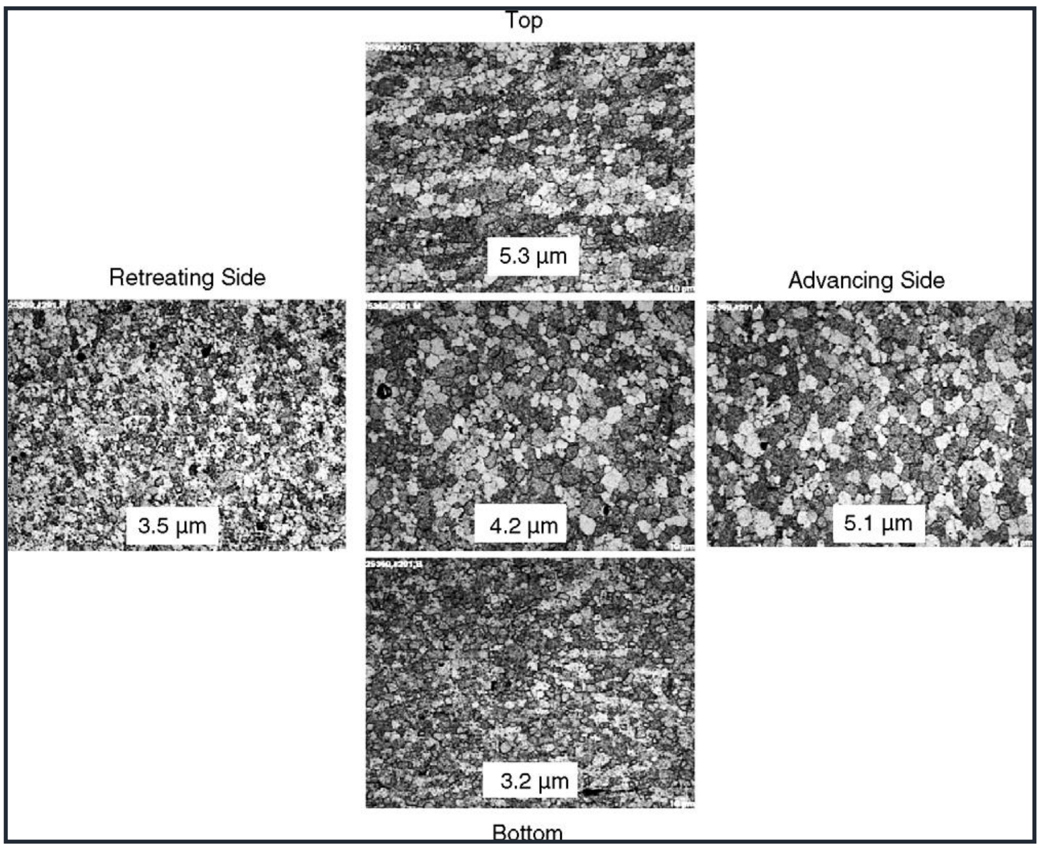

***Figure* 7: Showing top to bottom and retreating side to advancing side, the variation of grain size in stirred zone.**

The pin and shoulder: diameter and height are also the influencing parameter in grain growth and recrystallization a large diameter shoulder generates more heat and lesser diameter would not able to generate sufficient heat which causes difficulty during stirring so the parameters should be optimized. The shape of the pin influences the flow of the material and as an overall result influences mixing and mechanical properties of the processed zone.

2.4.2 Advantages of FSP

FSP is showing there prominent effect in the solid-state processing technique. In short duration of time, few successful and effective applications of FSP have been carried out for localized microstructural modification for specific property enhancement. For example, high-strain rate super-plasticity was obtained in commercial 7075Al alloy by FSP. Furthermore, FSP technique has been used to produce surface composite on Al substrate, homogenization of powder metallurgy Al alloy, microstructural modification of MMCs and property enhancement in cast Al alloys furthermore various benefits are tabulated in *Table* 1.

***Table* 1: Benefits of FSP**

Metallurgical benefits	Environmental benefits	Energy benefits
• Solid state process • Good dimensional stability • No loss of alloying elements • Excellent metallurgical properties. • Fine microstructure	• No use of harmful gases • Surface preparation not required • Material saving	• Improved materials use allows reduction in weight • Decreased fuel consumption due to high strength to weight ratio. • Aircraft, automotive and structural applications

2.5 OBSERVATION OF THE LITERATURE REVIEW

Thus, from the above discussed literatures on various topics such as composite casting technique secondary machining process effects and FSP effects on various substrates and the influence of parameters on the properties has been discussed. So the basic highlights of the entire discussion are as follows.

- ❖ Moulds should be preheated to avoid porosity in the cast composite, presence of Mg reduces formation of porosity and oxides, and extra care should be given on the temperature and the vortex formation during stirring ensures proper distribution which also influenced by the time and temperature of stirring.

- Two step mixing techniques using SL-route is an adaptable way of casting composites
- FSP is found to be very good processing technique for grain refinement and homogeneous distribution of reinforcing phase as well as solute which in turns gives better strength and ductility.

2.6 AIM AND OBJECTIVE

Metal matrix nanocomposites shows very outstanding property. However, manufacturing of metal matrix nanocomposites and proper mixing of reinforcing phase by conventional stir casting route is very tedious job. So, this project has been carried out to find an alternate way for modification of stir cast Al6061-SiC nanocomposites by FSP.

As per above discussed background, the objectives of the present work are envisaged as:

- **Preparation** of nano- microcomposite having Al-6061 as a matrix phase and SiC as reinforcement by stir casting and then reinforced SiC particles will be homogeneously dispersed into the matrix using FSP.
- **Characterization** of the composites by optical microscopy, SEM and XRD analysis.
- **Comparison** of mechanical properties of the nanocomposite to that of microcomposite having same wt.% of reinforcement.
- **Analysis** of the strengthening mechanisms of the composite on the basis of microstructural evolution and mechanical properties and setting up of structure-property relationship for the best combination of strength and ductility for practical applications.

This chapter deals with the thorough study of the experimental procedures carried out during the investigation. The microstructural evolution was characterized by optical microscopy, XRD and electron microscopy. The fractography after the tensile test had been carried out by scanning electron microscopy to correlate the ductility obtained by tensile tests as well as SEM of wear surface had been taken to analysis wear behavior pattern in composite. The wear behavior of the material has been carried out using a pin on disc. Scanning electron microscopy (SEM) was used for fracture analysis after the tensile test. The mechanical properties were evaluated by tensile testing, Vickers hardness measurements and wear properties. The detail of each process is described in the following divisions and the experiment flow chart has been described in *Figure* 8

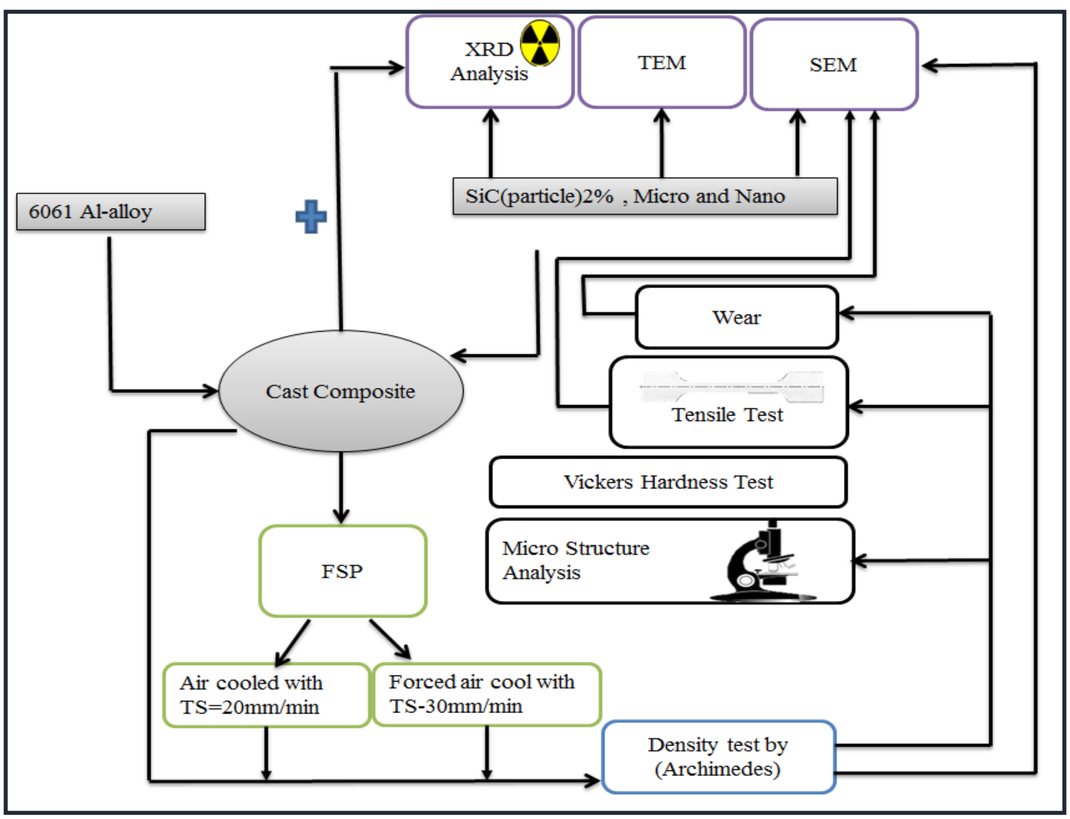

***Figure* 8: Flow diagram**

3.1 MATERIAL SELECTION

Commercial Al 6061 alloy (composition given in *Table* 2) plates supplied by M/s. Hindalco Industries, India were used for the present study.

***Table* 2: Composition of 6061 Al-alloy**

Elements	Cu	Mn	Si	Mg	Zn	Fe	Al
Composition (wt. %)	0.1	0.1	0.6	0.9	0.12	0.5	Bal

3.2 BALL MILLING

Ball milling had been done using Planetary Mono Mill Pulverisette 6 classic line machine *Figure* 9 To achieve nano-SiC .initially taken SiC of Alfa Aesar brand of particle size having 20-160µm and was put in a tungsten carbide jar in the ball to weight ratio of 15:1 using tungsten carbide ball. Wet milling had been performed and milling was carried out for 45 hrs and later on particles were characterized by TEM for particle size and Crystallinity.

***Figure* 9: Planetary Ball Mill**

3.3 CASTING SET UP

Casting had been done in open muffle furnace *Figure* 10(a) by heating the alloy at 700℃ in a graphite crucible *Figure* 10(b) later on after melting SiC particle which was preheated at 300℃ for 1 hour had been introduce into the molten metal at the rate of 2grams/sec through a Cu pipe and simultaneously stir the system using stirrer fitted on drilling machine at 200 rpm creating vortex inside the molten metal for 2 min later on the molten metal had been poured in cast iron mould which was preheated at 150℃ of dimension having 55mmX55mmX120mm than left the simple to cool naturally and finally got billet of dimension having 50mmX50mmX110mm. Which was then cut into 4pices using bend hacksaw of dimension 100X50X10 and finally finished the surface by shaper. The casting was done to achieve 3 billets of the above mentioned dimensions (a) Al-6061+SiC (micro-reinforced) (b) Al-6061+SiC (nano-reinforced) later on after surface finishing by shaper the sample was carried out for FSP.

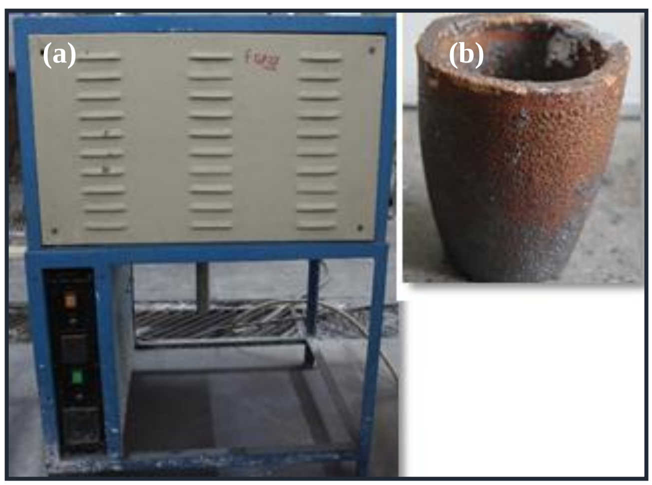

***Figure* 10: Casting Equipment (a) muffle furnace (b) crucible**

3.4 FSP SET UP

A milling machine arrangement had been modified to work accordingly as an FSP *Figure* 11 (a) universal adjustable fixture was placed over the bed of the machine and FSP tool *Figure* 11 (c) of dimension listed in the table. The FSP machines provide 3 different rotational speeds 720rpm, 1200rpm, 1500rpm which is governed by belt and pulley arrangement and wide range of traverse speed which is governed by hydraulic mechanism.

An Experiment was conducted with 2 parameters: in 1st tool traverse speed is 20mm/min with 720 rpm of traverse speed and natural cooling and in 2nd tool traverse speed is kept 30mm/min with 720 rpm along with air cooling while machining so as to minimize the machining temperature in hope to achieve better mechanical properties. Two pass nearby near has been taken for nano and microcomposites *Figure* 11b. For each sample of all two composite material viz. a) Al-6061+SiC (micro-reinforced) (b) Al-6061+SiC (Nano-reinforced). One had been used for tensile specimen sample and another had been used for microstructural and wear examination.

