

# **Development and Characterization of Al-E Glass Fiber Composites**

**A Thesis submitted in partial fulfillment  
of the requirements for the Degree**

*of*

**Master of Technology (M. Tech.)**

*in*

**Metallurgical & Materials Engineering**

*by*

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**2015**



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**Certificate**

This is to certify that the project entitled “**Development and Characterization of Al-E glass Fiber Composites**” submitted by **Mr. Dipanshu Verma** in partial fulfilments for the requirements for the award of **Master of Technology** Degree in **Metallurgical & Materials Engineering** at **National Institute of Technology Rourkela** (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the report has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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## **Acknowledgement**

I would like to thank **NIT Rourkela** for giving me the opportunity to use its resources and work in such a challenging environment. First and foremost, I take this opportunity to express my deep regards and sincere gratitude to my guide **Prof. S.N.Alam** for his able guidance and constant encouragement during my project work. This project would not have been possible without his help and the valuable time that he has given me amidst of his busy schedule.

I would like to express my utmost gratitude to **Prof. S.C.Mishra, Head of the Department, Metallurgical& Materials Engineering**, for allowing me to use the departmental facilities.

I would also like to extend my heartily gratitude to my friends Lailesh kumar ,Harshpreet Singh Deepankar panda and Nidhi Sharma and senior students of this department who have always encouraged and supported in doing my work. Last but not the least, I would like to thank all the staff members of the Department of Metallurgical and Materials Engineering who have been very cooperative to me.

**Dipanshu Verma**

**Place: NIT Rourkela**

# Contents

<b>Certificate</b>	<b>i</b>
<b>Acknowledgement</b>	<b>ii</b>
<b>Contents</b>	<b>iii</b>
<b>List of Figures</b>	<b>v</b>
<b>List of Tables</b>	<b>vii</b>
<b>Abstract</b>	<b>viii</b>
<b><i>Chapter 1 Introduction</i></b>	<b>1</b>
1.1- Motivation and Background of the Present Investigation	2
1.2- Polymer matrix composites (PMCs), Ceramic matrix composites (CMCs) and Metal matrix composites (MMCs)	4
1.3-Powder Metallurgy	5
1.4-Aluminium (Al)	6
1.5-Aluminium metal matrix composite	7
1.6- E-glass fibre	8
1.7 Scope and Objective of the Work	9
<b><i>Chapter 2 Literature Review</i></b>	<b>11</b>
2.1 Composites	12
2.2 Metal Matrix Composites	12
2.3 Al-Based Metal matrix Composites	13
2.4-Al-Eglass fibre composites	14
2.5 Processing Techniques for Metal Matrix Composites	14
2.5.1 Liquid State processing Techniques	14
2.5.1.1 Infiltration Process	15
2.5.1.2 Dispersion Process	15
2.5.1.3 Spray Process	16

2.5.1.4 In-Situ Process	16
2.5.2. Solid State processing Techniques	16
2.5.2.1 Diffusion Bonding	17
2.5.2.2 Powder Blending and Consolidation	17
2.5.2.3 Mechanical Alloying	18
2.6-Conclusion	19
<b>Chapter 3 Experimental Details</b>	20
3.1- Equipment used in the present investigation	22
3.2-Selection, Synthesis, and characterization of raw material	29
1-Al powder	29
2-E-glass fibre	30
<b>Chapter 4 Result and Discussion</b>	32
4.1-Mechanical milling of Al	33
4.2-Al-E-glass fibre composites	41
<b>Chapter 5 conclusion</b>	42
<b>Reference</b>	

## List of Figures

Figure No.	Figure Description	Page No.
<b>Chapter 1 Introduction</b>		
Fig.1	Use of composite material	3
Fig.2	The sequence of operation in powder metallurgy process	6
<b>Chapter 2 Literature Review</b>		
Fig.3	Metal Matrix Composites used for various applications	13
Fig.4	shows the process of squeeze casting infiltration	15
Fig.5	Ball–Powder- Ball collision during mechanical alloying	18
<b>Chapter 3 Experiment procedure</b>		
Fig.6	work plan for the present investigation	21
Fig.7	(a) Planetary Ball Mill (b) Schematic view of mechanism of ball milling	23
Fig.8	Schematic Diagram of Uniaxial Pressing	23
Fig.9	Uniaxial Hydraulic Press	23
Fig.10	X ray Diffraction	24
Fig.11	Tubular furnace	25
Fig.12	Scanning Electron Microscopy(SEM)	26
Fig.13	Transmission Electron Microscopy(TEM)	27
Fig.14	Density Measurement	28
Fig.15	Vickers Hardness Tester	29
<b>Chapter 4 Result and discussion</b>		
Fig.16(a-b)	X-ray diffraction plot of Al milled for various periods of time	33
Fig.16(c)	Variation of crystallite size with milling time	33
Fig.16(d)	Variation of strain with milling time	33
Fig.17(a)	HRTEM image of 20h milled Al	34
Fig.17(b)	SAD pattern of 20h milled Al	34
Fig.18(a-c)	Optical images of E-glass fibre used in composite	34
Fig.19(a-c)	Optical image of unmilled Al-E glass fibre composites for different vol. % (1,2and 5%) having 2mm length of E-glass fibre	35

Fig20(a-c)	Optical image of unmilled Al-E glass fibre composites for different vol. % (1,2and 5%) having 5mm length of E-glass fibre	36
Fig.21(a-c)	optical image of unmilled Al-E glass fibre composites for different vol. % (1,2and 5%) having 8mm length of E-glass fibre	36
Fig.22(a-c)	SEM images of unmilled Al-1, 2, and 5 vol. % E-glass fibre composites having fibre length of 2 mm as reinforcement	36
Fig.23(a-c)	SEM images of unmilled Al-1, 2, and 5 vol. % E-glass fiber composites having fiber length of 5 mm as reinforcement	37
Fig.24(a-c)	SEM images of unmilled Al-1, 2, and 5 vol. % E-glass fiber composites having fiber length of 8 mm as reinforcement	37
Fig.25(a-b)	Relative Density and Vickers's hardness plot of various sintered unmilled Al-E-glass fiber composites at different vol. % having different length of E-glass fiber as reinforcement	38
Fig.26(a-c)	SEM image of the fracture surface of (a) unmilled Al- 1 vol. % E-glass fiber unmilled (b) Al- 2 vol. % E glass fiber unmilled (c) Al- 5 vol. % E glass fiber having fiber length of 8 mm as reinforcement	39
Fig.27(a-c)	Optical image of milled Al-E glass fiber composites for different vol. % (1,2and 5%) having 8mm length of E-glass fiber	39
Fig.28(a-c)	SEM images of milled Al-1, 2, and 5 vol. % E-glass fiber composites having fiber length of 8 mm as reinforcement	40
Fig.29(a)	Vickers's hardness plot of various sintered milled Al-E-glass fiber composites at different vol. % having 8mm length of E-glass fiber as reinforcement	41

### List of Tables

<b>Figure No.</b>	<b>Table description</b>	<b>Page No.</b>
1.1	Properties of different fibers	9
3.1	Properties of Al	30
3.2	Properties of E glass fiber	31
3.2	Different Fiber length and its aspect ratio	35



## Abstract

Metal Matrix Composites (MMCs) have brought a keen interest in current scenario for potential applications in automotive industries owing to their superior strength to weight ratio. In the present work a modest attempt has been made to develop aluminium (Al) based E-glass fibre composites with an objective to produce economic method of obtaining high strength MMCs. E-Glass fibers are the most widely used glass fibers as reinforcement in composites. E-glass fibers show proficient bulk properties such as high hardness (6000 MPa), high strength, dimensional stability, resistance to chemical attack. It has density of 2.58 gm/cc, tensile strength of 3500 MPa and Young's modulus of 85 GPa along with compressive strength of 5000 MPa. The present work investigates the microstructure and hardness of an Al-based MMC reinforced with different (1, 2, 5) vol. % of E-glass fiber developed by powder metallurgy route. The E-glass fiber having different length (2, 5 and 8 mm) were chosen for each composition. Both unmilled and 20h milled nanostructured Al were used as matrix and mixed with E-glass fiber in different vol. % and compacted under uniaxial load of 222 MPa and sintered at 500°C for 2h in Ar atmosphere. For a period of upto 20 h Al was milled in a high energy planetary ball mill in order to develop nanocrystalline Al powder. The milled Al powder was analyzed using scanning electron microscope (SEM), x-ray diffraction (XRD), and high resolution transmission electron microscope (HRTEM). The effect of nanocrystalline Al on densification and sintering was analyzed. The microstructure of the various Al based MMCs were analyzed using SEM and optical microscope. The hardness of the various composites was measured using a Vickers microhardness tester. SEM was used to analyse the fracture surface of the composites. The relative density of sintered samples was calculated from Archimedes' principle.

**Keywords:** Al-matrix composites; Metal Matrix Composites (MMCs); E-glass fibre; Microstructure;

# **CHAPTER 1**

## **INTRODUCTION**

## **Motivation & Background**

Mankind has tried to search material which can perform under adverse environmental conditions. Researchers have tried to develop materials that have the desired properties. This is also the main reason behind the creation of composite materials. In composites one of the components is the homogenous matrix and the other is reinforcement which is stronger and stiffer. Composites are used extensively for making prototype parts because different types of shape can be fabricated quickly and inexpensively. Composites have many advantages for the design of structural devices. It has high stiffness, strength and toughness compared to metal alloys. Many structures experience fatigue loading, i.e., and as a result the internal stress varies with time. The composite materials are light as well as strong and it is their major advantage compared to metals and alloys. Fatigue stress leads to failure of the component. Composites have excellent fatigue resistance compared to metals and alloys. Composites also have excellent tribological property and its sliding friction could approach those of lubricated steel. Particles or fibres fixed in matrix are the best example of composite materials which are generally useful in structural and automotive applications where the reinforcement material generally uses the load and provide the desirable properties. The property of these composites is determined by the property of reinforcement. Fig.1 shows the different types of parts in the aircraft manufactured by composite materials [1-6].