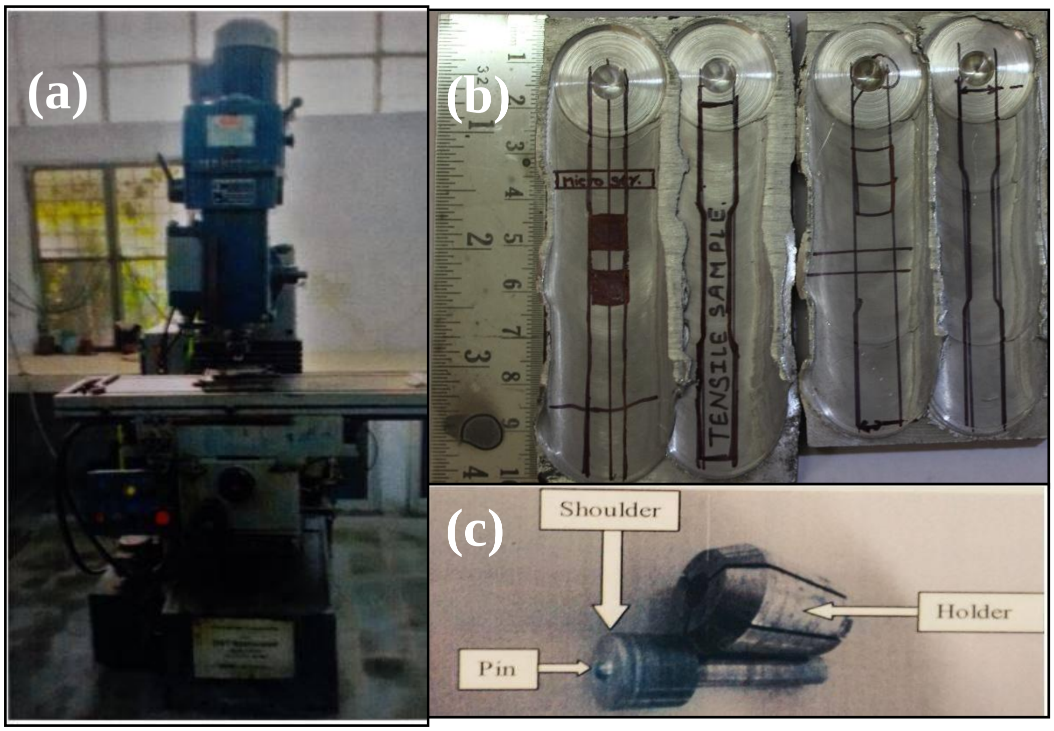

***Figure* 11: (a) FSP machine (b) composite sample after FSP (c) FSP tool and tool holder**

3.5 XRD

The evaluation of the microstructure had been carried out in a Rigaku Smart Lab *Figure* 12 X-Ray Diffractometer using Cu-Kα radiation. The XRD pattern was recorded from angle

***Figure* 12: X-Ray Diffractometer**

20˚ through 90˚. A scan rate of 0.5˚/min was maintained during the collection of the data. The X-Pert High score Pro software was used to carry out the background subtraction and Cu Kα2 peak stripping and analysis of present phase.

3.6 TEM

TEM of powder sample had been done using machine *Figure* 13 sample was prepared earlier by ultra-sonication of the powder particle for 45 min later on a drop casting technique.

***Figure* 13: TEM machine**

3.7 MICROSTRUCTURAL CHARACTERIZATION

3.7.1 Optical microscopy

Microstructural characterization to observe grain refinement and particle segregation had been carried out using Carl Zeiss Optical microscopy instrument (*Figure* 11). But before taking snaps of optical microscopy image the sample had been thoroughly mirror polished and etched and the micro structure had been taken from sample having following parameters.

- 1st type FSP had been carried out using following parameter (1) Tool traverse speed is 20mm/min with (2)720 rpm of traverse speed and (3) air cooling.
- 2nd type FSP had been carried out using the following parameters: tool traverse speed is (1) 30mm/min with (2) 720 rpm of traverse speed and (3) air cooling.

***Figure* 14: Carl Zeiss optical microscope**

Sample preparation

A sample was taken out from all three cast and also from the FSP^{ed} one. The FSP sample was taken out from a transverse section of the processing length. Initially sample was polished against different grit size paper of 150→300→800→1500→2000 after that mechanical cloth polishing had been done.

Mechanical cloth polishing

Cloth polishing was done in two steps i.e. rough and fine polishing. A variable speed wheel is preferred for polishing. The rough polishing was done with MgO. A teaspoon full of abrasive is applied at the Centre of cloth, moistened with distilled water then transformed into a paste. MgO solution in distilled water was used for final polishing on fine velvet cloth. After final polishing the samples were thoroughly cleaned and etched with Keller's reagent having composition listed in *Table* 3. For 15 seconds. Microstructural characterization was carried out using optical microscopy (Model: Leica DMI 200) (*Figure* 11). Under polarized light.

***Table* 3: Composition of Keller's reagent**

Keller's	HNO_3	HCl	HF	H_2O
Composition %	2.5	1.5	1	Rest

3.7.2 Scanning Electron Microscopy (SEM)

Fine grains at stirred zone, segregation of reinforcing particles, wear surface analysis and the failure mode during tensile tests were evaluated by the fractography analysis of the fractured surface using ZEISS 51-ADD0048 scanning electron microscope and FESEM. Scanning was done at appropriate accelerating voltages for the best possible resolution. SEM of stirred zone, fracture surface and wear surface had been taken for analysis.

3.8 EVALUATION OF MECHANICAL PROPERTIES

The mechanical properties were evaluated by tensile testing, Vickers hardness measurements, and wear test.

3.8.1 Vickers hardness measurement

Vickers hardness testing was performed on FIE–VM50 (*Figure* 1) to determine hardness values of all the specimens with a load of 5 kg for a dwell time of 15 s. Prior to hardness testing, the samples were well polished and there should not be any scratches and stains. During hardness measurement, minimum 5 readings were taken.

***Figure* 15: Vickers hardness tester**

3.8.2 Tensile Testing

Tensile test was conducted at room temperature on standard size specimen's *Figure* 16 of 25 mm gauge length using H25K-S universal testing machine at a constant strain rate of 1.66×10^{-2} mm/s to calculate the tensile strength, yield strength and ductility of the specimens.

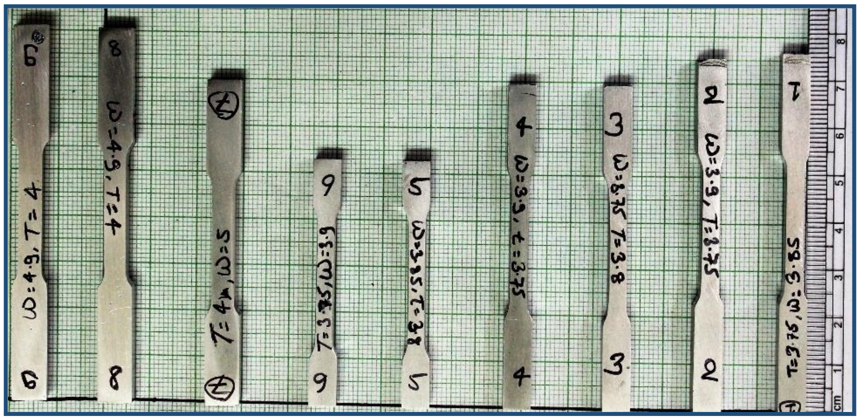

***Figure* 16: Different nano-reinforced (1, 2, 3, 4), micro-reinforced (6, 7, 8) and 6061-alloy (5, 6) tensile sample.**

3.8.3 Wear testing

The test had been done on a pin on disc arrangement, using a square sample of size (6mmX6mmX6mm) from stirred zone of a FSPed sample and cast samples were taken (list of the sample done for a wear testing table.) And perfectly glued on a stub of 6mm diameter, which was then fixed to the tool holder arrangement and the test had been carried out for 20 minutes and coefficient of friction and wear rate had been calculated.

Details of the wear test conditions used in this study

Pin material Al 6061 nano and cast composite with and without FSP of 4mmX4mmX6mm, Stub *Figure* 17 to fix specimen.

- Disc material EN36 steel with a hardness of 65 HRc.
- Sliding speeds: 1mm/Sec.
- Normal load: 10N, 20N.
- Sliding distance: 1200 m.

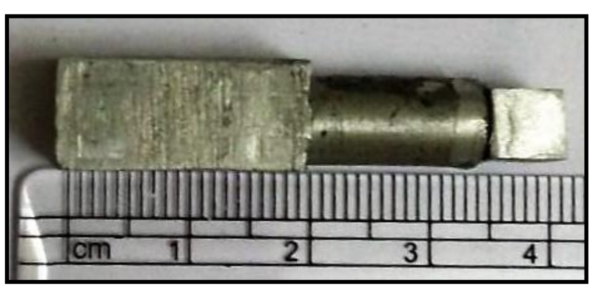

***Figure* 17: Showing stub and wear sample**

Chapter 4 RESULTS AND DISCUSSION

4.1 MICROSTRUCTURAL INVESTIGATION

4.1.1 SiC Powder characterization

Before the incorporation of nano and micro-particle into the mould SEM had been performed on micro SiC and TEM on nano-SiC to know its shape and size. During analysis of SEM (*Figure* 18(a) and TEM (*Figure* 18(b) it was found that the size of the micro SiC was 20-160 µm and nano-SiC was 30-80 nm and crystallinity was maintained after analyzed.

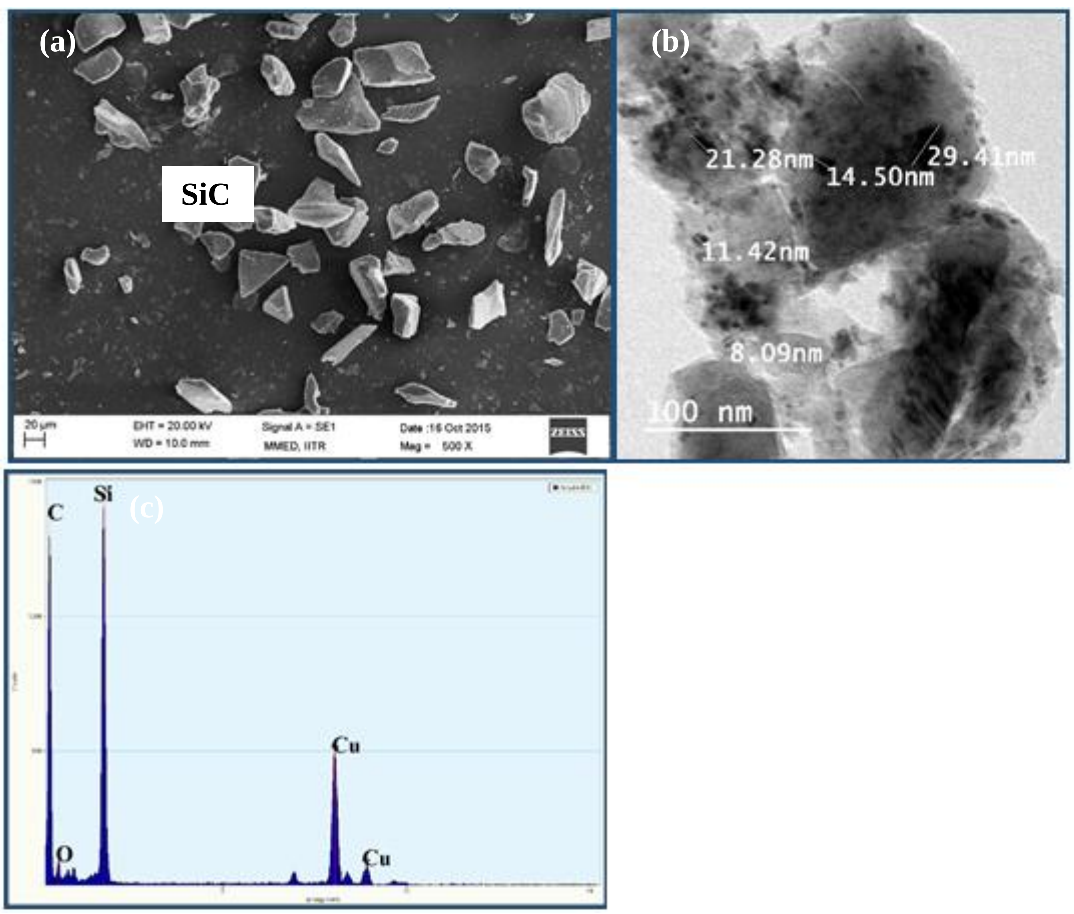

Figure: 18 (a) SEM image of SiC micro size particle (b) TEM image of Nanosize particle (c) EDx Nanosize particle showing peaks of C, Si, and O

4.1.2 Characterization of cast composites

Microstructural investigation of cast sample of micro and nano-composites reveals that, porosity was present after casting, during investigation, it was found that the amount of porosity was more in the nano as compared to that of micro-reinforcement this was due to high surface area and low wettability of nano particles. Later it was confirmed by performing the Archimedes buoyancy test. The *Figure* 19 shows the porosity presented by the black arrow (A) nano and (B) micro-reinforced composite, an agglomeration of SiC has also been shown in *Figure* 19 with blue arrow and SiC particles with yellow arrow. In cast material, even after stir casting lots of agglomeration of SiC was present. The FE-SEM image also show that coarse grains are present on the order of 150-200µm.

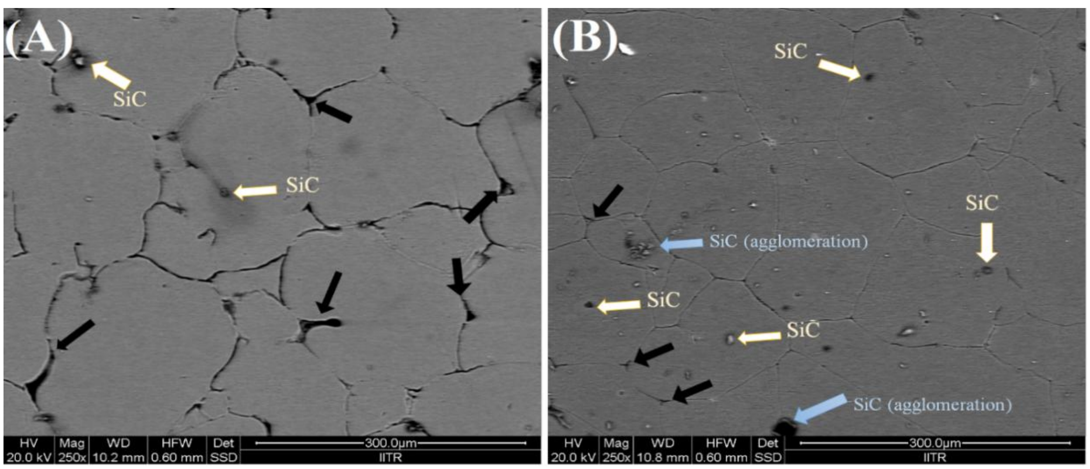

***Figure* 19: SEM image showing agglomeration, porosity in Cast (A) nanocomposite (B) microcomposite.**

4.1.3 Characterization of FSPed composites

Optical microscopy

After FSP there is a significant reduction in grain size in both the castings in stirred zone, because of dynamic recrystallization comparing the two different parameters of FSP the one with forced air cooling gives better grain refinement which is shown in *Figure* 2 (b), this can be attributed because of reduced processing temperature during FSP the *Figure* 20 shows the nanocomposites after FSP (a) with air cooling and 20 mm/Sec traverse speed and (b) with forced air cooling and 30 mm/Sec.