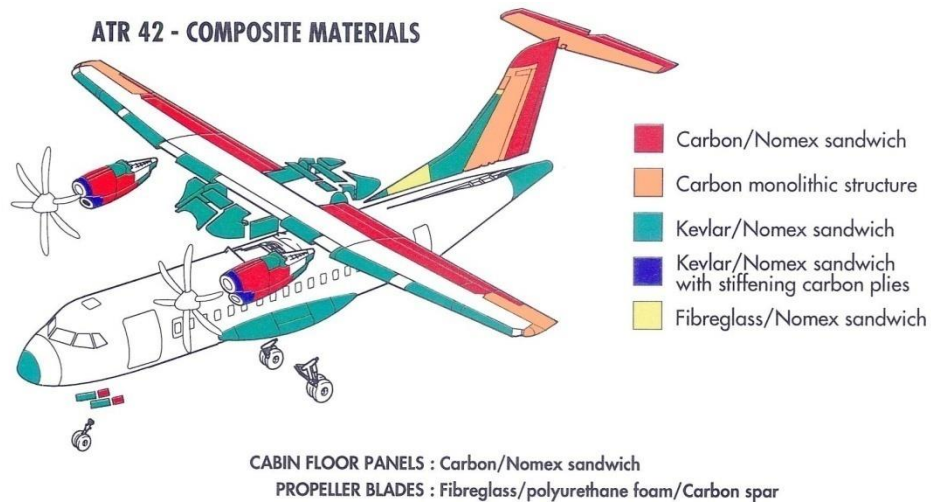


Fig.1 Use of composite materials in aircraft

These reinforcements or filler materials have various objectives according to the application parameters. The condition here is the progress in the resultant component properties. There are various objectives for improvement of MMCs which are reported as:

- Increment in Yield Strength
- Increment in Tensile Strength
- Increment in Creep Resistance
- Increment in Fatigue Strength
- Increment in Thermal shock Resistance
- Increment in Young's Modulus
- Increment in Corrosion Resistance

## **Reinforced Composites: Polymer matrix composites (PMCs), Ceramic matrix composites (CMCs) and Metal matrix composites (MMCs)**

Polymer matrix composites are composed of wide range of continuous or short fibres bound together by an organic polymer matrix. PMC constitutes two phases; one is the matrix and the other is the reinforcement. Mainly two types of matrix are use, thermosets and thermoplastics. For enhanced properties we use fibers such as carbon, boron, andaramid.The reinforcement in the PMC provides good strength and stiffness. The function of matrix is binding the reinforcement and transfer the load between them. The properties of PMCs mainly depend on the matrix, the reinforcement, and the interphase. PMCs used in a wide variety of industries such as civil , aerospace, sports, tanks and vessel in the chemical industries etc. Ceramic matrix composites (CMCs) have properties like good strength and high stiffness at high temperature, low density and chemical inertness etc. However CMCs have low toughness. CMCs find application in many areas like aerospace because it has high specific strength and stiffness leading to reduce the weight, decrease fuel consumption, longer service life and reduction in maintenance cost. Another important application of CMCs is cutting tool inserts because of their good abrasion resistance, thermal shock resistance and high strength. Metal matrix composites (MMCs) have come out as a useful innovation which has a very wide range of application. This material group can be used for constructional and functional purposes In the modern generation MMCs have created a varied interest in research because of their properties like stiffness , high strength, fracture toughness and lightweight which are far better as compared to the polymer matrix composites (PMCs). They can positively resist elevated temperature in corrosive atmosphere in comparison to PMCs. The MMCs have better mechanical properties and have a wider temperature range of application compared to the polymer matrix composites.The basic properties of fiber reinforced metal matrix mixtures are determined by the fibre content, fibre orientation, fibre

material, fibre length and the distribution of fibre in the composites. Military and aerospace industries are the chief users of metal matrix composites. Primarily MMC components are used in jet engines, aircraft and missiles. In Japan, an annual estimate of selling 300,000 such pistons is reported. It can be concluded by the market reports that the consumption of composite materials for several applications for MMCs is not that expensive when viewed with respect to their application. Metal matrix composites contain metals, compounds and alloys of metals such as copper (Cu), aluminium (Al), magnesium (Mg) reinforced with ceramic particulate, fibres or whiskers. The reinforcement is a vital role for deciding the economic value and mechanical properties for proper application. MMCs give more prominent mechanical properties because it has high modulus and quality [7-10].

### **Powder Metallurgy**

For the development of MMCs, one of the recent material technologies used is powder metallurgy. Powder metallurgy is a material processing technique used to develop new materials and parts by the sintering process. In Powder metallurgy, the sequence of processes is blending, compaction and sintering. Powder metallurgy are mainly used in four areas such as: (i) when we use high melting point metals (like tungsten, molybdenum and tantalum), (ii) porous materials such as oil impregnated bearings and filters can be developed by this process, (iii) it has excellent economic efficiency and (iv) it is an excellent method for developing composites [11,12].

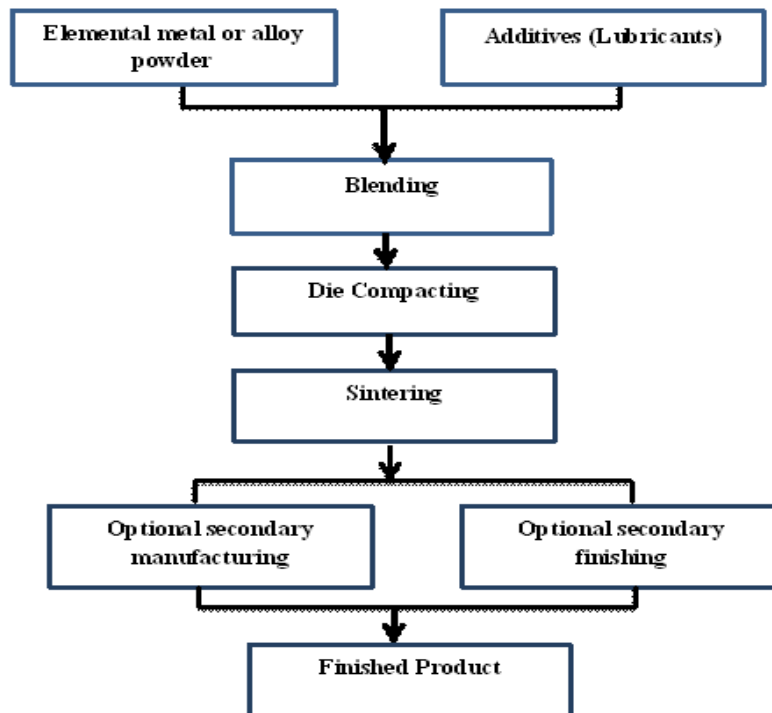


Fig.2 The sequence of operation in powder metallurgy process

### Aluminium (Al)

The modulus of elasticity of aluminium (Al) is 69 GPa. The tensile strength of pure Al is around 90 MPa. Its density is 2.70 gm/cc. It is highly malleable and has good machinability. Aluminium is readily available and has a low cost as compared to other materials. Al can be easily fabricated and it can be easily blended with two different materials with a clear interface. Al shows following advantages over other materials:

Advantages of MMCs	Disadvantages of MMCs compared to PMCs
<ul style="list-style-type: none"> <li>-High toughness</li> <li>-High transverse strength</li> <li>-High damage tolerance</li> <li>-Improved environment Resistance</li> <li>-High thermal and electrical conductivity</li> </ul>	<ul style="list-style-type: none"> <li>-Less advanced technology</li> <li>-Less data base properties</li> <li>-Higher cost</li> </ul>

Table-1.1

The fibre reinforced composites have these fundamental highlights:

- Variability of quality
- Decrease of wear and drag
- Anisotropic properties
- Enhanced wear quality

Innovation in composite materials offers an interesting chance to modify the properties of aluminium. This could provide enhanced quality, lesser weight, and high service temperature, enhanced wear resistance, higher versatile modulus, controlled coefficient of thermal extension, and enhanced fatigue properties [13-15].

### **Al-Matrix Composites (AMC)**

Al-matrix composites belong to the cluster of sunshine weight, high performance metallic element matrix system. AMCs were being used for the high tech-functional and structural application including automotive, aerospace, defence and sports etc. The major advantages of AMCs can be given as follows –

- Increased strength
- Enhanced stiffness
- Greater high temperature properties
- Improved abrasion and wear resistance
- Improved damping capability

AMCs are utilised in the mechanical, non-mechanical, and practical application in various areas due to their increased performance, greater economic value and environmental profits. The major benefits of the AMCs in the sector of transportation are less noise generation and lesser fuel intake. AMCs provide superior properties and can be used as a replacement for current materials. AMCs can enable necessary changes in design of products. Besides, by



using selective-reinforcement techniques and near-net shape forming AMC's can deal economically possible output for a various range of available commercial applications. Latest development in military and commercial importance of AMC's are much constructed on such advanced changes seen in the constituent design.

### **Types of AMC:**

1. Particle reinforced AMC
2. Whiskers or-short fibre reinforced AMC
3. Continuous fibre reinforced AMC
4. Mono-filament reinforced AMC

The achievement of Al-matrix composites is extremely charged by the control and the upgrade of the interfaces between the Al-matrix and the reinforcement. A decent wetting is important to encourage the manufacture, particularly when utilizing fluid state system with low weight..

### **Application of AMC's:**

- Automotive sector
- Aerospace application
- Electronic and electrical application
- Sports and leisure application

### **E-Glass Fiber**

Glass fiber are the best industrial material at present. They can be made freely from raw materials, existing in abundance. They show better properties like resistance, transparency, prevention to chemical attack, stability, inertness, and fibre properties like stiffness, flexibility, and strength. Glass fibres area unit is divided in two classes, low cost all-purpose

fibres and premium special-purpose fibers. More than 90% of all glass fibers are unit general purpose products and 10% percent use for the special purpose

<b>Fiber</b>	<b>Forming temperature</b>	<b>Liquid us temperature</b>	<b>Softening temperature</b>	<b>Annealing temperature</b>	<b>Straining temperature</b>	<b>Bulk density, annealed g/cm<sup>3</sup></b>
Boron-containing E-glass	1160–1196	1065–1077	830–860	657	616	2.54–2.55
Boron-free E-glass	1260	1200	916	736	691	2.62

Table -1.2

### **Scope and Objective of the Present Work**

By the addition of large volume fraction of fibre and particulate reinforcement, the performance of metal matrix composites can be enhanced. An example is the development of Aluminium matrix composites by the adding of various fillers such as E-glass fiber to study their effect and change in properties in the matrix metal. The objectives of the present investigation are:

1. Al powder is milled in the high energy planetary ball mill, in order to develop nanostructured Al powder. The milling characteristics of Al were studied using various techniques like high resolution transmission electron microscope (HRTEM), X-ray diffraction and scanning electron microscope (SEM).
2. Both 20 h milled nanostructured Al and as-received Al were used as matrix for growth of the Al-E-glass fiber composites. Its composites were established by powder metallurgy method. The outcomes of using nanostructured Al on the properties of the Al-E-glass fiber composites was analysed.
3. Al-E-glass fibre composites having different vol. % (Al-1, 2 and 5 vol. % of E-glass fiber) and different lengths of E-glass fibre (2, 5 and 8 mm) as reinforcement were

produced through powder metallurgy method. The different properties of the produced composites were studied.