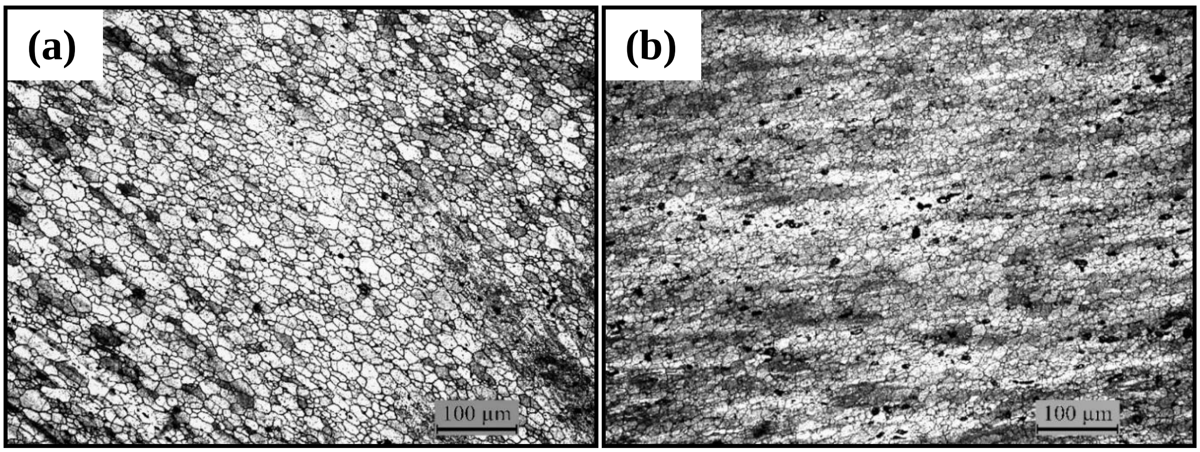

***Figure* 20: Optical Micrograph showing variation in grain size with changing processing parameter in FSP and SiC particles (in nanocomposites) (a) with traverse speed 20mm/min (b) traverse speed 30mm/min+air cooling.**

If we compare nanocomposites to that of micro, keeping the parameter constant Nanocomposite gives finer grain than microcomposite which is attributed by the pinning effect of nano particles during grain growth In nanocomposites more grain refinement had taken place after FSP has been shown in *Figure* 21 (a, c) represents gasped micro-reinforced composite with traverse speed 20mm/min and (b, d) represents gasped nano-reinforced composite with composite with traverse speed 20mm/min. From the above microstructures it is clear that the grain refinement is taking place after FSP which leads to the improvement in mechanical property after FSP the homogeneous dispersion of particle has taken place as well as a huge reduction in porosity in both nano and microcomposites. In starting cast nanocomposites shows more defects and inferior microstructure than micro-reinforced composite but after FSP nano is showing distinguishing property which indicates the effectiveness of the FSP with cast nanocomposites.

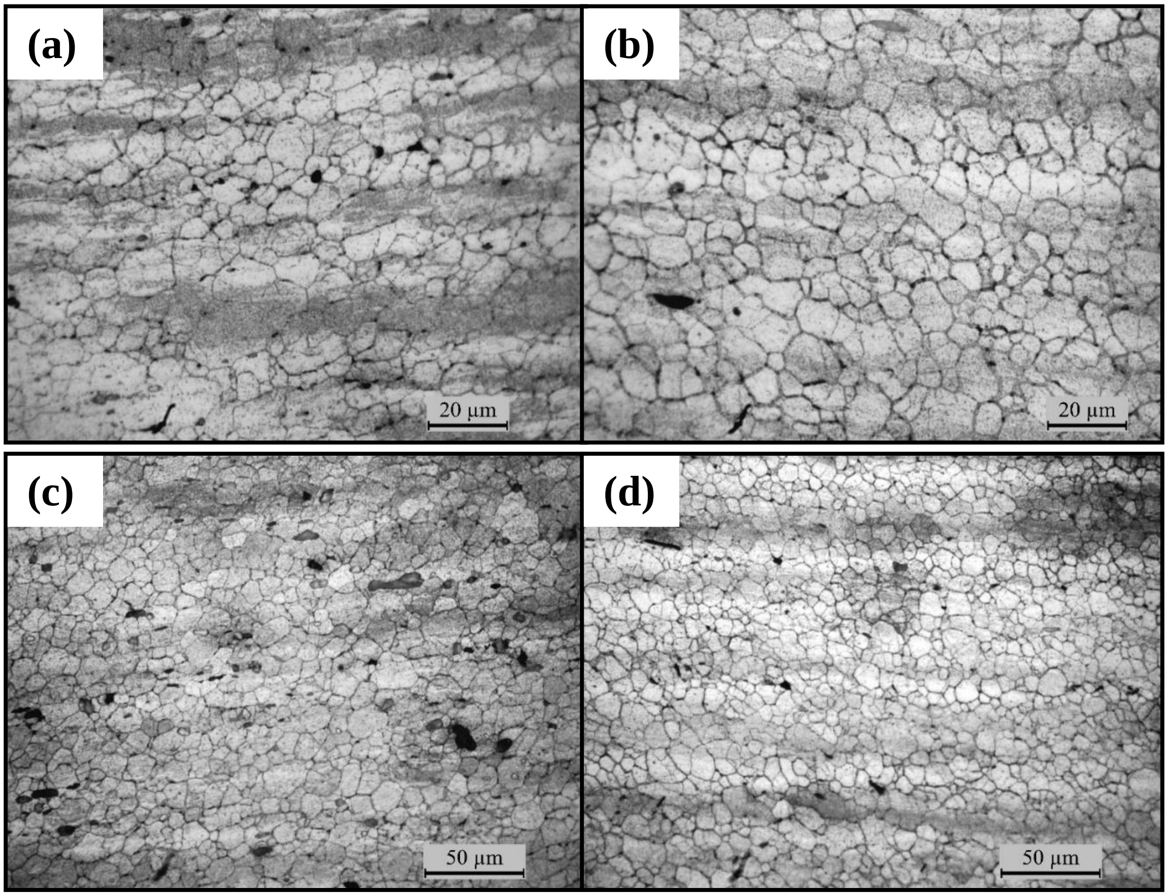

***Figure* 21: optical micrograph showing stirred zone (a, c) microcomposite and (b, d) Nanocomposite, in traverse speed 20 mm/min**

XRD

XRD of nanocomposite sample was carried out for investigation of the presence of SiC nano particle in the nanocomposites to confirm the proper stirring and mixing during casting. Presence of SiC peak in the XRD pattern (*Figure* 22) confirms the incorporation of reinforcement in the matrix of 6061. Plane (101) and (104) of SiC_p peaks had been identified during investigation using X-pert high score.

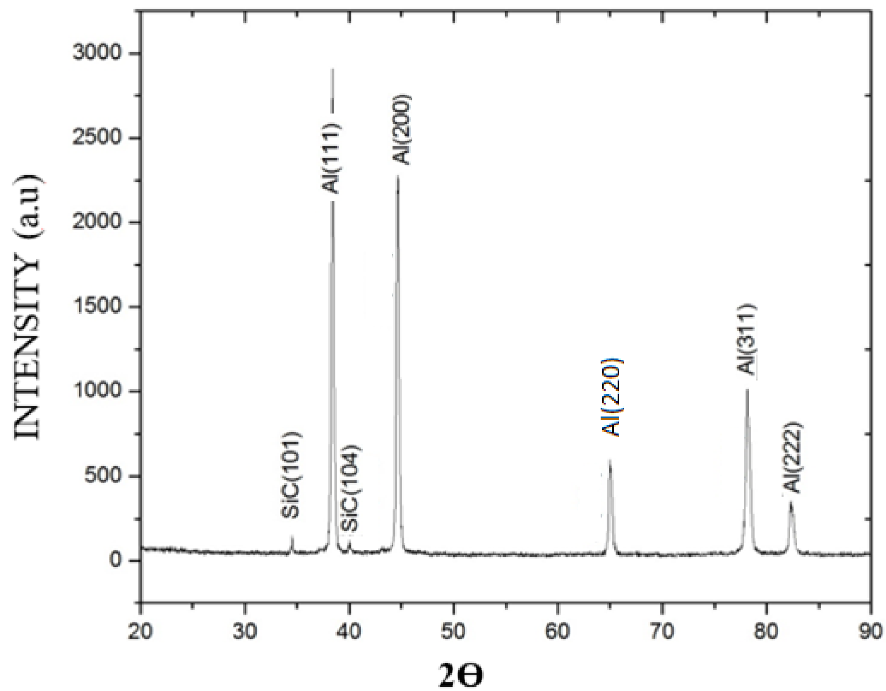

***Figure* 22: XRD pattern of Nanocomposite showing peaks of Al and SiC**

4.2 MECHANICAL PROPERTIES

Since this composite has an application in brakes, automobile frames and structures it should possess sufficient strength so that it can function well during its application, so to check its strength and tribological properties tensile test, Vickers hardness test and wear test had been carried out.

4.2.1 HARDNESS RESULTS

The results are obtained after Vickers hardness test indicates the inferior property of cast nanocomposites with respect to microcomposites, as their respective value and comparison can be seen in *Figure* 27 which is due to porosity and agglomeration present in the sample but after FSP the nanocomposites gives better property than microcomposites with both processing parameters. Which indicates the removal and proper mixing of material.

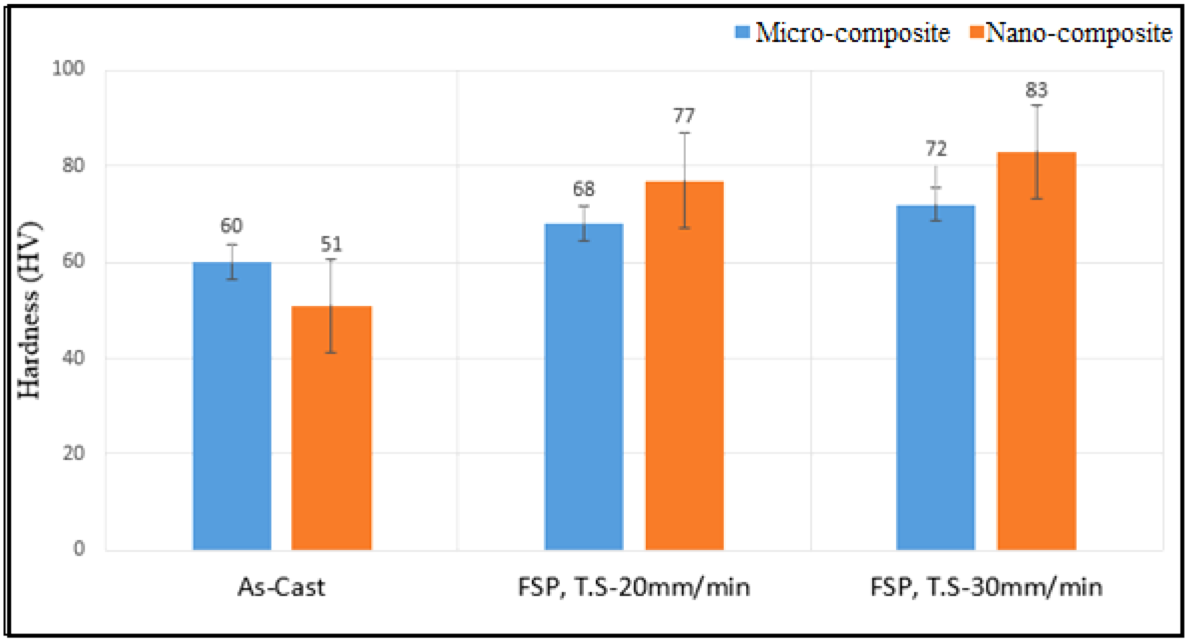

Figure **23: Hardness of samples**

4.2.2 Tensile test

The tensile test had been carried out for the samples of nano and micro-composites at its 2 different processing parameters. The *Figure* 23 shows the stress-strain relationship. The value of strength and ductility is tabulated in *Table* 4.

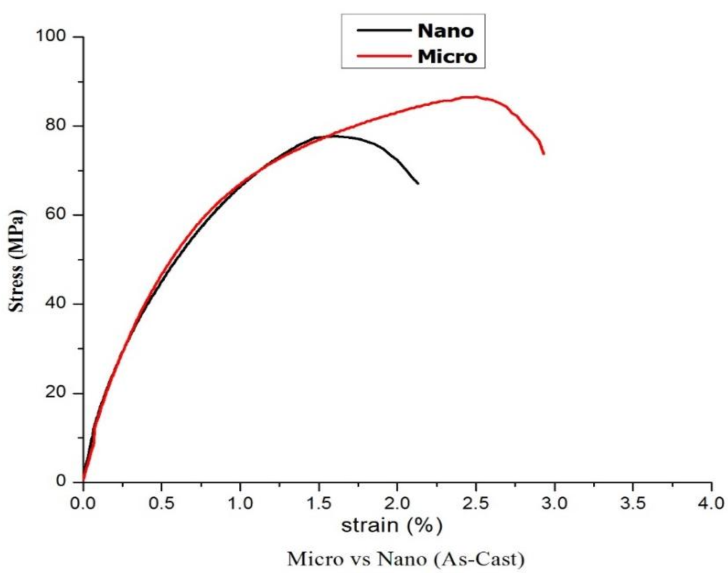

Figure **24: Stress vs. Strain curve of Nano(black line) and micro(red line) composite after casting.**

***Table* 4: Strength and ductility of nano and micro-reinforced cast composite.**

	0.2% yield strength	UTS (MPa)	Ductility at fracture (%)
Nano	64	77.08	1.49
Micro	65	87.6	2.46

As from the above *Figure* 23 and table it is clear that ductility is very less in both material, whereas as nano-reinforced composite shows inferior property than micro because the presence of porosity and agglomeration in it.

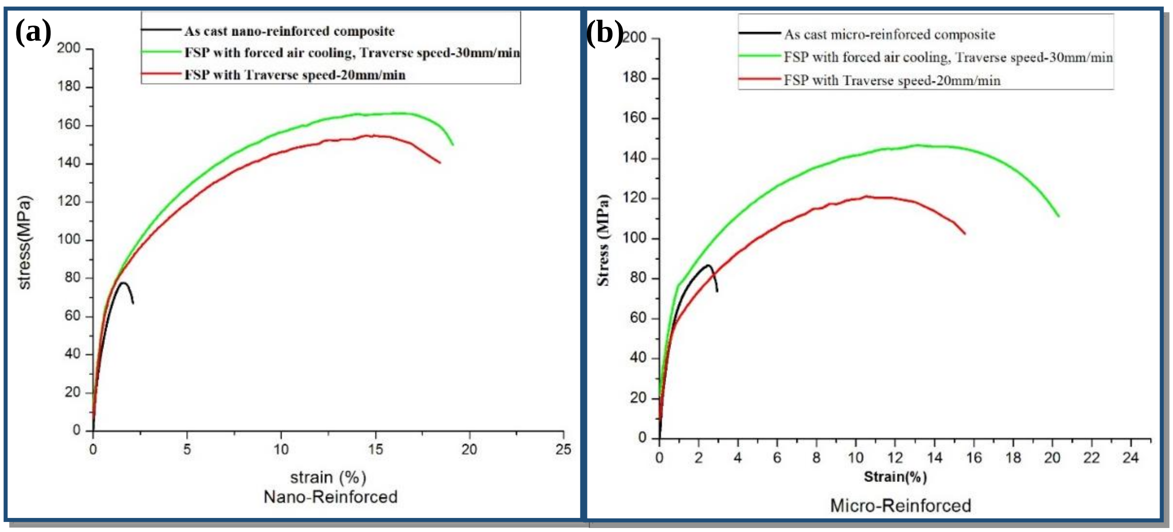

***Figure* 25: Stress vs. Strain curve of (a) Nano (b) Microcomposite in various conditions.**

The *Figure* 24 (a) Shows the effect of processing FSP parameters in nanocomposites as well as comparison between FSP and the cast of the respective composite and *Figure* 24 (b) shows the effect of processing parameters in microcomposites as well as comparison with its cast one.

It is clear from the above curves (*Figure* 24) that after FSP there is a huge increment in strength and ductility in both the reinforcements this shows the effectiveness of the FSP in cast composites. Various strengths and ductility values are tabulated in *Table* 5 for nano and microcomposites.

***Table* 5: Nano and microcomposites strength and ductility.**

	SAMPLES	0.2% yield strength (MPa)	UTS (MPa)	Ductility at fracture%
Nano-reinforced	As cast	64	77.08	1.49
	Air cooled	78.57	154.76	16.15
	Forced air cool	75.67	170	19.13
Micro-reinforced	As cast	65	77.08	1.49
	Air cooled	60.9	120	14.7
	Forced air cool	85.57	147	20.3

Out of performed two parameters of FSP they has an influential effect in strength and ductility of the composite this can be viewed in the *Figure* 24 (a and b).

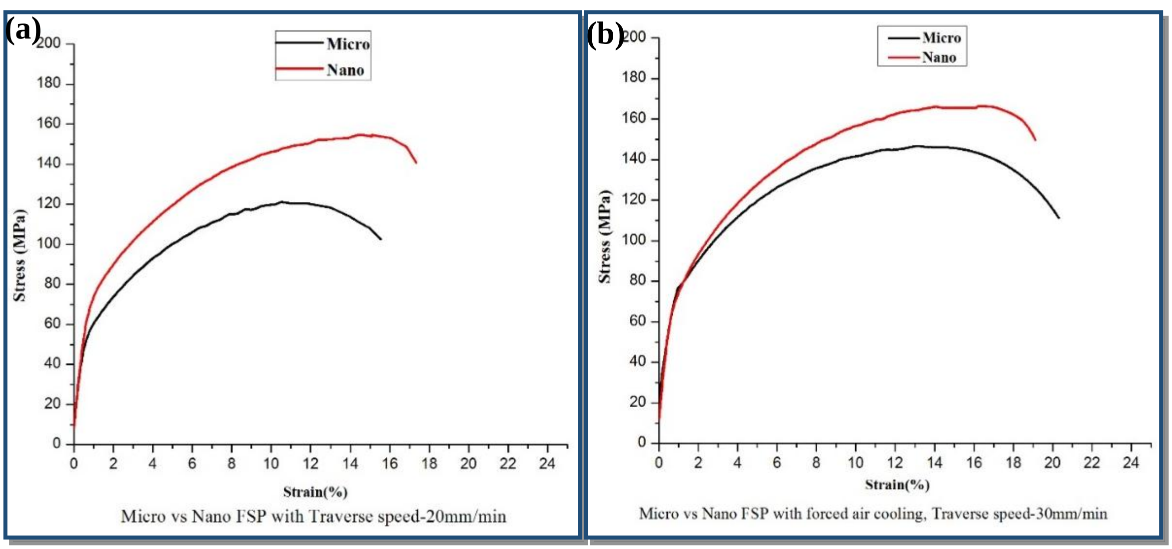

***Figure* 26: Stress strain curve of FSPed micro (black line) and nano (red line) composite with (a) traverse speed -20mm/min (b) traverse speed 30 mm/min +air cooling.**

In both the composite after FSP, nanocomposites shows superior property than microcomposites. This is due to the effective elimination of defects from nanocomposites and the strengthen of nanocomposites is governed by dispersed nano ceramics which restricts the dislocation movement since in nanocomposites the gap between two consecutive particle is small, so large shear force is required to move the dislocation through it, in comparison to micro-reinforced, in the nanocomposites the operative strengthening mechanism is around strengthening which has an influential effect in strength of the material.

4.3 Fractography analysis

Fractography of tensile specimen had been carried out under SEM. Which reveals the mechanism of failure mode in the composite. Fractography had been done for micro and nano composite samples of FSP in both the parameters. The *Figure* 26(a) shows the fractography of nanocomposites at 500X resolution of air cooled sample, whereas *Figure* 26(b) shows the fractography of microcomposites at 500X resolution of air cooled sample.

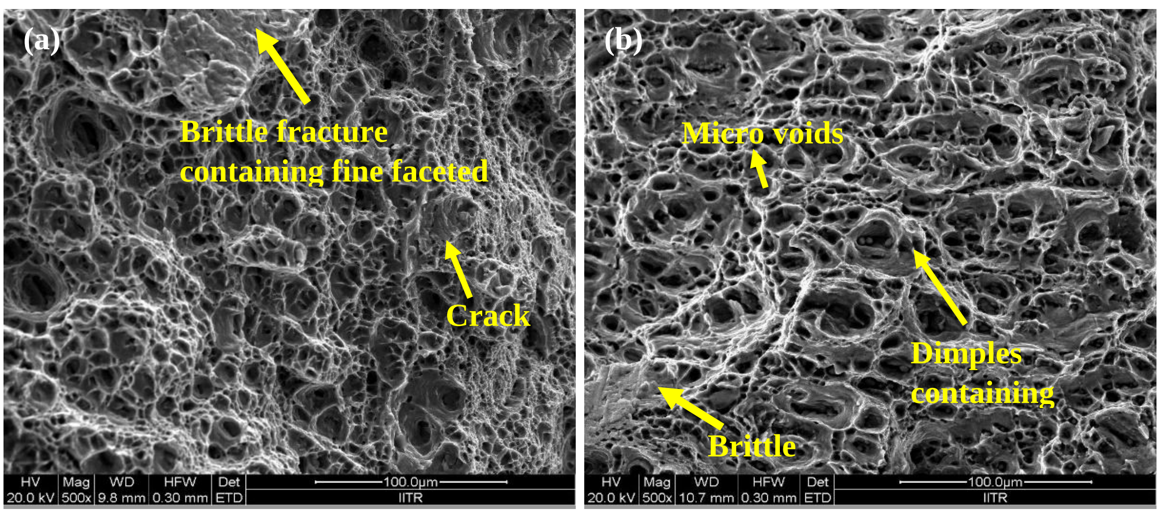

***Figure* 27: Fracture surface of (a) Nanocomposite and (b) Microcomposite.**

In the both ductilmoodsod oa fracturere has been characterized which is indicated by the dimples formation. The cavity arises from the inclusion and second phase particle which is enlarged by the continuous yielding and also the material between them is nicked and sheared. The mirror like faces showing cleavage which indicates the brittle manner of failure.

4.4 WEAR TEST

Pin on disc wear test of the specimen reveals the extinguishing wear rate of the nano FSPed material the test had been conducted on micro and nanocomposites after FSP in , forced air cool and cast , nano and microcomposites. The wear results, shown in *Figure* 29, reveal that before FSP wear (mass loss) is more in Nanocomposites because of its less hardness value kg and, 2kg of load and their respective coefficient of friction vs. sliding distance has been shown in *Figure* 28 (a) and *Figure* 28 (b). And agglomeration of particles due to which "material as a whole" comes out with the specimen which increases the wear rate in composite.

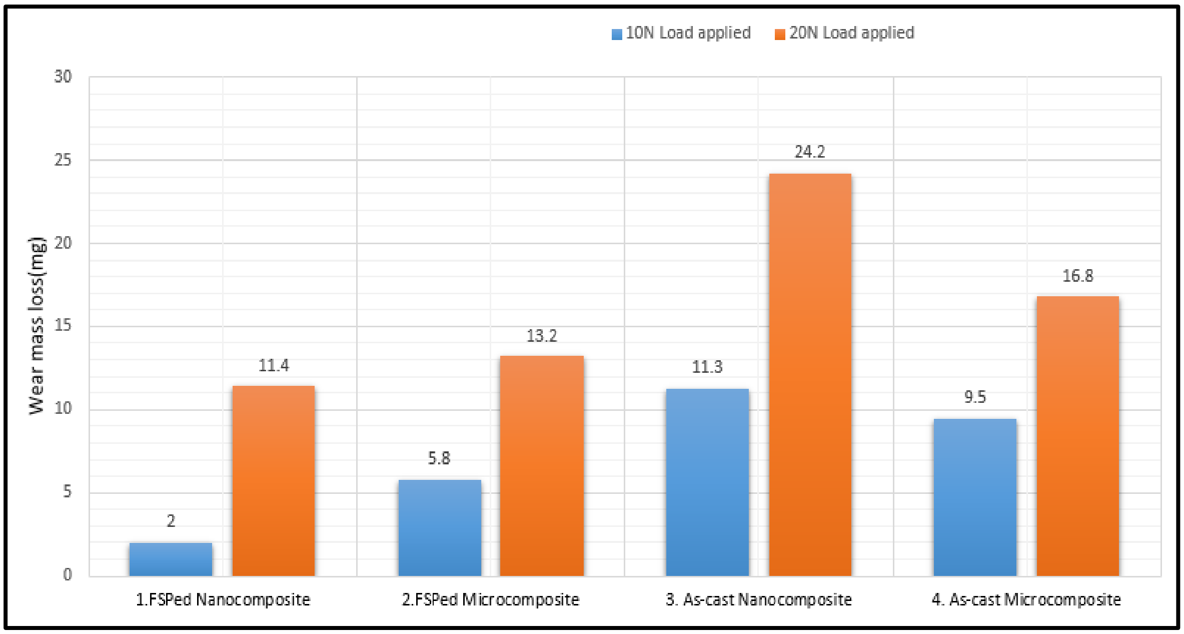

Figure 28 Curve showing wear loss mass at 10N and 20 N loading of various sample.

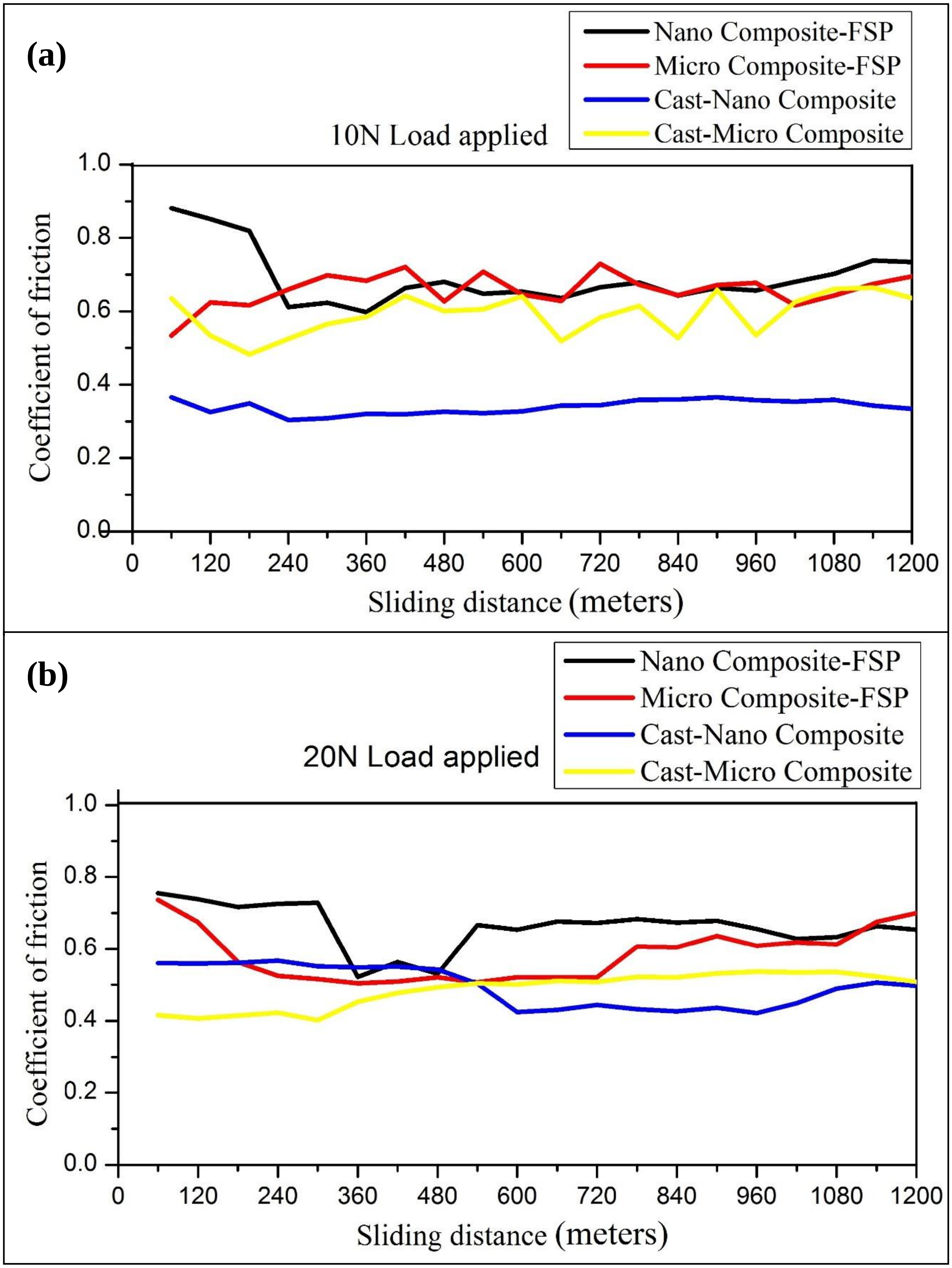

Figure 29 Variation of coefficient of friction with sliding distance of various samples with (a) 10N (b) 20N load applied.