# **CHAPTER 2**

# **LITERATURE REVIEW**

## **Composites**

Mixture of various materials which results in good properties than those of the specific individual components used alone is known as composite materials. The two elements are reinforcement and a matrix. When compared with bulk materials the composite materials have an advantage of high stiffness and strength. The reinforcement is stronger, harder and stiffer than the matrix in most of the cases and provides strength and stiffness to the material. It can be a fibre or a particulate. Particulate reinforcements can be spherical, regular or can have irregular geometry. Particulates composites are weak and less stiff than the fibre composites but are less expensive.. The advantages of composites are many such as light weight, the ability to modify the layup for optimum stiffness, strength and fatigue. There are several characteristics of composites such as:

- Great specific strength, high modulus, increased strength and fatigue damage tolerance.
- Anisotropic.
- Enhanced Corrosion resistance and durability.
- More exceptional functional stuffs such as damping, low CTE (coefficient of thermal expansion).

## **Metal Matrix Composites**

Metal matrix composite (MMC) consists of two elements one is matrix and the other is reinforcement. Metal matrix composites are unique materials for used in structural application such as automotive and aerospace industries because high strength and thermal stability. Metal matrix composites exist with the combination of both metallic properties with ceramic properties and possess higher strength in shear and compression and provide service at high temperatures. Nowadays nano composites are gaining popularity in the material

industry replacing the monolithic metals. The comparison of microstructural and mechanical attributes of nano composites and micro composites is a hot area of research. There has been an increase in wide use of MMCs in aircraft and automotive industry for structural applications due to the cost effective processing routes [16-18].

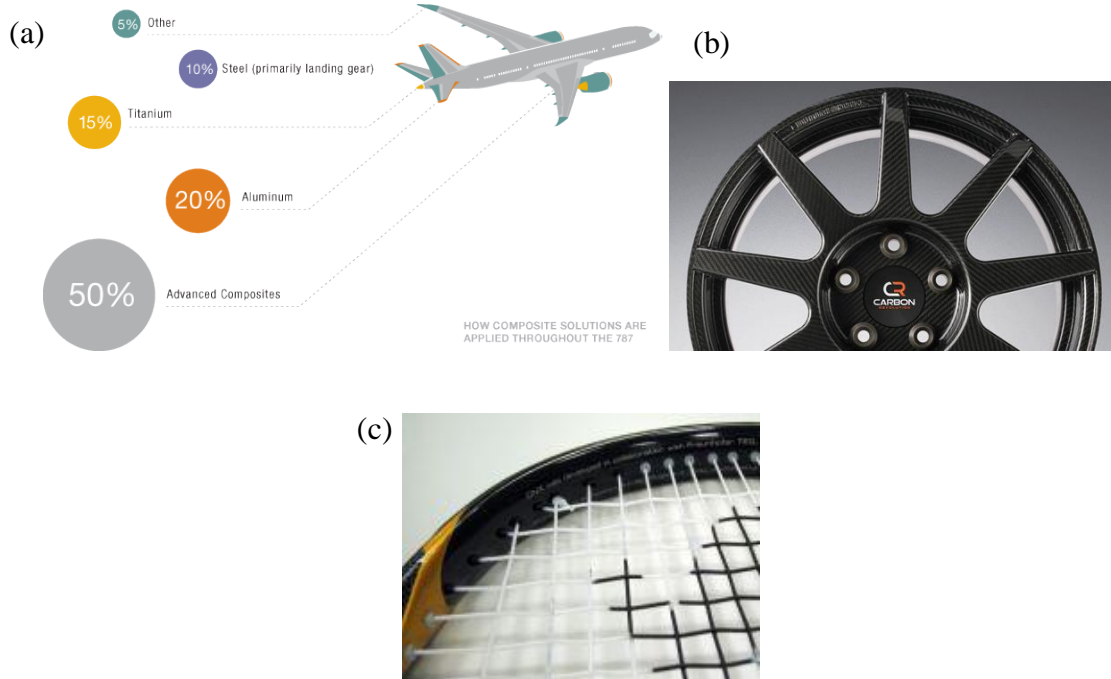


Figure3(a-c) Metal Matrix Composites used for various applications

## Al - Matrix Composites

Structural parts developed from aluminium and its alloys are of great importance to industries and are important in structural materials. When compared to metal matrix and organic composites, aluminium metal matrix composites are simpler to use and easier to fabricate. The commercial possibilities of aluminium based metal matrix composites have been recognized for many years and extensive research was conducted into the properties and in-flight behaviour of these alloys, and these efforts resulted in the successful use of the material in the composite systems. Interest remained in aluminium metal matrix composites because of their outstanding processing characteristics and specific stiffness. The metal matrix

composites are weldable, and can be formed, machined, and brazed like conventional aluminium metal components.

### **Al-E-Glass Fiber Composite**

Glass fibers are mostly made by using melt spinning techniques. The composition of E– glass fibers are  $\text{SiO}_2$  54wt%,  $\text{Al}_2\text{O}_3$  14wt%,  $\text{CaO}+\text{MgO}$  22wt%,  $\text{B}_2\text{O}_3$  10wt%, and  $\text{Na}_2\text{O}+\text{K}_2\text{O}$  lesser than 2wt%. Some additional materials could be present as impurities too. Glass fibers are used for numerous ranges of applications. Glass fibers have sensible arrangement of properties from high strength to refractive resistance. Fibers could be a dimensionally stable material, studied in a engineering methods for the covered fabrication process of polymer-based composite elements by using short and discontinuous fibers as reinforcement material. It had been reportable for that ecological attack by wet will debase the standard of the fibre [19].

### **Processing Techniques for Metal Matrix Composites**

Two type of processing methods are used which are:

1. Liquid State Processing Techniques
2. Solid State Processing Techniques

#### **2.5. Liquid State processing Techniques**

It has the ability of treatment the liquid metal more associated to the powder. There is minor expense in finding fluid metals than to metal powder and it has probability of creating different shapes by receiving a few systems in casting industry. Then again fluid state handling experiences various variables that incorporate partial control of the handling specifications and some unwanted chemical responses take place at the boundary of the reinforcement and the liquid metal [20, 21].

### 2.5.1.1 Infiltration Process

The liquid mode infiltration comprises infiltration of a fibrous or particulate filler by a liquid metal. The procedure for infiltrating MMCs is not easy and straight forward primarily due to issues with wetting of the reinforcement by the liquid metal. While the infiltration procedure of a fiber preform there is a probability of responses between the liquid metal and the fiber which fundamentally debase the attributes of the fiber. But it can be disadvantageous if the fibre coating is uncovered to air earlier to infiltration since there could be phenomena of surface oxidation will take place.

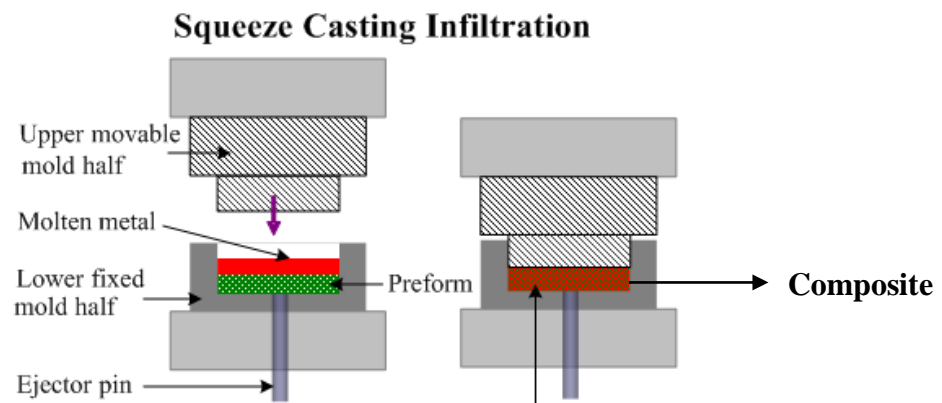


Figure 4 Schematic diagram showing the squeeze casting infiltration process

The figure shows the pressure less liquid metal infiltration process of developed MMCs. It can be used along with reactive metal alloys like Al- Mg to infiltrate ceramic preforms.

### 2.5.1.2 Dispersion Process

The reinforcement is combined in unattached form into the metal matrix in this method. The mechanical force is crucial to mix the two phases together by the method of stirring as most of the systems don't have good wettability. The benefit of dispersion method is its small cost and moreover it can often be managed by extrusion. The main limitation of this method is that the presence of continuous deformation due to gas penetration throughout combining and



reaction process between the matrix and reinforcement because of greater contact time and clustering throughout combining [22, 23].

### **2.5.1.3 Spray Process**

Particle strengthened MMCs can also be developed by the methods that involve the utilization of non-continuous spray forming procedures that solely could be used to turn out monolithic alloys. The particular sample of this procedure could be a co – spray method that utilizes a shower gun to atomize the liquid atomic number 13 composite matrix within that warm carbide particles area unit infused. For the procedures to be effective a perfect particle size is desired for stubbles area unit which are too fine to be changed. This method is sort of quick and automatic however it's noted that it's a liquid based process. Carbide particles of a side quantitative relation (length/diameter) between 3–4 and volume fractions up to 20 can be incorporated into metallic alloys. The method has a positive point that the flexibility it provides in developing differing kinds of composites is better than other methods. On the contrary the method is sort of costly owing to expensive capital instrumentation [24, 25].

### **2.5.2 Solid State Processing Techniques**

In solid state process techniques, the MMC can be developed as results of bonding among the matrix metal components and therefore the distributed reinforcement section because of mutual diffusion found between them at elevated pressure and temperatures. Strong state manufacturing at high temperature diminishes unwanted reaction on the boundaries of matrix and reinforcement stages once contrasted with fluid state infiltration procedures. Metal matrix composites obtained by this procedure can further be twisted, rolled and extruded. Distortion of sintered composite materials in scattered stage with short fibre brings about a favoured orientation.

### **2.5.2.1 Diffusion Bonding**

It is usually employed solid state handling method for connection alike - unlike metals. At raised temperatures the lay dispersion of molecules in metals results in bonding. The first points of interest of this procedure area unit are the ability to vary variations of metal matrix and management of fibre introductions and volume fractions. For metal matrix mixtures vacuum hot pressing is a vital step. In its place of uniaxial pressing, hot isostatic pressing (HIP) can likewise be utilised. In HIP, gas weight merges with the composite. With HIP it's something however tough to use high weights at raised temperatures with versatile geometries. The disadvantage of the method is high process temperature, long interval and pressures. Because of want of high process temperature and pressure the method becomes expensive [26].

### **2.5.2.2 Powder Blending and Consolidation**

The short fibre reinforced composites or particulate can be fabricated through powder metallurgy process. This generally contains cold pressing followed by sintering, or hot pressing to create whisker or particle reinforced MMCs. The matrix and the reinforcement are mixed together to develop green sample which is relatively dense to be controlled. The green sample was hot pressed or sintered, isostatically or uniaxially pressed to develop a dense composite. During this process dynamic recrystallization occurs at the interface between the reinforcement and the matrix [27, 28].

### 2.5.2.3 Mechanical Alloying (MA)

During mechanical alloying (MA) a powder blend is put into the ball process and is subjected to high-energy impact from the ball. Earlier this process was used to improve oxide-dispersion strengthened (OD). This method was initially proposed by Benjamin and his co-workers at the International Nickel Company in the late 1960s. Whereas high-energy milling of the powder particles are frequently planar, broken and cold welded. In lower matter holdings the balls can crash perpetually with some of powder particles that get caught within the periphery of the balls. Ordinarily rough one thousand particles with a approximate weight of zero. Two mg are unbroken throughout every impact of balls. Within the mechanical alloying includes perennial attachment and cracking of powder substrates in an exceedingly high energy mill. This system could effectively create fine, uniform dispersion of oxide particles ( $\text{Al}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{ThO}_2$ ) in nickel-base super alloys which were unrealistic to be produced by more traditional powder metallurgy systems. This development has changed the traditional system where the material was created by high temperature combination MA is also a medium of inducing phase transformation processes like amorphization or polymorphic transformations of compounds, disordering of ordered alloys, etc. in the milled powder.

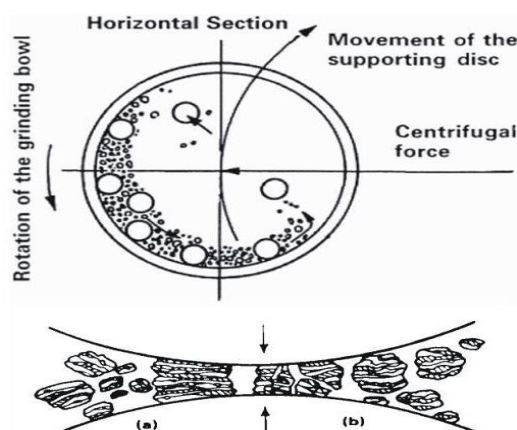


Figure 5 Ball-Powder-Ball collision during mechanical alloying

During mechanical alloying the force at the time of collision can plastically deform the powder particles leads to fracture and work hardening. During this procedure new surfaces are created. Cold welding between the particles also takes place which leads to the increment of the size of particle. Within the starting phases of process the particles area unit softens and their affinity to weld and being structured as the larger molecule is high. A mixture of particle sizes creates with a number of them as intensive as thrice larger because the 1st particle. When proceeded with deformation, the particles get worked up as solid and break by a fatigue failure mechanism. Sections formed by this procedure could stick with it change in size while not solid agglomerating power. At this stage, the attraction to fracture is greater to the cold welding. With the prolonged continuous influence of crushing balls, the construction of the atoms also gets refined [29, 30].

## **2.6-Conclusions**

A comprehensive literature review has been done. The research findings of several researchers in this area have been studied and their conclusive findings have been recorded. In the literature survey the study on several processing techniques for metal matrix composites has been carried out to report the advantages and limitation of each of the processing technique. Study has been done in reference with Al metal matrix composites. Several mechanical and physical properties of Al-based metal matrix composites have been thoroughly analyzed on the work conducted by various researchers.

# **CHAPTER 3**

## **EXPERIMENTAL DETAILS**

# Experimental procedure

## Introduction

This part characterizes the experimental method utilized as a part of the present investigation. The instrument utilized as a part in the project is listed below along with their particulars and specifications in details. A complete information is given on the raw material acquired and made which is utilized for the manufacture of the Aluminium based metal matrix mixtures. The accompanying work arrangement has:

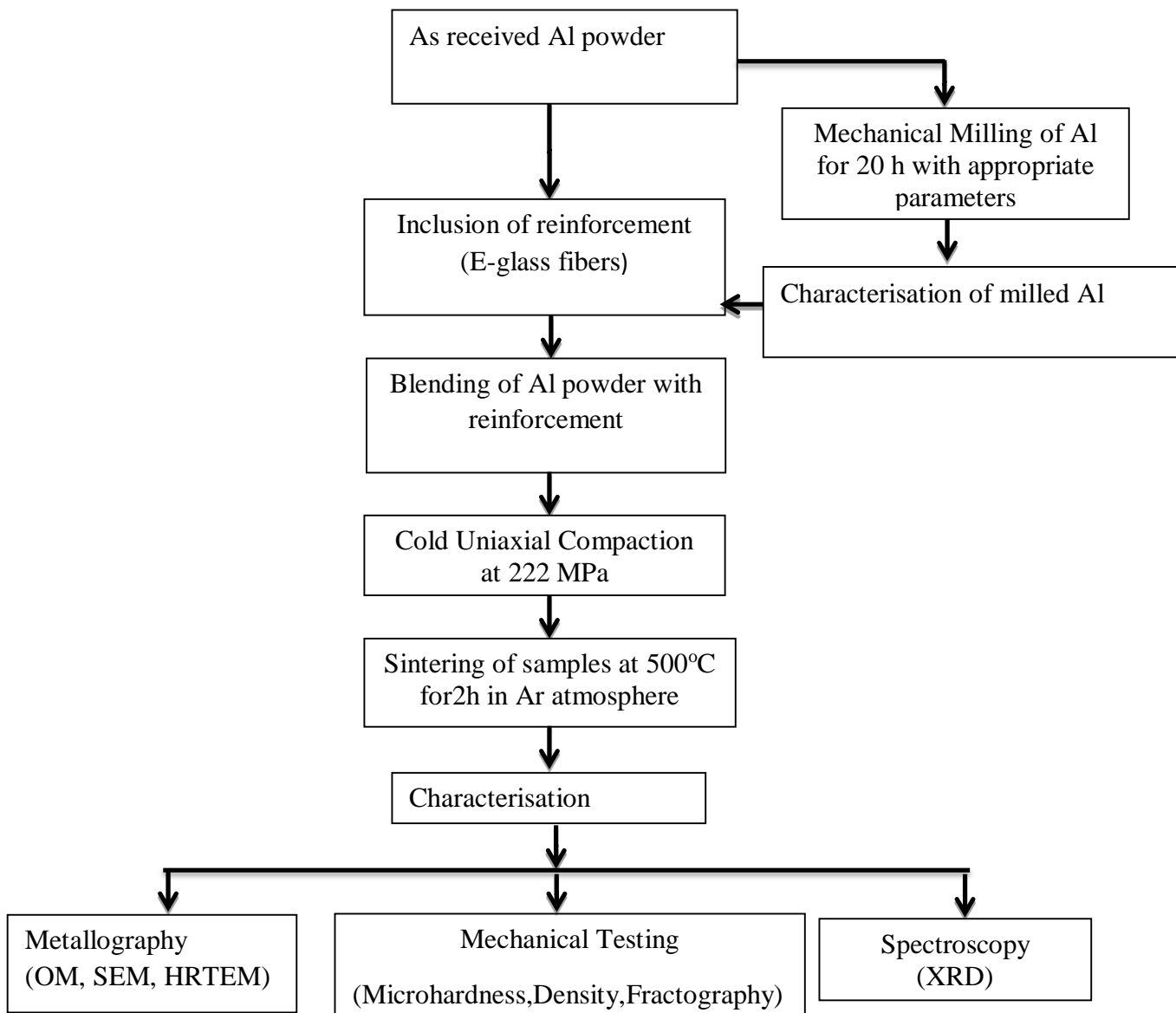


Fig.6 -work plan for the given investigation

### **3.1 Equipment used in the Present Investigation**

#### ***1. Planetary Ball Mill***

In the milling a great energy ball mill is employed for the processing of essential Al to decrease its crystal dimension into nm scale. The as-milled processed nanocrystallite Al is further used for the advancement of Al-based metal matrix mixtures. The powder particles throughout the procedure are once straightened, cold welded, and rewelded. Whenever there is a collision of balls, there's always some quantity of powder particles got tangled in between the balls. At first the particles have robust attraction to weld along as a result of their soft nature causing in formation of larger particle. With continuous changes, the particles become work hardened and get fractured by the mechanism of fatigue failure. Fragments created by this mechanism could still scale back in size due to the absence of robust agglomerating forces.

There are many benefits of Ball milling such as:

- i. Lesser installation price
- ii. Lesser powder price
- iii. Lower grinding medium required.