4.5 WEAR SURFACE CHARACTERIZATION

SEM image of wear surface in *Figure* 30(a) FSped nanocomposites (b) FSPed microcomposites (c) cast nanocomposites, (d) cast microcomposites, indicates the mood of fracture occurs in various samples.

Adhesive wear occurs in all composites and the SiC which is an abrasive particle after coming out from the matrix creates abrasive grooves in the composite, presence of deep scratches on the worn surface which indicated that wear progressed by an abrasive mechanism and can be seen in *Figure* 30 given below indicated by black arrow. Due to the adhesion between sample and disc material the lamina gets pulled out creating delaminated surface and grooves which is indicated by the yellow arrow. As further sliding between disc and a composite sample occurs, some of the SiCp have been pulled out acts as an abrasive medium. The powdered SiCp along Al-oxides from the pin (sample) in combination acts as an abrasive media for wear. Close examination of sample reveals the formation of short cracks and shallow craters which is indicated by the white arrow.

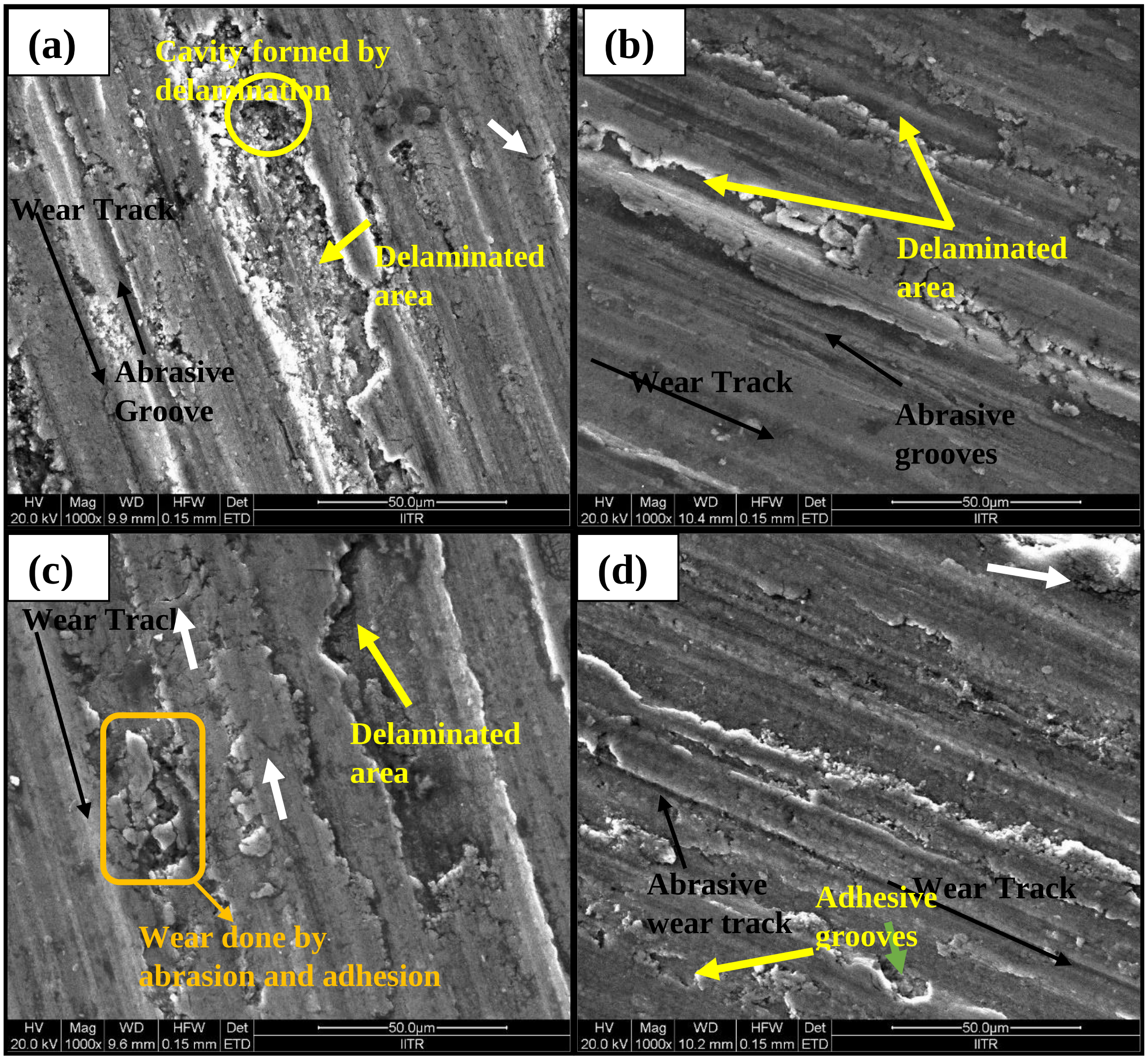

Figure 30: (a) FSped nanocomposite (b) FSPed microcomposite (c) cast nanocomposite, (d) cast microcomposite, indicates the mood of fracture occurs in various samples at 20N load.

Chapter 5	CONCLUSIONS AND FUTURE SCOPE

5.1 CONCLUSIONS

Micro and nanocomposites were prepared using stir casting route and FSP used for modification of microstructure and for uniform distribution of particles added. Detailed investigation of microstructure and mechanical properties were made. On the basis of obtaining results and their analysis, the conclusions are as follows:

1. After stir casting, the porosity and inhomogeneous distribution found to be a major problem which decreased mechanical properties. The amount of porosity and agglomeration found to be more in nanocomposite, which is due to the large surface area of nanosize particles although same weight. % of particles used. This results in the inferior mechanical properties in nanocomposite as compared with microcomposites.

2. FSP drastically enhances the strength and ductility of micro- and nanocomposites due to grain refinement, homogeneous distribution and porosity elimination.

3. The higher traversed speed (30 mm/min) showed better mechanical properties than that processed at lower traversed speeds (20 min). In case of nanocomposite, the FSP with 35 mm/Sec traverse speed and forced air cooling resulted better UTS (170MPa) and ductility (20%) compare to that of the microcomposite produced at same traversed speed. It is because of better grain size refinement due to less heat input and processing temperature at the time of FSP and more the pinning effect of nano size particles (as the number of particles are more for same wt.% In nanocomposites). The similar effect was observed in case of hardness and wear test also. The hardness and wear resistance of the FSPed nanocomposite (with 35mm/Sec traverses speed and forced air) found to be more than the others.

4. The strengthening mechanisms involved in the composites are grain size refinement and Orowan strengthening due to particle reinforcement. The effect of grain size refinement is more in case of nanocomposite than microcomposite because of more pinning effect of nanosize particles. The influence of Orowan strengthening is also more effective in case of nanocomposite because of less inter particle spacing available.

5. The fractography analysis showed presence of dimples in both samples; microcomposite showed larger size deeper dimples compare to that of nanocomposite.

This indicates the nucleation and growth of voids occurred from the second phase SiC particle. The wear surface analysis showed both adhesive and abrasive wear mechanisms.

5.2 FUTURE AND SCOPE OF THE STUDY

- Further improvement in mechanical properties can be done using other parameters of FSP by optimizing between machine variables and processing temperature.
- The results can be used to compare the properties of this composite with other reinforced composite.
- Corrosion behavior of the composite can also be studied.

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ABSTRACT Al-6061 alloy is mostly used for automobile parts because of its good corrosion behavior and high stiffness. However, it has poor wear resistance which limits its wide applications. Metal matrix composite (MMC) in which hard ceramic particles such as SiC is dispersed leads to an improvement in wear resistance and stiffness but in the expense of its ductility. Major problem of making the MMC is the uniform distribution of the ceramic particles.

18**In the present study, an attempt has been made to**

distribute SiC particles uniformly within the matrix by stirring casting and further modified by friction stir processing (FSP) and achieving enhanced property. FSP is a novel technique and can be useful to distribute SiC particles along with the refinement of matrix grains. Thus, mechanical properties could be improved by 2 ways: due to ceramic reinforcement and grain size refinement. In this work, Al 6061 alloy matrix composites (AMCs) reinforced with 2 wt. % SiC particles of micro (size 20-160 μm) and nano (size

30-80 nm) was prepared using casting via stir casting technique. The FSP was conducted at a speed of 720rpm using a tool of cylindrical pin in two condition first in normal air cooling with traverse speed of 20 mm/min and second in forced air cooling with fan and traverse speed 30 mm/min to lower its processing temperature to the minimum possible value. The microstructure and mechanical properties (hardness, tensile strength, and wear resistance) were compared for the both micro and nano composites, with and without FSP. The characterization

43**was carried out** using **X-ray diffraction (XRD), optical and scanning**

electron microscopy. After FSP of the composite, it was observed that mechanical properties was improved

2**due to** homogeneous **distribution of SiC particles and** grain refinement **of**

the alloy matrix. Significant improvement in the ductility was also measured after FSP as compared to that of the cast composite. Nano reinforces ceramic gives better property than micro-reinforced after FSP but at the same time nano reinforce composite gives very inferior property before FSP due to the high amount of porosity and inhomogeneity present in the material

20**due to the** large **surface area** to weight ratio **of nano** particle, **which**

was verified by Archimedes principal. The influence of porosity had been observed in wear rate also

40**wear rate of** nano **composite is found to be** more **than that of**

micro reinforcement, but after FSP the significant removal of defects, porosity and homogeneous distribution the strength, ductility and hardness had been recovered. Which shows the effectiveness of the FSP in nano composite. i Chapter 1 INTRODUCTION 1.1 INTRODUCTION Aluminum (Al) alloys are showing their prominent role in various applications such as:

29**structural applications, aerospace, military, and transportation industries due to their** (1) **light weight,** (2) **high** stiffness, and (3) **corrosion**

resistant behaviour [1]. However, it has some drawback which inhibits its application and is due

10**their poor wear resistance, manifested by severe adhesive wear** [2] [3]. **In**

comparison with the unreinforced Al **alloys,** Al **matrix composites reinforced with ceramic phases exhibit higher strength and stiffness,** improves its **tribological characteristics, and**

increases its workability in high temperature applications [4]. Composite refers to the combination of two or more materials having distinct features in which one is reinforcement, distributed in another matrix phase, which in combine gives the distinguishing property than the parent material.

34**On the basis of physical nature**

this is classified as:

34**Polymer Matrix** Composite, **Metal Matrix** Composite (MMCs) **and ceramic**

composite [5] [6], but this project is specifically concerned with Al- 6061 alloy MMC in Al-MMC the Al-6061 is a matrix phase which serves as a binder for reinforcement phase which usually a ceramics such as SiC, Al2O3, TiC but in the current emphasis is only on SiC. Moreover, recent research reveals that the MMCs having Nano- reinforcement are showing superior wear resistance, but these composites lacks in thermal conductivity and ductility [7]. However, there are various processes by which the ductility of the composites can be regained without affecting the strength in that queue a very prominent and efficient processing route FSP has been demonstrated [8]. The

19**Friction Stir Processing (FSP) is a** solid state processing **technique** which was **developed** in 1999; **by Mishra et al.** It comes after **the** invention **of friction stir welding (FSW)**

process which has completely changed the joining process, this

39**was invented by Wayne Thomas at The Welding Institute** (TWI Ltd.) **In 1991[**

9]. After that, various researches and successful application has been reported and is used vigorously in metal fabrication industries [10]. FSP uses same thermo-mechanical principal as used by FSW to enhance the microstructural characteristics for property enhancement. In starting FSP was used to produce high grain boundary miss-orientation [11], super plastic Al alloy with ultra-fine grain size [12] and was limited to Al and Mg alloy only because FSP becomes easy for those material which possess low yield strength, low hardness and high ductility, but further the successful research has been carried out on the alloy of copper, steel and titanium [13][14]. Furthermore, research has been carried out successfully to produce surface composite on the Al substrate Property enhancement of cast Al alloy [15]. As well as cast MMCs [14], homogenization and property enhancement of powder metallurgy Al alloy [16]. It also eliminates casting defects Since there has been a lot of successful demonstrations are proposed in very short time, which shows that this technique is growing as a very effective means of solid state fabrication/processing [17]. 1.2 MECHANISM OF FSP This processing technique works on a very simple mechanism, it uses rotating non- consumable tool which consists of accurately designed pin and shoulder arrangement as shown in Figure1 the

> 21**pin is inserted into the** monolithic **work piece**

such that the shoulder just touches the workpiece and traverses along the straight line as shown in due to this, heating and material flow takes place.