#### ***Milling Conditions:***

- Ball Mill used: Fritsch P5
  - Milling Medium used: Wet (Toluene)
  - Vials and Balls used: Hardened chrome steel
  - Milling Speed employed: 300 rpm
  - Diameter of Balls taken: 10mm
- 
- Ball to powder weight ratio taken = 10:1

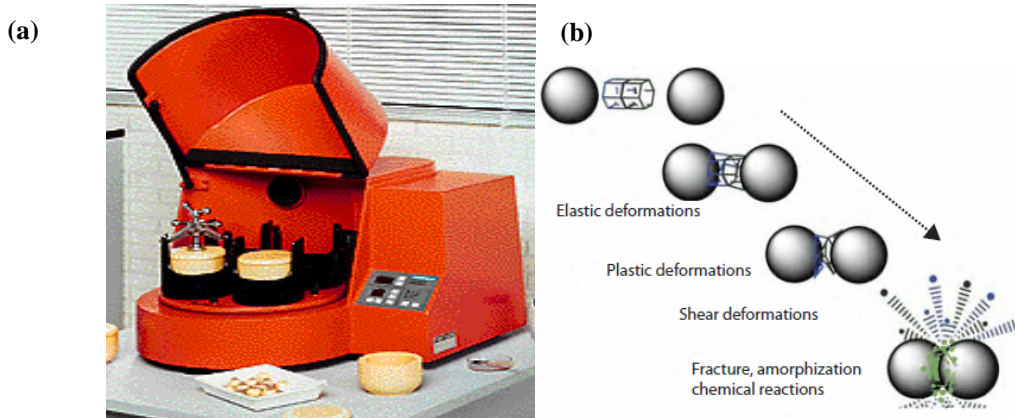


Fig.7- (a) Planetary Ball Mill (b) Schematic view of mechanism of ball milling

The Fig. 3.2(a) The Fritsch planetary ball mill. Fig. 3.2(b) The schematic diagram of the milling process and the behaviour of powder particles during milling.

## 2. Cold Uniaxial Hydraulic Press

It is used for making the green pellets after that pellets will sintered. Uniaxial pressing includes the compaction of powder particles in a die by providing pressure in a uniaxial direction through a punch.

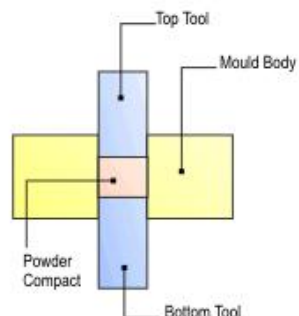


Figure8- Schematic Diagram of Uniaxial Pressing



Figure9-Uniaxial Hydraulic Press



or piston. The presses used are generally hydraulic or mechanical in nature and the pressing cycle repeats itself in a range of 6 to 100 times/min. In the present work the die with 15mm diameter is used and the applied stress is 681MPa for all the samples.

### 3. X Ray Diffraction

A diffractometer is a determining system for examining the structural property of a material from the scrambling example created once a lightweight emission or particles, (for example, X-beams or neutrons) communicate with it. X-beam optical phenomenon of the processed Aluminium powder at the several time interim of process has been completed to review the stages obtained throughout the process to analyse the range of crystallization size, r.m.s. strain and lattice parameter with respect to processing time.



Figure10- X ray Diffraction

A Philip's X'pert Pro high-resolution X-ray diffractometer has been used for the purpose whose maximum scanning range ( $2\theta$ ) for the instrument is 0 to 160°.

### 4. High Temperature Horizontal Tubular Furnace

A great temperature tubular furnace was used for sintering of the green compact. The sintering of the specimens was performed in an inert atmosphere of argon gas. A tube heater is an electrical gadget used to direct blends and sanitizations of inorganic mixes and periodically in natural combination. One possible configuration comprises of a tube shaped

depression encompassed by warming curls which are inserted in a thermally protecting lattice. Temperature could be controlled by means of a thermocouple. The models were warmed in a cauldron in tubular heater in the vicinity of inactive argon gas. The casting temperature of the heater was kept up at 500°C with the holding time of 2 h for all the specimens



Figure11- Tubular furnace

A vacuum controlled atmosphere furnace, manufactured by Naskar& Company with maximum attainable temperature of 1750°C was used for the investigation.

## **5. Microscopic Analysis : Scanning Electron Microscopy(SEM)**

It uses high energy beam of electrons which are directed at the specimen for producing the two dimensional image of a specimen of any dimension. The electrons delivered by a hot fiber were quickened by electric and attractive fields subsequently interfacing with the specimen and delivering signs which contains data about the surface or close periphery geology, structure and different properties, for example, electrical conductivity. In the given examination unadulterated Al and the composites created were examined. In order to deliver pictures, the electron bar is centered into a fine test, which is checked over the surface of the example with the assistance of examining curls. Every point on the specimen that is struck by the high velocity electrons transmits motion as electromagnetic radiation. Gathered results from this radiation, generally optional (SE) and/or backscattered electrons (BSE), are

collected by a finder and the subsequent sign is intensified and showed on a monitor screen. The resulting image is depicts the topographic imaging of objects at low magnifications.



Figure12-Scanning Electron Microscopy

In the present investigation A JEOL -JSM-6480LV has been used. For EDX analysis, the beam energy of 20 kV with a spot size of 100 Å has been used.

## **6. High Resolution Microscopic Analysis : Transmission Electron Microscopy (TEM) and Selected Area Diffraction (SAD)**

In Transmission electron microscopy (TEM), a beam of [electrons](#) is transferred through an ultra-thin specimen, interacting with the specimen as it passes through. It is a microscopic analysis technique. A picture is framed from the cooperation of the electrons transmitted through the example. The particular sample position is amplified and centered onto an imaging gadget, for example, a fluorescent screen or on a layer of photographic film to be recognized by a sensor, for example, a CCD camera. To watch the effect of the mechanical processing of the powder (MWCNTs) , analysis in a transmission electron magnifying instrument (TEM) of the samples was being done which were previously ultrasonicated in acetone. By using a pipette ,the spread powder is then taken out and drops of acetone containing the powder were placed on a carbon coated copper grid for observant in the TEM.



Figure13-Transmission Electron Microscopy

For analyzing the samples, a Philips CM12 TEM has been used and the accelerating voltage of 120 kV was employed. Selected area diffraction (SAD) pattern of all the samples is also taken using the TEM.

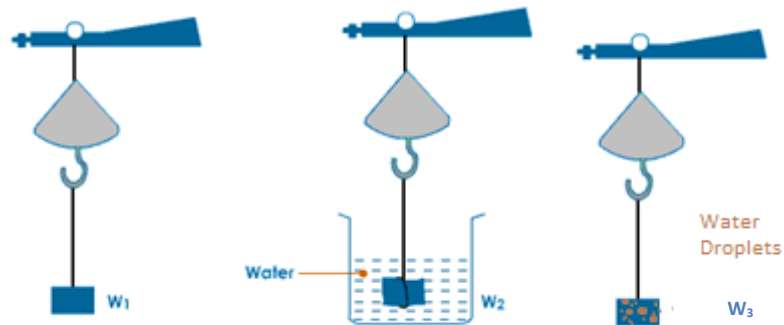
## **7. Measurement of Density**

For the amount of density of samples, Archimedes principle was used. The estimation of the density of shape specimens was done accurately. The number of units per area of variations in density estimation of the shapes considered are usually because of the overlapping pores and some due to the scattered pores present within the samples. Liquid gets into the interrelated pore and impacts the density amount. To rectify errors, three estimations were taken, weight in air, weight of the specimen dispersed in fluid, and weight of the specimen in taken from air and then emerged in fluid for an extended time. The fluid used is refined water ( $\rho = 1.0 \text{ gm/cc}$ )



Figure14- Density Measurement

Three weights were taken for each sample and then density was calculated from the following formula :



- Weight of the sample taken in air  $W_1 = W_{air}$ ,
- Weight of the sample taken in liquid  $W_2 = W_{liquid}$ ,
- Weight of the sample soaked in liquid for a long time  $W_3 = W_{soaked}$ ,

$$\text{Density of the composite} = W_{air} / (W_{soaked} - W_{liquid}) / \rho_{liquid}$$

## 8. Micro Vickers Hardness

In the present work, the load (F) to be considered was taken as 0.98 N and Vickers hardness number is calculated by 1Å diamond indenter, with the shape of a

upstraightbpyramid with square base and an angle  $136^{\circ}$  between opposite faces, which is forced into the sample under a load  $F$ .  $X$  and  $Y$  are the two diagonal of the indentation left on the surface of the sample after elimination of the load square is measured and their first moment  $L$  is calculated using the following equation:

$$H_V = \frac{0.1889F}{L^2} \text{ And } L = \frac{X+Y}{2}$$

where,  $F$  is the applied load (N),  $X$  is the horizontal length (mm),  $L$  is the diagonal of square impression (mm) and  $Y$  is the vertical length (mm).

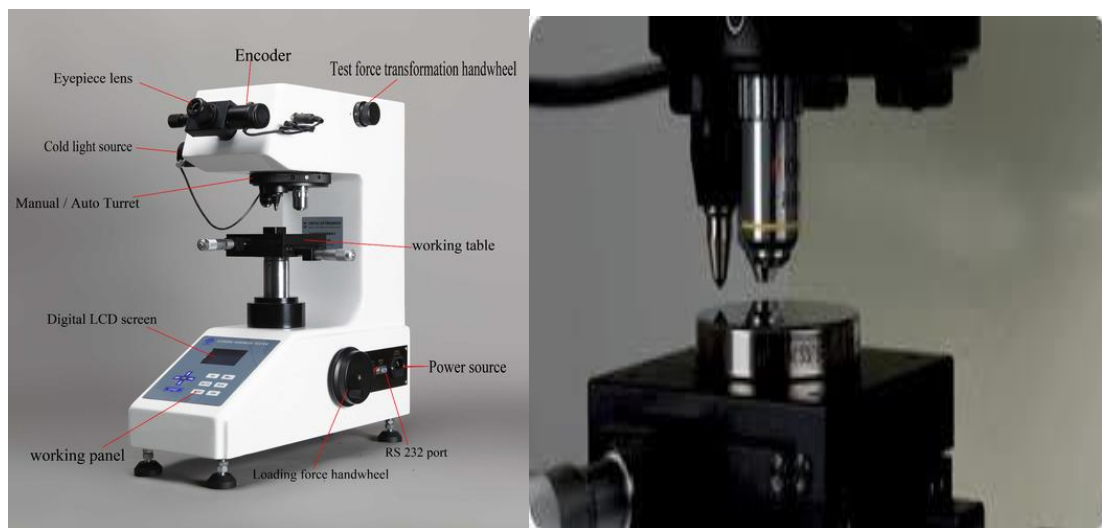


Figure 15- Vickers Hardness Tester

Using a Lecco Vickers Hardness (LV 700) with a diamond indenter, Micro-hardness measurement was performed in the above investigation.