> 21**Heat is generated** due to **friction between** shoulder **and work piece and** partly with **a**

pin and shoulder friction which softens the material and the combined action of translation and rotation moves the material from front to back. In that way stirring is done, which leads to the development of refined microstructure in the processed zone. The main mechanism which works behind the development of fine grains [18]. Figure 1: Schematic diagram showing Mechanism of FSP. Chapter 2 LITERATURE REVIEW 2.1 INTRODUCTION Al

> 12**alloys are very promising for structural applications in aerospace, military, and transportation industries due to their light weight, high strength-to-weight ratio, and resistance to corrosion**

[19]. Heat treatable alloy such as 6000 series has good strength at room temperature and strengthening is due to solid solution strengthening, Hall Petch strengthening and precipitation strengthening [20] but in this the most effective precipitation strengthening is given by the formation of coherent, meta-stable θ" [3], phase which comes after solutionizing and aging for 1hr at 200℃ beyond this over-aging may starts which reduces the strength and hardness because of formation of θ' phase which is semi coherent phase, that is why its application is limited at higher temperature because working for few hours at higher temperature results in reduction of strength due to over aging and is beyond recovery it will not regain its strength even if its temperature lowered to room temperature. This is the disadvantage of precipitation hardened alloy over MMCs since the presence of insoluble phase in it can provide strength to the system for longer duration of time, even at high temperature application [21], there is the requirement of composite material. That is the reason why application of these alloys is limited in high temperature applications [23]. The vigorous investigation on MMCs reinforced with nano particle has been carried out worldwide in recent decades and investigation is still going on [24-27], because of their exceptional and suitable application in

the field of transportation, defence, structural and mechanical [19]. Reducing the particle size to nano range introduce an exceptional change in mechanical properties and gives other strengthening effect because of significant interaction of dislocation with particle. The main issue with the Nano reinforcement is there improper incorporation of particles into the matrix phase which is due to large surface area and low wettability [28]. Which makes it difficult to produce it by conventional processes. Powder agglomerates forms clusters which inhibit the homogeneous mixing and its effect can be visualized on mechanical properties and strengthening of material. Several alternatives to overcome this issue had been proposed, broadly the production of MMCs 3

17**can be** divided **into two groups** in **-situ** [17] **and** ex **-situ** [29]. **In the** former method **the**

reinforcing particle is introduced into the matrix by external means while in later one the reinforcing phase generates due to chemical reaction involved. Several other techniques also had been employed for the production like powder metallurgy, spray forming, stir casting route etc. Out of this stir casting route is most suitable because of its cost effectiveness and easy set up [30]. 2.1.1 Recrystallization mechanism Grain refinement in the stirred zone is governed by dynamic recrystallization which occurred due to

14**intense plastic deformation at elevated temperature.** A different mechanism **of**

dynamic recrystallization had been proposed in the Al alloy such as

33**continuous dynamic recrystallization (CDRX), discontinuous dynamic recrystallization (DDRX), geometric dynamic recrystallization (GDRX)**

[31]. Because of high stacking fault energy present in Al due to their FCC structure DDRX doesn’t happen in them, as they recover very fast [32]. DDRX is indicated by the formation of newly formed

14**grains at high angle boundaries.** Whereas sub **grain**

35**growth, lattice rotation associated with sliding, and lattice rotation associated with slip**

on the other, hand, shows the mechanism of CDRX. Research done by Jata and Semiatin [33]. Because of

46**continuous rotation of the original low angle boundaries during** machining, CDRX **is**

an

14**operative dynamic nucleation mechanism during FSW/**

FSP. 2.1.2 Strengthening mechanism a) Grain refinement MMCs the hard phase provides pinning effect of the grain boundary movement that's why the growth process hindered resulting in relatively finer grains than unreinforced one, in the present study the grain is further refined by the FSP process which provides the additional strength of the material, and is governed by Hall-Petch effect which is given by the Equation--(1) $\sigma Hall{-}Petch$ = σ + $K\sqrt{dy}$ -------- (1) Since

16**finer the grain will be the** mayor is **the grain**

boundary and grain boundaries are an obstacle for the dislocation movement resulting in strengthening of material. b) Orowan strengthening This strengthening effect caused by the presence of hard particle in the matrix phase, which restricts the dislocation movement. The effect is more pronounced in nano reinforcement rather than micro reinforcement because of the large size of particle and greater inter particle distance. The cutting or bowing mechanism may happen in the material the Figure 2 describing this phenomena. It has been established fact that creep resistance, Fatigue properties and lifespan of Al-MMCs improves with increasing percent of reinforcing phase [34] because orowan bowing is necessary for bypassing the dislocation. The Equation--(2) governs the orowan effect [35][36]. $\Delta\sigma OR$ = 03.113$bG\ ln$ $(d2bp)$ -------- (2) $dp($ $\sqrt{2vp}{-}1)$ Figure 2: Mood of operation of Orowan Mechanism. In this b is the burgers vector and G isthe shear moduluss. Since current work is performed on 6061 Al-alloy which is a precipitation hard enable alloy as it contains Mg and Si the precipitate forms into the material also provide strength by orowan strengthening mechanism. 2.1.3 Other strengthening a) Load transfer effect In the composite presence of reinforcing phase, which is hard and stiff takes a part of load into it providing strength to the base material. To calculate the contribution in strengthening Nardone and Prewo [20], proposed a modified shear lag model can be used. So the equation for the load transfer to the material can be given by Equation (3) $\Delta\sigma$ = $\sigma Matrix$ × $Vparticles$ $[(l{+}4tl)A$] -------- (3) Where "l" and "t" are the lengths along the load and perpendicular to the load, A is the cross sectional area Vparticles is the volume fraction of the particle. b) Coefficient of thermal expansion

45**mismatch Due to the difference in coefficient of thermal** expansion there developed **a**

strain field around the particle or there is a very high dislocation density around particle which, when interact with dislocation hinders its movement resulting in greater strength of material [37]. However the contributions of different strengthening mechanism are different and varied according to particle size and volume fraction it can be understood from the Figure 3 a research done on Al/Al2O3 by R. Casati, et al. [38]. Reveals the effect of strengthening Figure 3: Effect of variation of various strengthening mechanism with Particle size. effect and their contribution. As can be seen from the above Figure 3 that there is very high contribution of orowan strengthening and coefficient of thermal expansion mismatch when particle size is very small and the contribution of load bearing phase effect is least. 2.2 PRESENT STATUS OF THE WORK A variety of research is currently going on in this field by taking various grades of alloys and reinforcing phase like SiC [39], Al2O3 [24], TiB2 [40] etc. In different shapes and sizes and fabrication of MMCs using these two components are done in various processing routes such as stir casting [41], spark plasma [42], squeeze casting [43], powder metallurgy [44] and ultrasonic spray forming. There are various challenges associated with the type and size of particles as well as with fabrication route. So this section will discuss about the current and past working/research status, which are going on worldwide in the perspective of Al MMCs their processing technique challenges and opportunity in the various fields such as aerospace, automobiles and metal fabrication [19]. The research done by Mohsen Hossein-Zadeh et.al [45]; Semnan University Iran ,in which they have taken Al2O3 powder which was

> 17**milled in a high-energy ball mill for** 20 hr, at **the**

same time 1 wt. % of Al2O3 was introduced into the melt. Various mechanical properties such as tensile test and wear and microstructural characterization of this fabricated composite had been carried out. A nano particle in the matrix had been revealed by peak broadening in X-Ray diffraction (XRD). Moreover the distribution of reinforcing phase was quite inhomogeneous. Because of the presence of nano hard particle grain size reduces as it acts as a pinning effect to the growing grains, it act as a heterogeneous nucleation site for the grain nucleation and due to the refinement of the grain hardness, yield strength and wear resistance of Al–Al2O3 has been raised significantly. Furthermore, there is always a well-known problem for metallurgist which is optimization of strength and ductility as they are mutually exclusive to each other was present in this case, but the research done by Gaohui Wu et al. [46], they show that

> 11**adding distinctive elements (Sc, Zr) refining the heterogeneous nucleation** cites **to**

nano level and due to this adding

> 11**high volume fraction** of **reinforcement during the fabrication**

had been achieved with adequate strength and ductility and this

> 11**process represents a very important tool** for achieving **outstanding** 7

combination of strength and **ductility** in **high**

volume fraction SiCp/Al composites. Even further research has been carried out on a cast A356 alloy reinforced with nano SiC Ali Mazahery et al. [27], and with Al2O3. Mazahery et al. [24], P. Rama Murty Raju et al. [47]. nanocomposites and also on Al/A206 S. Tahamtan et al. [48], and Al/2024 Hai Su et al. [49], with Al2O3 using stir casting routes which shows almost uniform distribution of reinforcing phase enhancement in ultimate tensile strength and ductility exhibited by nano- Figure 4: The tensile curve of (a) nano and (b) micro composite at 800℃ Al2O3 reinforced Al and is attributed by uniform distribution and grain refinement. Although stir casting is widely used due to its cost effectiveness and easy fabrication method S.A. Sajjadi et al. [50] shows in his work in which they compare composites in which one is

28**fabricated by stir** casting **and** another by **compo-casting processes,**

they found that

28**using compo-casting process** gives **best mechanical properties**

in turn. There is an effect of casting temperature on mechanical properties which was demonstrated by M. Karbalaei Akbari et al. [40]. In which TiB2 particle as a reinforcement in A356 Al alloy using casting route they conclude that the effect of casting temperature on its increment reduces the mechanical property of material as more porosity likely to form at high temperature. Their stress-strain relationship is given in the Figure 4. Fracture surface reveals the particle segregation at grain boundaries along with fine dimples and nano particle in it. The results show an evidence of Si cracking and de- bonding, which is

4**one of the main characteristics of fracture mechanism of Al–Si alloys** which **was detected on** the **fracture surface of** composite **matrix,**

which is reinforced with TiB2 Nano and micro particles Figure 5 (a) (b) (c) (d) Figure 5: The

4**fracture surface (a) shrinkage void (b) fine dimples with smooth surface in** nano composite **(c) ultrafine dimples with rough surface in** nano composite **(d)** imbedded nano particles **in dimples.**

Moreover, for the improvement of mechanical properties hybridization technique is implemented using stir casting and ultrasonic vibration impairment during the introduction of reinforcing phase S. Tahamtan et al. [51] proposed a technique in which they enhance

27**tensile properties of a network-structure** Al-A206matrix **composite by hybridizing micron- sized** composites **with Al2O3 Nano particulates,**

and furthermore this hybridizing technique had also been used by Weiping Chen et al. [46] By squeeze casting route in which (SiCp- Ti)/7075 Al

7**hybrid composites with added minor Ti particles were fabricated by squeeze casting.** This way **the feature** comes out on **the**

turn in which the ductility was unaffected and significant improvement of strength

7**compared with Ti particle** one. **This may provide a new method** of **fabricating composites with excellent mechanical properties.** More on **squeeze casting**

technique, Al–Si base composites reinforced with different mixtures of Ni and nano-Al2O3 particles have been fabricated by squeeze casting and their metallurgical and mechanical characterization has been done by Hashem et al. [43]. In which they found that the ductility of the fabricated composites is significantly improved by increasing the added Ni particles. The composite material, reinforced with 5 wt. % Ni and 2 wt. % nano- Al2O3 particles showed superior ultimate tensile strength and good ductility compared with any other added particles in their investigation. But in various cases there has been a decrease in strength when working under elevated temperature, but the research done on nano-SiCp/Al2014composites by Long-Jiang Zhang et al. [52], using

30**stir casting combined with** hot **extrusion** shows **high strength and good ductility** even **at elevated temperature.**

Besides the major reinforcing phase like SiC and Al2O3composite had also been fabricated by nano MgO particles A. Ansary Yar et al. [41]. In which, Al

32**alloy (A356) matrix composites reinforced with**

nanoparticle of nanoparticles

32**were fabricated via stir casting method.**

6**Hardness and compression tests were carried out in order to identify mechanical properties. The results reveal that the composites containing 1.5 vol. % reinforcement particle fabricated at 850°C** has **homogenous microstructure as well as improved mechanical properties. The**

major challenge in stir casting is wettability of reinforcing phase where poor wettability hinders the fabrication of good casting by agglomeration and inhomogeneous distribution. One of the remedy for this issue is pre-coating of particle is a good way of increasing irritability. In the study by M. Heydarzadeh Sohi [53], on AA8011and Al2O3,

26**a mixture obtained by mechanical alloying of 40 wt. % Al and 60 wt. % of Al2O3**

shows good distribution and mechanical property. Beside stir casting ultrasonic cavitation method used by S. Gopalakannan, and T. Senthilvelan [54] is also in great use which shows uniform distribution of, SiC nanoparticles in the MMC. Moreover spark plasma sintering method was also applied for the purpose of fabricating composite the work presented by D. Garbiec et al. [42] Shows the fabrication and characterization of Al–Al2O3 composite materials taking 5%, 10%, 15% and 20% volume fraction of Al2O3. The spark plasma sintering method was applied for the purpose of fabricating these materials. The obtained Al–Al2O3 composites were characterized with porosity from 1.27% to 5.07%. It was proven that as vol. Fraction of 10 reinforcing phase increase, its density, hardness, and compression as well as tensile strength also increases. Out of all above discussed method homogeneous distribution of particle is very difficult and require lots of prior effort to achieving sound casting with good wettability and homogenization. But the FSP comes with a very good and easy method of post fabrication and modification method. Various attempts had been done in improvising the property of micro and nano MMCs. For achieving homogeneous distribution in the matrix phase refinement of grains and dendrites and to improve metallurgical and mechanical properties, various research has been proposed to demonstrate the effect of FSP on the Al/alloy-SiCp composite. 2.3 CASTING OF MMCs The suitable and economical method for composite fabrication is stir casting route. The main effect was observed in the mould preheat temperature as the temperature increases the porosity level goes down, whereas but inhomogeneous mixing dominates on the counterpart. From this literature is that the mould temperature should be optimized in order to get minimum porosity and agglomeration and SL route should be practiced while casting [55]. Previously a research has been carried out to find appropriate ways of manufacturing composite by minimizing the flaws produced in compo-casting

2**Composites based on two** Al **alloys (A536 and 6061) reinforced with 10% or 20% volume fraction of SiC particles were produced by gravity casting and a novel two-step mixing method was applied successfully to improve the** wettability and distribution of the particles [39]. Property of the

composite can be easily altered by varying composition of reinforcing phase and as well size of the reinforcing phase incorporation of Nano particle leads to the improvement in various properties only being the problem is. A research has been carried taking various reinforcing phase B4C, SiC and Al2O3 (0–20 vol. %) and properties, interfacial reactions were analyzed deeply[56].

5The stir-casting route followed by hot extrusion

had been adopted.

5Clear interfacial reaction product/layer was found at Al–SiC interface for composites held for a relatively long processing time 11 (>30 min). No reaction product was observed at Al–B4C and Al– Al2O3

15two secondary phases (alumina and another phase containing Al, boron and carbon) were found in the Al matrix away from the interface in Al– B4C composites [57]. For the

last two decades composite material is showing their benchmarks in various fields of engineering, which attracts researcher to produce various composite by easy and economical route. Manufacturing of Al/Al-alloy-SiC particle can be done in various ways, such as stir casting, powder metallurgy and spray casting routes. Shriyash S. Shinde et al. [58], Demonstrated how stir casting route is an economical and effective way to produce composite

8in which a dispersed phase (ceramic particles, short fibres) is mixed with a molten matrix metal by means of mechanical stirring. The liquid composite material is then cast by conventional casting methods.

In preparing MMCs by stir casting method some of the factors that need considerable attention are as follows:- ? To achieve

25uniform distribution of the reinforcement material. ? To achieve wettability between the two main substances. ? To minimize porosity in the cast

MMCs. 2.4 FRICTION STIR PROCESSING (FSP) 2.4.1 Microstructural and mechanical properties in FSP This section thoroughly discusses about the parameters or variables and their effect on the microstructure and other mechanical properties in processed zone. Grain size is influenced by the operating temperature of FSP as the temperature of machining increases grain becomes coarse. These variables in the FSP are optimized in such a way to obtain required microstructure such as tool rotation speed increases the heat Figure 6:

9**Effect of FSP parameters on nugget grain size in FSP 7075Al-T7651 at processing parameter of: (a) 350 rpm, 152 mm/min and (b) 400 rpm, 102 mm/min.**

generation is more and the consequence of this relatively coarser grains are formed. Which leads to the lesser strength and high ductility. There is an another effect of the tool traverse

16**speed as the tool traverse speed increases** less **heat**

is generated and finer grains are produced. In combination the effect of grain size and correspondingly strength and ductility is influenced by the ratios of the rotation speed and tool traverse speed as this ratio increases coarser grains are formed as seen in Figure 6. In stirred zone the temp distribution from advancing side to the retreating side changes and it also changes from top to bottom this thing also affects the grain size throughout the nugget zone Figure 7. Figure 7: Showing top to bottom and retreating side to advancing side, the variation of grain size in stirred zone. The pin and shoulder: diameter and height are also the influencing parameter in grain growth and recrystallization a large diameter shoulder generates more heat and lesser diameter would not able to generate sufficient heat which causes difficulty during stirring so the parameters should be optimized. The shape of the pin influences the flow of the 13 material and as an overall result influences mixing and mechanical properties of the processed zone. 2.4.2 Advantages FSP is showing there prominent effect in the

14**solid-state processing technique. In short duration**

of time, few successful and effective applications of FSP have been carried out

3**for localized microstructural modification for specific property enhancement. For example, high-strain rate super-plasticity was obtained in commercial 7075Al alloy by FSP. Furthermore, FSP technique has been used to produce surface composite on** Al **substrate, homogenization of powder metallurgy** Al **alloy, microstructural modification of** MMCs **and property enhancement in cast**

Al **alloys**

furthermore various

31**benefits are** tabulated **in Table 1. Table 1: Benefits of** FSP **Metallurgical** benefits **Environmental** benefits **Energy** benefits ? **Solid**

state process use allows reduction in weight ? Improved materials ? Good stability dimensional ? No use of harmful gases ? Decreased fuel ? No loss of alloying ? Surface preparation consumption

44**due to high strength to** elements not required **weight ratio. ? ? Excellent** metallurgical **properties.** Fine microstructure ? **Material**

saving ? Aircraft, automotive and structural applications 2.5 OBSERVATION OF LITERATURE REVIEW Thus, from the above discussed literatures on various topics such as composite casting technique secondary machining process effects and FSP effects on various substrates and the influence of parameters on the properties has been discussed. So the basic highlights of the entire discussion are as follows. ? Moulds should be preheated to avoid porosity in the cast composite, presence of Mg reduces formation of porosity and oxides, and extra care should be given on the temperature and the vortex formation during stirring ensures proper distribution which also influenced by the time and temperature of stirring. ? Two step mixing techniques using SL-route is an adaptable way of casting composites ? FSP is found to be very good processing technique for grain refinement and homogeneous distribution of reinforcing phase as well as solute which in turns gives better strength and ductility. 2.6 AIM AND OBJECTIVE Metal matrix Nanocomposites shows very outstanding property, however manufacturing of metal matrix Nanocomposites and proper mixing of reinforcing phase by conventional stir casting route is very tedious job[59] so this project has been carried out to find an alternate way modification of Nanocomposites using FSP. The aim of this project is to form a metal matrix Nanocomposites via the casting route and modified without degrading its properties, processing has been accomplished on the initially prepared composite of Al6061-SiC (2%wt.) via stir casting route and having reinforcement particle of micron size in the range of ~ 20-80 microns and nano particles in the range of ~ 30-80 nm. Finally processed composite should possess sufficient ductility and strength and homogeneous dispersion of reinforcing phase and solutes into the matrix moreover emphasis is on to improve the flaws observed in the conventional cast metal matrix [23]. As per above discussed the background, the objectives of the present work are visualized as: • Preparation of nano composite having Al-6061 as a matrix phase and SiC as reinforcement which is homogeneously dispersed into the material using FSP as a tool for modification. • Characterization of the composite by optical microscopy SEM and XRD analysis. • Determination of mechanical and wear properties. • Analysis of the mechanisms of strengthening of the composite

24**on microstructural** evolution **and mechanical properties.**• Comparison **of the** property of nano **composite**

to that of microcomposite Analysis of mechanical properties and wear behavior and setting up of structure-property relationship for the best combination of strength and ductility for practical applications. Chapter 3 EXPERIMENTAL PROCEDURES This chapter deals with the thorough study of the experimental procedures carried out during the investigation. The

1**microstructural evolution was characterized by optical microscopy,** XRD **and electron microscopy. The fractography after the tensile test** had been **carried out by scanning electron microscopy to** correlate **the ductility obtained by tensile tests**

as well as SEM of wear surface had been taken to analysis wear behavior pattern in composite. The wear behavior of the material has been carried out using a pin on disc.

24**Scanning electron microscopy (SEM) was used for** fracture analysis after **the**

tensile test. The

1**mechanical properties were evaluated by tensile testing, Vickers hardness measurements** and wear properties. **The**

detail of each process is described in the following divisions and the experiment flow chart has been described in Figure 8 Figure 8: Flow diagram 3.1 MATERIAL SELECTION

1**Commercial Al** 6061 **alloy (composition given** in Table 2) **plates supplied by M/s. Hindalco Industries, India** were **used** for **the present study.**

Table 2: Composition of 6061 Al-alloy Elements

23**Cu Mn Si Mg Zn Fe Al Composition (wt. %) 0.** 1 **0.** 1 **0.** 6 **0.** 9 **0.** 12 **0.** 5 **Bal**

3

41.**2 BALL MILLING Ball milling** had been done **using Planetary Mono Mill Pulverisette 6**

classic line machine Figure 9 To achieve nano SiC .initially taken SiC of Alfa Aesar brand of particle size having 20-160μm and was put in a tungsten carbide jar in the ball to weight ratio of 15:1 using tungsten carbide ball. Wet milling had been performed and milling was carried out for 45 hrs and later on particles were characterized by TEM for particle size and Crystallinity. Figure 9: Planetary Ball Mill 3.3 CASTING SET UP Casting had been done in open muffle furnace Figure 10(a) by heating the alloy at 700℃ in a graphite crucible Figure 10(b) later on after melting SiC particle which was preheated at 300℃ for 1 hour had been introduce into the molten metal at the rate of 2grams/sec through a Cu pipe and simultaneously stir the system using stirrer fitted on drilling machine at 200 rpm creating vortex inside the molten metal for 2 min later on the molten metal had been poured in cast iron mould which was preheated at 150℃ of dimension having 55mmX55mmX120mm than left the simple to cool naturally and finally got billet of dimension having 50mmX50mmX110mm. Which was then cut into 4pices using bend hacksaw of dimension 100X50X10 and finally finished the surface by shaper. The casting was done to achieve 3 billets of the above mentioned dimensions (a) Al-6061+SiC (micro- reinforced) (b) Al-6061+SiC (nano-reinforced) later on after surface finishing by shaper the sample was carried out for FSP. (a) (b) Figure 10: Casting Equipment (a) muffle furnace (b) crucible 3.4 FSP SET UP A milling machine arrangement had been modified to work accordingly as an FSP Figure 11 (a) universal adjustable fixture was placed over the bed of the machine and FSP tool Figure 11 (c) of dimension listed in the table. The FSP machines provide 3 different rotational speeds 720rpm, 1200rpm, 1500rpm which is governed by belt and pulley arrangement and wide range of traverse speed which is governed by hydraulic mechanism. An Experiment was conducted with 2 parameters: in 1st tool traverse speed is 20mm/min with 720 rpm of traverse speed and natural cooling and in 2nd tool traverse speed is kept 30mm/min with 720 rpm along with air cooling while machining so as to minimize the machining temperature in hope to achieve better mechanical properties. Two pass nearby near has been taken for nano and microcomposites Figure 11b. For each sample of all two composite material viz. a) Al-6061+SiC (micro-reinforced) (b) Al-6061+SiC (Nano reinforced). One had been used for tensile specimen sample and another had been used for microstructural and wear examination. (a) (b) (c) Figure 11: (a) FSP machine (b) composite sample after FSP (c) FSP tool and tool holder 3.5 XRD The evaluation of the microstructure had been carried out in a Rigaku Smart Lab Figure 12 X-Ray Diffractometer using Cu-Kα radiation. The XRD pattern was recorded from angle Figure 12: X-Ray Diffractometer 20˚ through 90˚. A scan rate of 0.5˚/min was maintained during the collection of the data. The X-Pert High score Pro software was used to carry out the background subtraction and Cu Kα2 peak stripping and analysis of present phase. 3.6 TEM TEM of powder sample had been done using machine Figure 13 sample was prepared earlier by ultra-sonication of the powder particle for 45 min later on a drop casting technique. Figure 13: TEM machine 3.7 MICROSTRUCTURAL CHARACTERIZATION 3.7.1 Optical microscopy Microstructural characterization to observe grain refinement and particle segregation had been carried out using Carl Zeiss Optical microscopy instrument (Figure 11). But before taking snaps of optical microscopy image the sample had been thoroughly mirror polished and etched and the micro structure had been taken from sample having following parameters. ? 1st type FSP had been carried out using following parameter (1) Tool traverse speed is 20mm/min with (2)720 rpm of traverse speed and (3) air cooling. ? 2nd type FSP had been carried out using the following parameters: tool traverse speed is (1) 30mm/min with (2) 720 rpm of traverse speed and (3) air cooling. Figure 14: Carl Zeiss optical microscope Sample preparation A sample was taken out from all three cast and also from the FSPed one. The FSP sample was taken out from a transverse section of the processing length. Initially sample was polished against different grit size paper of 150→300→800→1500→2000 after that mechanical cloth polishing had been done. Mechanical cloth polishing Cloth polishing was done in two steps i.e. rough and fine polishing. A variable speed wheel is preferred for polishing. The rough polishing was done with MgO. A teaspoon full of abrasive is applied at the Centre of cloth, moistened with distilled water then transformed into a paste. MgO solution in distilled water was used for final polishing on fine velvet cloth. After final polishing the samples were thoroughly cleaned and etched with Keller’s reagent having composition listed in Table 3. For 15 seconds.

1**Microstructural characterization was carried out using optical microscopy (Model: Leica DMI 200)**

(Figure 11). Under polarized light. Table 3: Composition of Keller's reagent Keller’s HNO3 HCl HF H2O Composition % 2.5 1.5 1 Rest 3.7.2 Scanning Electron Microscopy (SEM) Fine grains at stirred zone, segregation of reinforcing particles, wear surface analysis and the failure mode during tensile tests were evaluated by the fractography analysis of the fractured surface using ZEISS 51-ADD0048 scanning electron microscope and FESEM. Scanning was done at appropriate accelerating voltages for the best possible resolution. SEM of stirred zone, fracture surface and wear surface had been taken for analysis. 3.8 EVALUATION OF

1**MECHANICAL PROPERTIES The mechanical properties were evaluated by tensile testing, Vickers hardness measurements,**

and wear test. 3.8.1 Vickers hardness measurement Vickers hardness testing was performed on FIE–VM50 (Figure 1) to determine hardness values of all the specimens

38**with a load of 5 kg** for a **dwell time of 15 s.**

Prior to hardness testing, the samples were well polished and there should not be any scratches and stains. During hardness measurement, minimum 5 readings were taken. Figure 15: Vickers hardness tester 3.8.2 Tensile Testing

1**Tensile test was conducted at room temperature on standard size**

specimen’s Figure 16 of 25 mm gauge length using H25K-S

36**universal testing machine at a constant** strain rate **of 1.** 66×10-2 **mm/s** to calculate **the**

tensile strength, yield strength and ductility of the specimens. Figure 16: Different nano reinforced (1, 2, 3, 4), micro reinforced (6, 7, 8) and 6061-alloy (5, 6) tensile sample. 3.8.3 Wear testing The test had been done on a pin on disc arrangement, using a square sample of size (6mmX6mmX6mm) from stirred zone of a FSPed sample and cast samples were taken (list of the sample done for a wear testing table.) And perfectly glued on a stub of 6mm diameter, which was then fixed to the tool holder arrangement and the test had been carried out for 20 minutes and coefficient of friction and wear rate had been calculated. Details of the wear test conditions used in this study Pin material Al 6061 nano and cast composite with and without FSP of 4mmX4mmX6mm, Stub Figure 17 to fix specimen. ? Disc material EN36 steel with a hardness of 65 HRc. ? Sliding speeds: 1mm/Sec. ? Normal load: 10N, 20N. ? Sliding distance: 1200 m. Figure 17: Showing stub and wear sample Chapter 4 RESULTS AND DISCUSSION 4.1 MICROSTRUCTURAL INVESTIGATION 4.1.1 SiC Powder characterization Before the incorporation of nano and micro particle into the mould SEM had been performed on micro SiC and TEM on nano SiC to know its shape and size. During analysis of SEM (Figure 18(a) and TEM (Figure 18(b) it was found that the size of the micro SiC was 20-160 μm and nano SiC was 30-80 nm and crystallinity was maintained after analyzed. (a) (b) SiC (c) Figure: 18