### 3.2. Characterization and Selection Process in Synthesis of Raw Materials

In the present study, we used Al powder and E-glass Fiber as the raw materials. Al powder and E-glass Fiber were taken from the market according to research criteria. The complete measurement and synthesis of the raw materials are given below.

#### Al Powder

Aluminium is the most generally used non-ferrous metal. Lightweight and high modulus aluminium. Metal Matrix Composites have better advantages over traditional aluminium and

organic composite materials. Aluminium metal matrix composites can also be invented using the same techniques and fabrication tooling currently used for aluminium alloys. Aluminium is beneficial for its low density and its capacity to resist corrosion due to the occurrence of passivation. The ductile behaviour of Al makes it an ideal material for toughness imparting substance. Aluminium is a good conductor of heat and electricity the well the conductivity of a material, the well the shielding qualities.

Properties	Metric
Density	2.7 g/cc
Hardness, Vickers	107
Ultimate Tensile Strength	310 MPa
Tensile Yield Strength	276 MPa
Elongation at Break	12 %
Modulus of Elasticity	68.9 GPa
Poisson's Ratio	0.33
Fatigue Strength	96.5 MPa
Fracture Toughness	29 MPa.m <sup>1/2</sup>
Shear Modulus	26 GPa
Shear Strength	207 MPa
Electrical Resistivity	3.99 X10 <sup>-6</sup> Ω.cm
CTE at Room Temperature	23.6 μm/m.°C
Specific Heat Capacity	0.896 J/g°C
Thermal Conductivity	167 W/m.K
Melting Point	582- 652°C

Table-3.1

- **E-Glass Fiber**

Glass fibers are among the simplest industrial materials known these days. They are primarily made of raw materials, that square measure open in nearly on infinite offer. they have useful properties like hardness, transparency, dependableness, and idleness, and attractive fiber properties, for instance, quality, flexibility, and stiffness.

Property	Minimum Value (S.I.)	Maximum Value (S.I.)	Units (S.I.)	Minimum Value (Imp.)	Maximum Value (Imp.)	Units (Imp.)
Bulk Modulus	43	50	GPa	6.23662	7.25188	10 <sup>6</sup> psi
Compressive	4000	5000	MPa	580.151	725.189	ksi

<b>Strength</b>						
<b>Hardness</b>	<b>3000</b>	<b>6000</b>	<b>MPa</b>	<b>435.113</b>	<b>870.227</b>	<b>ksi</b>
<b>Tensile Strength</b>	<b>1950</b>	<b>2050</b>	<b>MPa</b>	<b>282.824</b>	<b>297.327</b>	<b>ksi</b>
<b>Young's Modulus</b>	<b>72</b>	<b>85</b>	<b>GPa</b>	<b>10.4427</b>	<b>12.3282</b>	<b>106 psi</b>

Table-3.2



# **CHAPTER 4**

## **RESULTS AND DISCUSSION**

## Mechanical alloy of Al

Elemental Al powder was milled in a high-energy planetary ball mill in order to produce nanocrystalline Al. Mechanical milling is a suitable and favourable process to create nanostructured powders. It is a processing method used for the planning of nanocrystalline metallic and ceramic powders. It is a solid-state powder processing method which involves frequent welding, fracturing and rewinding of powder atoms in a high-energy ball mill. Heavy deformation of particles takes place and growth in strain can be depicted by the presence of peak broadening.

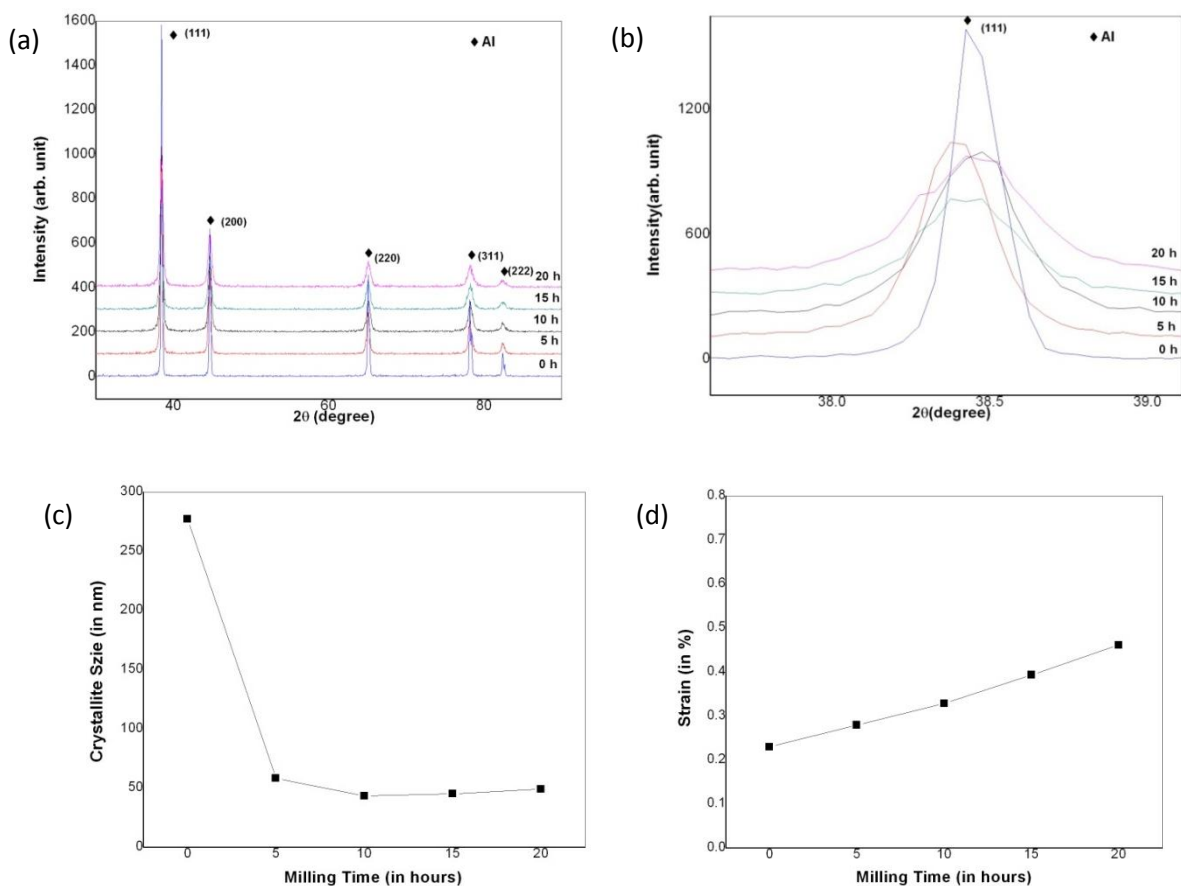


Fig16 (a, b) X-ray diffraction pattern of Al milled for various periods of time. Variation of (c) crystallite size of Al and (d) strain in Al lattice with milling time

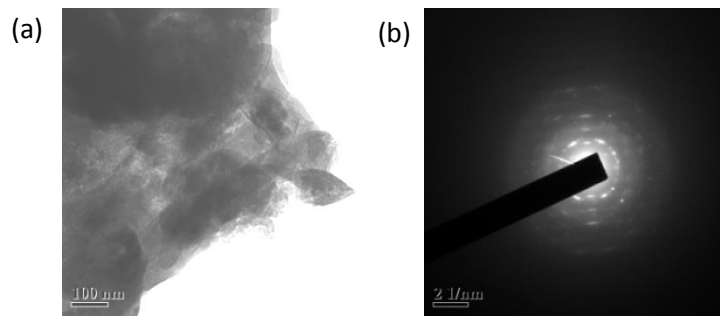


Fig. 17 (a) HRTEM image and (b) SAD pattern 20 h milled Al

Fig.17(a) shows the HRTEM image of 20 h milled powder. The HRTEM image clearly shows that the powder has been reduced to nanometric dimension after milling for 20 h . The selected area diffraction (SAD) pattern in Fig. 17(b) indicates sharp ring pattern which confirms the nanocrystalline nature of the 20 h milled Al powder.

### E-Glass Fiber

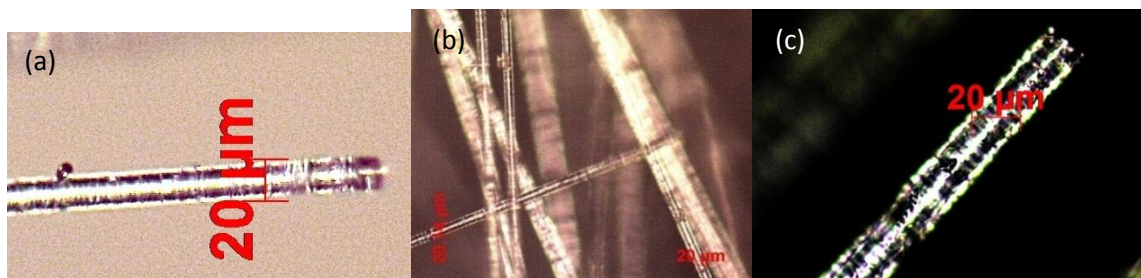


Fig. 18 (a-c) optical images of E-glass fiber used in composite

### Al-E glass fiber composites

E-Glass fibers are mainly used as reinforcement in the composites. E-glass has a hardness of 6000 MPa and its tensile strength is 3500 MPa. Its Young's modulus is 85 GPa. Its compressive strength is 5000 MPa. It has a density of 2.58 gm/cc. It is resistant to chemical attack and has high dimensional stability. Here in our study we have used three different fiber lengths of 2, 5 and 8 mm. Using each particular fiber length we have developed Al-1, 2 and 5 vol. % E-glass fiber composite. The aspect ratio for the various fiber lengths have been

calculated using the diameter of the fiber as 20  $\mu\text{m}$ . The table below shows the aspect ratio of the various fibers that have been used for developing the various Al-E-glass fiber composites.