22**(a) SEM image of** SiC **micro** size particle **(b)** TEM **image of**

Nanosize particle (c) EDx Nano size particle showing peaks of C, Si, and O 4.1.2 SEM images Microstructural investigation of cast sample of micro and nano reveals that, porosity was present after casting, during investigation, it was found that the amount of porosity was more in the nano as compared to that of micro reinforcement this was due to high surface area and low wettability of nano particles. Later it was confirmed by performing the Archimedes buoyancy test. The Figure 19 shows the porosity presented by the black arrow (A) nano and (B) micro, reinforced composite, an agglomeration of SiC has also been shown in Figure 19 with blue arrow and SiC particles with yellow arrow. In cast material, even after stir casting lots of agglomeration of SiC was present. The FE-SEM image also show that coarse grains are present on the order of 150-200μm. Figure 19: SEM image showing agglomeration, porosity in Cast (A) nano composite (B) micro composite. 4.1.3 Optical microstructure After FSP there is a significant reduction in grain size in both the castings in stirred zone, because of dynamic recrystallization comparing the two different parameters of FSP the one with forced air cooling gives better grain refinement which is shown in Figure 2 (b), this can be attributed because of reduced processing temperature during FSP the Figure 20 shows the nanocomposites after FSP (a) with air cooling and 20 mm/Sec traverse speed and (b) with forced air cooling and 30 mm/Sec. (a) (b) (c) (d) Figure 20: Optical Micrograph showing variation in grain size with changing processing parameter in FSP and SiC particles (in nano composites) (a) with traverse speed 20mm/min (b) traverse speed 30mm/min+air cooling. If we compare nanocomposites to that of micro, keeping the parameter constant Nano gives finer grain than micro which is attributed by the pinning effect of nano particles during grain growth In nanocomposites more grain refinement had taken place after FSP has been shown in Figure 21 (a, c) represents gasped micro-reinforced composite with traverse speed 20mm/min and (b, d) represents gasped nano-reinforced composite with composite with traverse speed 20mm/min. From the above microstructures it is clear that the grain refinement is taking place after FSP which leads to the improvement in mechanical property after FSP the homogeneous dispersion of particle has taken place as well as a huge reduction in porosity in both nano and microcomposites. In starting cast nanocomposites shows more defects and inferior micro structure than micro-reinforced composite but after FSP nano is showing distinguishing property which indicates the effectiveness of the FSP with cast nanocomposites. (a) (b) (c) (d) Figure 21: optical micrograph showing stirred zone (a,

22**c) micro composite and** (b, **d) Nano composite, in**

traverse speed 20 mm/min 4.1.4 XRD XRD of nano sample had been carried out for investigation of the presence of SiC nano particle in the nanocomposites to confirm the proper stirring and mixing during casting. Presence of SiC peak in the XRD pattern (Figure 22) confirms the incorporation of reinforcement in the matrix of 6061. Plane (101) and (104) of SiCp peaks had been identified during investigation using X-pert high score. Al(220) Figure 22: XRD pattern of Nano composite showing peaks of Al and SiC 4.2 MECHANICAL PROPERTIES Since this composite has an application in brakes, automobile frames and structures it should possess sufficient strength so that it can function well during its application, so to check its strength and tribological properties tensile test, Vickers hardness test and wear test had been carried out. 4.2.1 HARDNESS RESULTS The results are obtained after Vickers hardness test indicates the inferior property of cast nanocomposites with respect to microcomposites, as their respective value and comparison can be seen in Figure 27 which is due to porosity and agglomeration present in the sample but after FSP the nanocomposites gives better property than microcomposites with both processing parameters. Which indicates the removal and proper mixing of material. 31 Figure 23: Hardness of samples 4.2.2 Tensile test The tensile test had been carried out for the samples of nano and micro at its 2 different processing parameters. The Figure 23 shows the stress-strain relationship. The value of strength and ductility is tabulated in Table 4. Figure 24: Stress vs Strain curve of Nano(black line) and micro(red line) composite after casting. Table 4: Strength and ductility of nano and micro cast composite. 0.2% yield strength UTS (MPa) Ductility at fracture (%) Nano 64 77.08 1.49 Micro 65 87.6 2.46 As from the above Figure 23 and table it is clear that ductility is very less in both material, whereas as nano-reinforced composite shows inferior property than micro because the (a) (b) Figure 25: Stress vs Strain curve of (a) Nano (b) Micro composite in various conditions. presence of porosity and agglomeration in it. The Figure 24 (a) Shows the effect of processing FSP parameters in nanocomposites as well as comparison between FSP and the cast of the respective composite and Figure 24 (b) shows the effect of processing parameters in microcomposites as well as comparison with its cast one.

37**It is clear from the** above curves **(Figure** 24) **that** after FSP **there** is **a** huge **increment in**

strength and ductility in both the reinforcements this shows the effectiveness of the FSP in cast composites. Various strengths and ductility values are tabulated in Table 5 for nano and microcomposites. Table 5: Nano and microcomposites strength and ductility. SAMPLES 0.2% yield strength (MPa) UTS (MPa) Ductility at fracture% Micro-reinforced Nano-reinforced As cast 64 77.08 1.49 Air cooled 78.57 154.76 16.15 Forced air cool 75.67 170 19.13 As cast 65 77.08 1.49 Air cooled 60.9 120 14.7 Forced air cool 85.57 147 20.3 Out of performed two parameters of FSP they has an influential effect in strength and (a) (b) Figure 26: Stress strain curve of FSPed micro (black line) and nano (red line) composite with (a) traverse speed -20mm/min (b) traverse speed 30 mm/min +air cooling. ductility of the composite this can be viewed in the Figure 24 (a and b). In both the composite after FSP, nanocomposites shows superior property than microcomposites. This is due to the effective elimination of defects from nanocomposites 35 and the strengthen of nanocomposites is governed by dispersed nano ceramics which restricts the dislocation movement since in nanocomposites the gap between two consecutive particle is small, so large shear force is required to move the dislocation through it, in comparison to micro-reinforced, in the nanocomposites the operative strengthening mechanism is around strengthening which has an influential effect in strength of the material. 4.3 Fractograph results Fractography of tensile specimen had been carried out under SEM. Which reveals the mechanism of failure mode in the composite. Fractography had been done for micro and nano samples of FSP in both the parameters. The Figure 26(a) shows the fractography of nanocomposites at 500X resolution of air cooled sample, whereas Figure 26(b) shows the fractography of microcomposites at 500X resolution of air cooled sample. (a) (b) Brittle fracture containing fine faceted cleavage Micro voids Crack Brittle fracture Dimples containing SiC Figure 27: Fracture surface of (a) Nano Composite and (b) Micro Composite. In the both ductilmoodsod oa fracturere has been characterized which is indicated by the dimples formation. The cavity

16**arises from the** inclusion and **second phase particle** which **is** enlarged **by**

the continuous yielding and also the material between them is nicked and sheared. The mirror like faces showing cleavage which indicates the brittle manner of failure. 4.4 WEAR TEST Pin on disc wear test of the specimen reveals the extinguishing wear rate of the nano FSPed material the test had been conducted on micro and nanocomposites after FSP in , forced air cool and cast , nano and microcomposites. The wear results, shown in Figure 29, reveal that before FSP wear (mass loss) is more in Nanocomposites because of its less hardness value kg and, 2kg of load and their respective coefficient of friction vs. sliding distance has been shown in Figure 28 (a) and Figure 28 (b). And agglomeration of particles due to which “material as a whole” comes out with the specimen which increases the wear rate in composite. Figure 28 Curve showing wear loss mass at 10N and 20 N loading of various sample. (a) (b) Figure 29

42**Variation of coefficient of friction with sliding distance** of **various** samples with **(a)**

10N (b) 20N load applied. 4.5 WEAR SURFACE CHARACTERIZATION SEM image of wear surface in Figure 30(a) FSped nanocomposites (b) FSPed microcomposites (c) cast nanocomposites, (d) cast microcomposites, indicates the mood of fracture occurs in various samples. Adhesive wear occurs in all composites and the SiC which is an abrasive particle after coming out from the matrix creates abrasive grooves in the composite,

13**presence of deep scratches** on **the worn surface which indicated that wear progressed by an abrasive mechanism**

and can be seen in Figure 30 given below indicated by black arrow. Due to the adhesion between sample and disc material the lamina gets pulled out creating delaminated surface and grooves which is indicated by the yellow arrow. As further sliding between disc and a composite sample occurs, some of the SiCp have been pulled out acts

13**as an abrasive medium. The powdered SiCp along** Al **-oxides from the**

pin (sample) in combination acts as an abrasive media for wear. Close examination of sample reveals the formation of short cracks and shallow craters which is indicated by the white arrow. (a) Cavity formed by (b) delamination Wear Track Abrasive Groove Delaminated area Wear Track Delaminated area Abrasive grooves (c) (d) Wear Track Delaminated area Abrasive AdhesivWeear Track Wear done by wear track grooves abrasion and adhesion Figure 30: (a) FSped nano composite (b) FSPed micro composite (c) cast nano composite, (d) cast micro composite, indicates the mood of fracture occurs in various samples at 20N load. Chapter 5 CONCLUSIONS AND FUTURE SCOPE 5.1 CONCLUSIONS Micro- and nanocomposites were prepared using stir casting route and FSP used for modification of microstructure and for uniform distribution of particles added. Detailed investigation of microstructure and mechanical properties were made. On the basis of obtaining results and their analysis, the conclusions are as follows: 1. After stir casting, the porosity and inhomogeneous distribution found to be a major problem which decreased mechanical properties. The amount of porosity and agglomeration found to be more in nanocomposite, which

20**is due to the** large **surface area of nano** size **particles**

although same weight. % of particles used. This results in the inferior mechanical properties in nanocomposite as compared with microcomposites. 2. FSP drastically enhances the strength and ductility of micro- and nanocomposites due to grain refinement, homogeneous distribution and porosity elimination. 3. The higher traversed speed (30 mm/min) showed better mechanical properties than that processed at lower traversed speeds (20 min). In case of nanocomposite, the FSP with 35 mm/Sec traverse speed and forced air cooling resulted better UTS (170MPa) and ductility (20%) compare to that of the microcomposite produced at same traversed speed. It is because of better grain size refinement due to less heat input and processing temperature at the time of FSP and more the pinning effect of nano size particles (as the number of particles are more for same wt.% In nanocomposites). The similar effect was observed in case of hardness

18**and wear test** also. **The hardness and wear** resistance of **the**

FSPed nanocomposite (with 35mm/Sec traverses speed and forced air) found to be more than the others. 4. The strengthening mechanisms involved in the composites are grain size refinement and Orowan strengthening due to particle reinforcement. The effect of grain size refinement is more in case of nanocomposite than microcomposite because of more pinning effect of nano size particles. The influence of Orowan strengthening is also more effective in case of nanocomposite because of less inter particle spacing available. 5. The fractography analysis showed presence of dimples in both samples; microcomposite showed larger size deeper dimples compare to that of nanocomposite. This indicates the nucleation and growth of voids occurred from the second phase SiC particle. The wear surface analysis showed both adhesive and abrasive wear mechanisms. 5.2 FUTURE AND SCOPE OF THE STUDY ‣ Further improvement in mechanical properties can be done using other parameters of FSP by optimizing between machine variables and processing temperature. ‣ The results can be used to compare the properties of this composite with other reinforced composite. ‣ Corrosion behavior of the composite can also be studied. REFERENCES [1] E. A. Starke and J. T. Staley, “Application of modern aluminum alloys to aircraft,” Prog. Aerosp. Sci., vol. 32, no. 2–3, pp. 131–172, 1996. [2] M. Avalle, “Casting defects and fatigue strength of a die cast aluminium alloy: a comparison between standard specimens and production components,” Int. J. Fatigue, vol. 24, no. 1, pp. 1–9, Jan. 2002. [3] R. L. Deuis, C. Subramanian, and J. M. Yellup, “Abrasive wear of aluminium composites—a review,” Wear, vol. 201, no. 1–2, pp. 132–144, Dec. 1996. [4] Y. Sahin, “Preparation and some properties of SiC particle reinforced aluminium alloy composites,” Mater. Des., vol. 24, no. 8, pp. 671–679, Dec. 2003. [5] W. H. Hunt, Jr., “Aluminum Metal Matrix Composites Today,” Mater. Sci. Forum, vol. 331–337, pp. 71–84, 2000. [6] D. MIRACLE, “Metal matrix composites – From science to technological significance,” Compos. Sci. Technol., vol. 65, no. 15–16, pp. 2526–2540, Dec. 2005. [7] S. A. Sajjadi, M. Torabi Parizi, H. R. Ezatpour, and A. Sedghi, “Fabrication of A356 composite reinforced with micro and nano Al 2O3 particles by a developed compocasting method and study of its properties,” J. Alloys Compd., vol. 511, no. 1, pp. 226–231, 2012. [8] W. Wang, Q. yu Shi, P. Liu, H. ke Li, and T. Li, “A novel way to produce bulk SiCp reinforced aluminum metal matrix composites by friction stir processing,” J. Mater. Process. Technol., vol. 209, no. 4, pp. 2099–2103, 2009. [9] R. S. Mishra, Z. Y. Ma, and I. Charit, “Friction stir processing: A novel technique for fabrication of surface composite,” Mater. Sci. Eng. A, vol. 341, no. 1–2, pp. 307–310, 2003. a. Dolatkhah, P. Golbabaei, M. K. Besharati Givi, and F. Molaiekiya, “Investigating effects of process parameters on microstructural and mechanical properties of Al5052/SiC metal matrix composite fabricated via friction stir processing,” Mater. Des., vol. 37, pp. 458–464, 2012. [11] K. J. Al-Fadhalah, A. I. Almazrouee, and A. S. Aloraier, “Microstructure and mechanical properties of multi-pass friction stir processed aluminum alloy 6063,” Mater. Des., vol. 53, pp. 550–560, 2014. [12] D. C. Hofmann and K. S. Vecchio, “Submerged friction stir processing (SFSP): An improved method for creating ultra-fine-grained bulk materials,” Mater. Sci. Eng. A, vol. 402, no. 1–2, pp. 234–241, 2005. [13] T. Nagaoka, Y. Kimoto, H. Watanabe, M. Fukusumi, Y. Morisada, and H. Fujii, “Friction stir processing of a D2 tool steel layer fabricated by laser cladding,” Mater. Des., vol. 83, pp. 224–229, Oct. 2015. [14] I. Dinaharan, R. Sathiskumar, and N. Murugan, “Effect of ceramic particulate type on microstructure and properties of copper matrix composites synthesized by friction stir processing,” J. Mater. Res. Technol., Apr. 2016. [15] S. Jana, R. S. Mishra, J. A. Baumann, and G. Grant, “Effect of process parameters on abnormal grain growth during friction stir processing of a cast

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