<b>Fiber Length</b>	<b>Aspect Ratio (l/d)</b>
2 mm	100
5 mm	250
8 mm	400

Table- 4.1

The optical micrographs in Fig. 19-21 show the microstructure of sintered Al-E-glass fibers composites containing different vol. % (1, 2 and 5%) of E-glass fiber having different fiber lengths (2, 5 and 8 mm). Sintering was done at 500°C for 2 h in Ar atmosphere. From the analysis we had seen that the composites have a random distribution of the E-glass fibre in the Al-matrix and it indicates very good bonding of the E-glass fibre and the Al-matrix. The fibre has good wettability on the Al matrix.

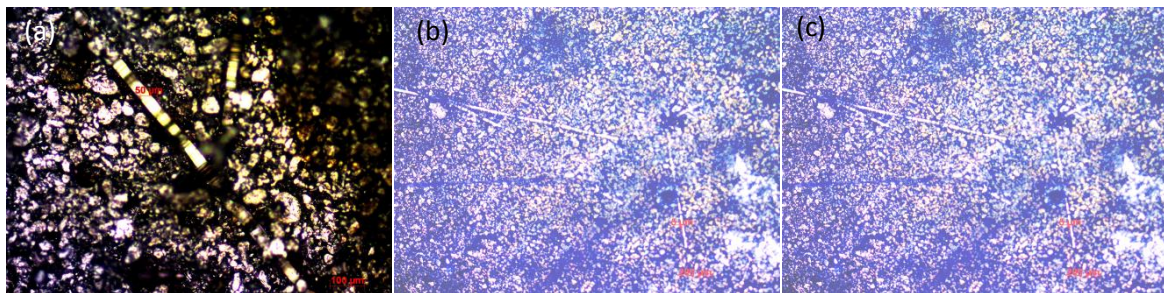


Fig.19 (a-c) shows the optical image of unmilled Al-E-glass fibers composites for different vol. % (1, 2 and 5%) having 2 mm length of E-glass fibre used as a reinforcement

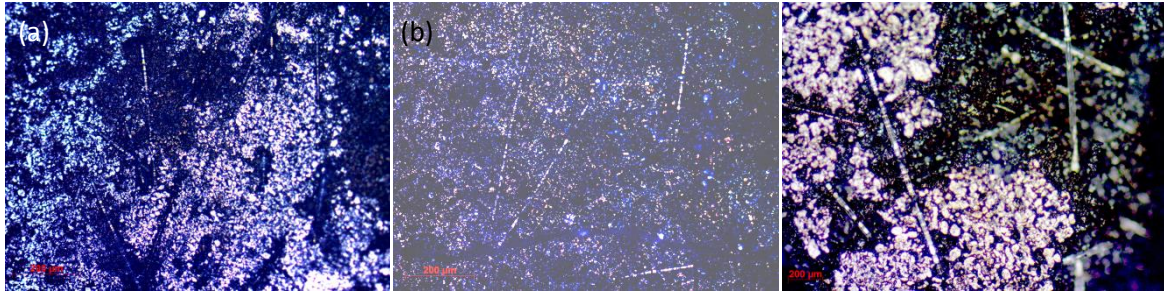


Fig.20 (a-c) shows the optical image of unground Al-E glass fibers composites for different vol. % (1, 2 and 5%) having 5 mm length of E-glass fibre used as a reinforcement.

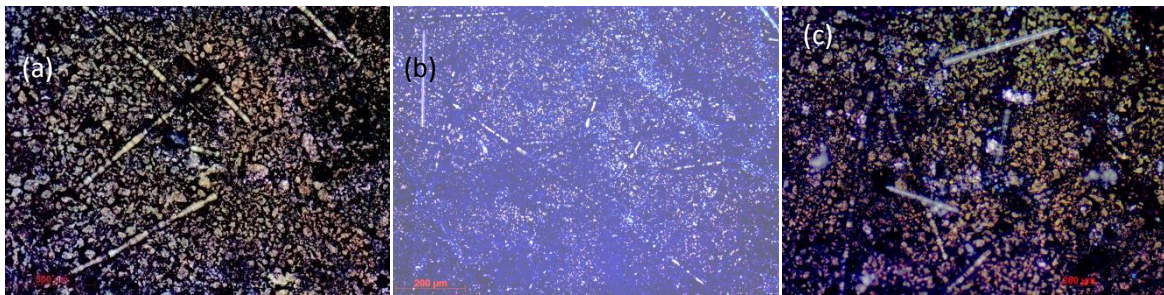


Fig.21 (a-c) shows the optical image of unground Al-E glass fibers composites for different vol. % (1, 2 and 5%) having 8 mm length of E-glass fibre used as a reinforcement.

The SEM images in Fig. 22-24 show the microstructure of sintered Al-E-glass fibre composites containing different vol. % (1, 2 and 5%) of E-glass fiber having different fiber lengths (2, 5 and 8 mm). It is evident from the SEM images that for a particular fibre length there is an increase in the volume fraction of pores as the volume fraction of the fiber increases. Higher volume fraction of the fiber leads to poor densification during sintering. As the sintering was done at 500°C solid state sintering takes place.

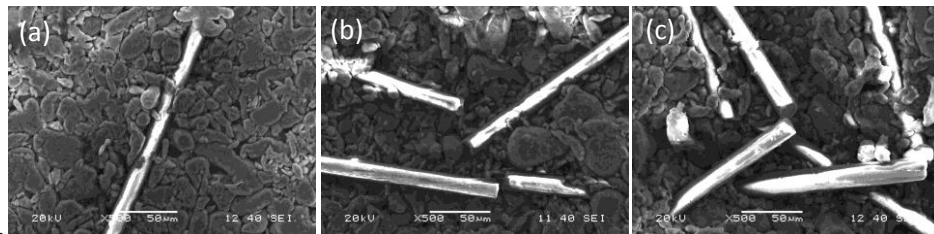


Fig.22 (a-c) SEM images of unground Al-1, 2 and 5 vol. % E-glass fiber composites having fiber length of 2 mm as reinforcement

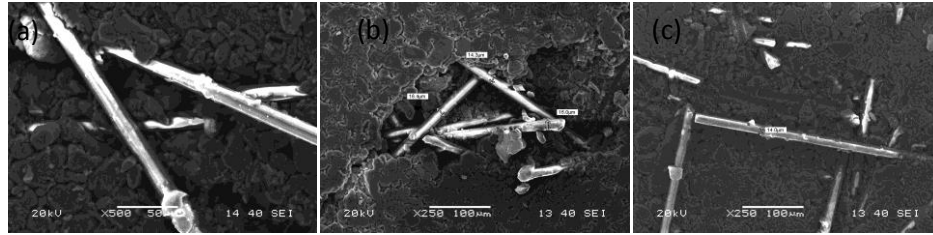


Fig.23(a-c)-SEM images of unground Al-1, 2, and 5 vol. % E-glass fiber composites having fiber length of 5 mm as reinforcement

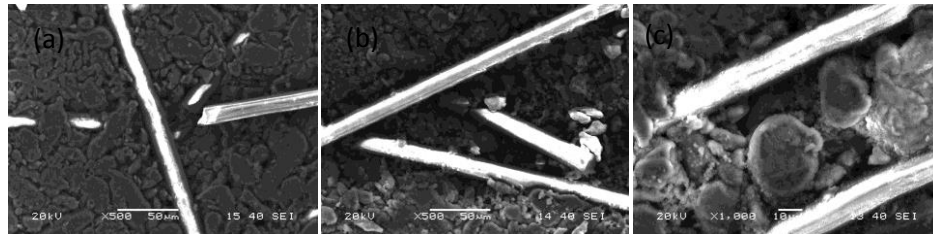


Fig.24(a-c) SEM images of unground Al-1, 2, and 5 vol. % E-glass fiber composites having fiber length of 8 mm as reinforcement

Fig 25 (a) shows the deviation of relative density of unground Al-E glass composite holding different vol. % of E-glass fibre reinforcement. The E-glass fiber also had varying lengths of 2, 5 and 8 mm for each vol. fraction of E-glass fiber. Density has been calculated by using Archimedes' method. It is evident from the plot in Fig. 25 (a) that low vol. % of E-glass fiber in the composite gives better densification. However better densification of the Al-E-glass fiber composites is seen when 2 mm and 8 mm length of E-glass fiber is used. This suggests that either very small aspect ratio (100) or very high aspect ratio (400) is capable of giving good bonding between the Al-matrix and the E-glass fiber. It is known that the critical length ( $L_c$ ) is a measure of the minimum perfectly aligned fiber dimension required for maximum stress transfer within the cured resin. Therefore by maximizing fiber-aspect ratio, one can reduce the debonding effects of high-modulus fiber end-shear through the matrix in order to increase the strength of the composite. The major factors that influence oriented  $L_c$  include the interfacial shear bond strength ( $\tau$ ) between the fiber and resin, the fiber tensile strength ( $\sigma_f$ ), and the fiber diameter ( $d$ ). E-glass is a customary high-tensile-strength silica-based fiber having a common pristine  $\sigma_f$  at 3.4 GPa

$$\tau = \sigma_f d / 2L_c \text{ or } L_c = \sigma_f d / 2\tau$$

$$\text{or } L_c / d = \sigma_f / 2\tau$$

As the  $\tau$  value for the interfacial shear bond strength between the E-glass fiber and the Al-matrix is not known therefore it is not possible to predict the exact value of the critical length ( $L_c$ ) or the critical aspect ratio [31-32].

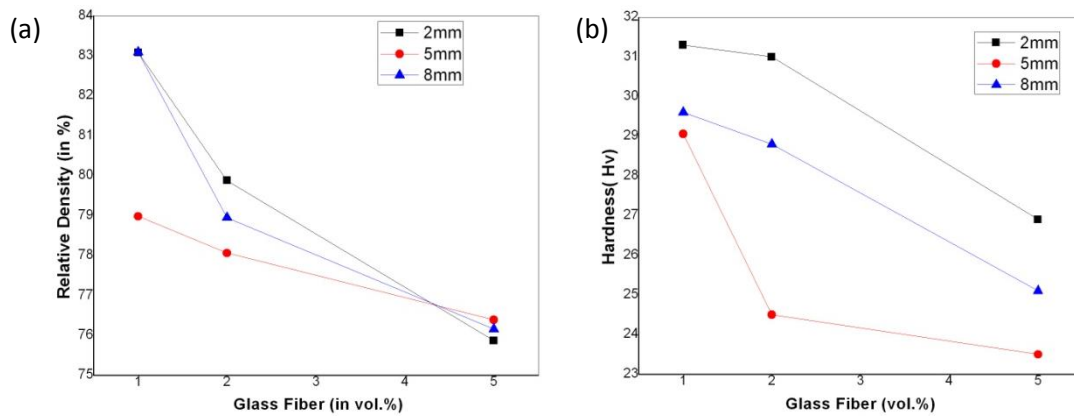


Fig.25 (a) Relative Density and (b) Vickers's hardness plot of various sintered unmilled Al-E-glass fiber composites at different vol. % having different length of E-glass fibers as reinforcement

Fig. 25(a) shows that the relative density decreases with the increase in the vol. % of E-glass fiber in the unmilled Al-E-glass fiber composite. For a particular fiber length the highest relative density was obtained for the lowest vol. % of the E-glass fiber in the Al-E-glass fiber composite. The maximum relative density achieved was ~83% in the case of Al-1 vol. % E-glass fiber having fiber length of 2 mm length and 8 mm. This suggests that the critical fiber length ( $L_c$ ) lies above 5 mm as 8 mm fiber length is able to give very good bonding between the fiber and the matrix. Very low fiber length of 2 mm also gives similar level of densification. Therefore aspect ratio of above 250 is capable of giving good densification. Fig. 25(b) shows the variation of Vickers hardness for unmilled Al-E-glass fiber composites containing different vol. % of E-glass fibre. Different fiber lengths were considered for each

vol. % of glass fiber. The hardness of the Al-E-glass fibre composite decreased with the increase in the vol. % of E-glass fiber in the composite for a particular length of the fiber. The hardness was found to be maximum for the 2 mm length (308.9 MPa) of E-glass fibre. 5 mm length of glass fiber resulted in least hardness of the composite. Whereas 8 mm fiber length gave better hardness compared to the 5 mm fiber length for any particular vol. % of E-glass fiber in the composite. This again suggests that critical length of the fiber lies above 5 mm as a result the 8 mm fiber length is capable of giving higher hardness values.

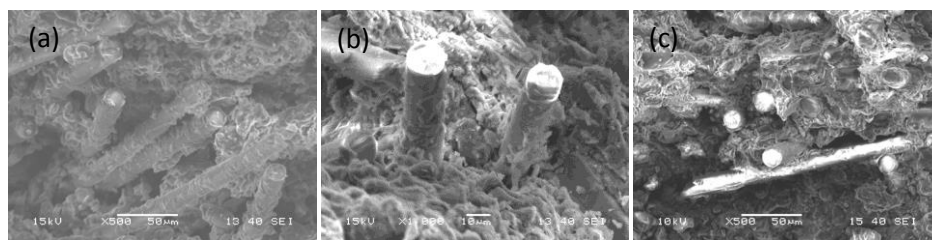


Fig.26 (a-c) SEM image of fractured sample of unmilled Al-E-glass fiber with different vol.%(1, 2 and 5%) having 8 mm length of E-glass fibre composite

The fractured samples were analyzed by SEM. The SEM images in Fig. 26(a-c) show the fractographs of various unmilled Al-E glass fibre composites where the length of the glass fiber is 8 mm. It shows ductile fracture in the Al matrix. Dimples could be seen on the fractured surface of the composites. The glass fibres were found to be embedded in the Al-matrix. Fibre pull-out is also seen in the composite. Higher stress on each fibre leads to pull out of the fibre. As the sintering was done at 500°C solid state sintering takes place. As a result the bond strength between the Al-matrix and the fiber is low. This leads to the lower load bearing capacity of the glass fibre. The stress develops on the fibre leads to plastic deformation of Al-matrix around the fibre. The SEM images show a random distribution of fibers inside the Al matrix.



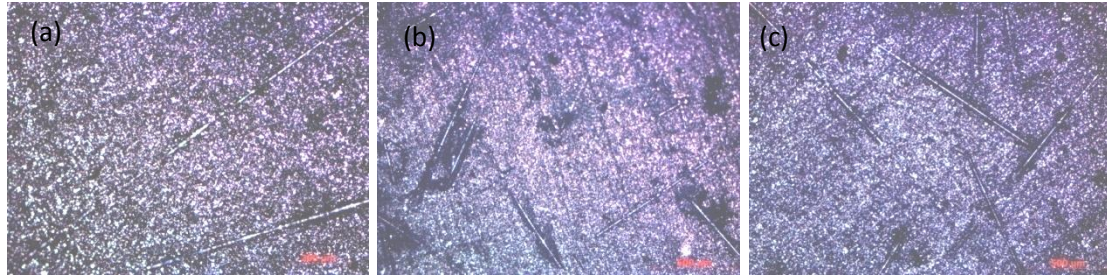


Fig.27 (a-c) Optical images of milled Al –E glass fibers with different vol. % (1,2and 5%) having 8 mm length of E-glass fibers.

Figs. 27(a-c) show the optical images of milled Al-E-glass fiber composites for different vol. % of E-glass fibre. Here a fiber length of 8 mm was taken. From the images it is clearly seen that the E-glass fibre are randomly distributed all over the Al-matrix composite. Good interfacial bonding can be observed between Al and E-glass fibers.

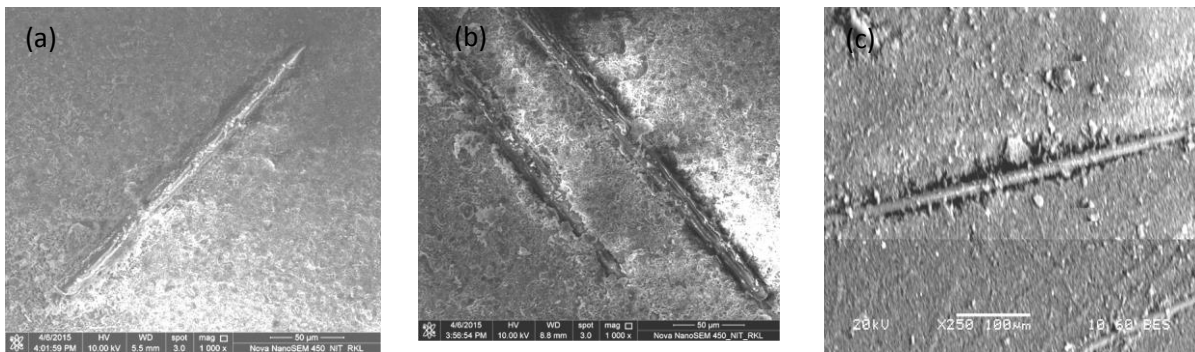


Fig.28 (a-c) SEM images of milled Al –E glass fibers with different vol. % (1, 2 and 5%) having 8 mm length of E-glass fibers.

Figs.28 (a-c) show the SEM images of milled Al-1, 2 and 5 vol. % E glass fiber composites. The length of the fiber was 8 mm. The E-glass fiber composite samples were sintered at 500°C for 2 h. It is clearly seen that there is random distribution of fibres throughout the Al-matrix and fiber are embedded in the Al-matrix.

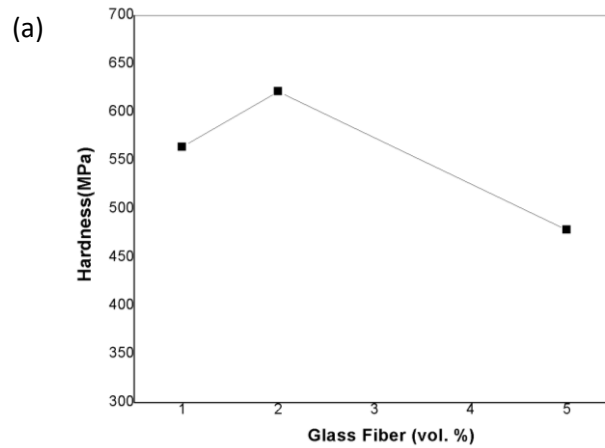


Fig.29 (a)Vickers's hardness plot of various sintered milled Al-E-glass fiber composites at different vol. % having fiber length of 8 mm as reinforcement

The hardness of the composites shown in Fig.29 indicates a much higher hardness for the milled Al-E glass fiber composite developed using 8 mm glass fiber length as compared to the unmilled Al-E glass fiber composite using the same length of glass fiber. Milled Al-E-glass fibre Al is nanocrystalline and as a result has good sinterability leading to better densification and as a result shows better hardness. Al has a melting temperature of 660°C. It is known that the nanocrystalline nature of Al could reduce the melting point of Al. As a result better sinterability and densification could be achieved at the sintering temperature of 500°C.

**CHAPTER 5**  
**CONCLUSION**

This thesis reports the results of a systematic study of development and characterization of Al-based metal matrix composites using E-glass fiber as reinforcements. The conclusions drawn from the present investigation are as follows:

- 1- Milling of elemental Al powder for 20 h led to the formation of nanostructured Al. The crystallite size of Al after 20 h of milling was found to be around 50 nm. This was confirmed both by x-ray diffraction analysis and high resolution transmission electron microscopy. The crystallite size of Al milled for 20 h was found to be below 50 nm and the lattice strain was found to be around 0.45 %.
- 2- X-ray diffraction analysis reveals that there was no trace of contamination from the milling media.
- 3- As-milled Al-E-glass fiber composites shows better densification and sinterability compared to the unmilled Al-E-glass fiber composites due to the finer size of Al particles in the milled Al powder. Better densification and sinterability has led to higher hardness. The maximum hardness of 630 MPa was found in the case of as-milled Al-2 vol. % E-glass fiber composite.
- 4- It is found that aspect ratio of 100 and 400 corresponding to fiber length of 2 mm and 8 mm respectively leads to better relative density and hardness values, whereas the aspect ratio of 250 corresponding to the fiber length of 5 mm resulted in least relative density and hardness values. Therefore the critical length of the fiber ( $L_c$ ) is expected to lie above 250 corresponding to fiber length of 5 mm.
- 5- The hardness of the Al-E-glass fibre composites decreased with the increase in vol. % of E-glass fiber in the composite for a particular fiber length. Higher length of fiber

showed poor densification and hardness both in the case of milled and unmilled Al-E-glass fibre composites.

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