

Development of Aluminium Composite and its Foams for Mechanical Applications

A

Thesis

Submitted for the award of degree of

Doctor of Philosophy

By

Suresh Kumar

(Regd. No. 901112009)

Under the supervision of

Dr. O.P. Pandey

(Senior Professor & DoRSP)



SCHOOL OF PHYSICS & MATERIALS SCIENCE

THAPAR UNIVERSITY, PATIALA – 147004

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CERTIFICATE

This is to certify that the thesis entitled “**Development of Aluminium Composite and its Foams for Mechanical Applications**” which is being submitted by *Suresh Kumar* in fulfillment of the requirements for the award of the degree of *Doctor of Philosophy* in the School of Physics and Materials Science, Thapar University, Patiala (Punjab), India is an exclusive record of candidate’s own research work under my supervision. The thesis in part or in full has not been submitted in any other university or institute for the award of any degree. The thesis is fit to be considered for the award of degree of Doctor of Philosophy.



Dr. O.P. Pandey

Senior Professor & Dean (Research and Sponsored Projects)

School of Physics and Materials Science

Thapar University

Patiala- 147004 (INDIA)

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Dedicate to my uncle
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LIST OF PUBLICATIONS

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- **Suresh Kumar**, Ranvir Singh Panwar, Vipin Sharma, O.P. Pandey; Tribological characteristics of Aluminium dual-reinforced particles (Al-DRP) composites developed by stir casting route” AFTMME'12(5-7 Oct., 2012) at PTU.
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PREFACE

The present work contains the details and discussion on the development and characterization of zircon sand reinforced LM13 aluminum matrix composites (AMCs) and LM13 aluminium matrix foam (AMFs) and its composite (AMCFs). The entire work in this thesis is presented in **seven chapters**.

In the **chapter 1**, general introduction to particle reinforced aluminium matrix composite is given. A summary of particle reinforced aluminum matrix composite, aluminum foam and aluminum composite foam along with their production methods are also presented in this chapter. The main factors responsible for the mechanical properties of the AMCs, AMFs and AMCFs along with their properties and applications in different sectors are also discussed in brief.

Chapter 2 covers the literature review regarding the work done on the development of AMCs, AMFs and AMCFs by many researchers using different fabrication techniques and their study based on wear behavior. As our work is based on the stir casting process, hence we focus on the works done by the researchers in the related area for development of AMC, AMF and AMCF and their physical properties, hardness, wear resistance and blast mitigation. The last section of this chapter presents the aims and plan of the work of the thesis.

Chapter 3 contains the experimental procedure followed in the present work. Detailed procedure to develop the AMCs, AMFs and AMCFs and their characterizations are given in this chapter. Different characterization techniques such as X- ray diffraction (XRD), Optical microscopy, Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), Vickers hardness testing and Wear testing with pin-on-disc machine and blast mitigation test along with different operating parameters are also discussed. A flow chart of the methodology used to develop for AMCs and AMF/AMCFs are also given in this chapter.

Chapter 4 deals with microstructural analysis and discussions of developed AMCs, AMFs and AMCFs. The structural analysis of the developed AMCs, AMFs and AMCFs with reinforcement of different size zircon sand particles in the alloy matrix at different conditions has been done critically. Al-Si alloy (LM13) was used as the matrix material for the composites. Composites with reinforcement 2.5- 15wt.% (average particle size range fine (1- 32 μm) and coarse size (106- 125 μm) were prepared. Optical microscopy and SEM were used to examine the distribution of reinforced particles in the matrix of AMCs and AMCFs. In this chapter, results of microhardness and wear tests obtained from cast AMCs, AMFs and AMCFs is discussed. Reinforcement of fine (1-32 μm) and coarse (106-125 μm) size ZrSiO_4 particles from 2.5- 15 wt.% have been used to develop AMCs and AMCFs. The particle bonding with matrix was tested by measuring hardness at the interface. Wear behavior with variation in applied load of fine and coarse size ZrSiO_4 particles reinforced composites is described in this chapter. The results show that wear characteristics of the $\text{AMC-}^{15}\text{C}_{\text{fine}}$, $\text{AMF-}^2\text{F}_{900}$ and $\text{AMCF-}^{15}\text{C}_{\text{fine}}$ is better than other developed AMCs, AMFs and AMCFs. The operative wear mechanism has been established by analyzing the wear track and wear debris under scanning electron microscope. The overall results of all the developed samples have also been discussed at the end of the chapter.

Chapter 5 gives the details of the blast mitigation test of AMFs and AMCFs. The effective range of blast pressure depends on the density and cell geometry of the AMFs and AMCFs. Blast mitigation test were performed on AMFs and AMCFs samples prepared by using different wt. % of blowing agent (for AMFs) and ZrSiO_4 reinforcement (for AMCFs). Blast mitigation trials were conducted using CE pellet having 35g of tetryl charge. The results of the blast tests and their failure study of deformed materials done on developed foams in Terminal Ballistics Research Laboratory, Defence Research and Development Organization, Ramgarh, Chandigarh, India have been compiled and discussed.

Chapter 6 describes the conclusion of the entire work done in the present investigation along with the future scope. The present work shows a good distribution of the zircon sand particles in LM13 alloy matrix developed by stir casting process. XRD data shows the presence of zircon sand particles as well as new phase developed during the casting process. Uniform distribution and bonding of zircon sand particles with the matrix was observed. It was found that hardness of the base alloy was improved with increasing the amount of zircon sand particles in the matrix. However, with decreasing the particle size hardness of the composites was increased significantly. Wear tests of all the developed composites were done at different loading conditions. It was observed that wear resistance of the base alloy improved with increasing the amount as well as decreasing the size of the reinforced particles. However, wear rate of the composites increases with increasing the applied load.

In case of AMFs and AMCFs factors which are affected by the foaming temperature on alloy foam and composite foams were investigated. It was found that the foaming temperature for alloy foam and composite foams influence the cell geometry which ultimately generates different structures. Addition of zircon sand particles influences the foaming temperature and viscosity for the internal diffusion pressure of the melt material. The optimized condition of foaming temperature for composite foam containing 5wt.% zircon sand and alloy foams are 875 °C and 850 °C respectively when 2wt.% CaCO_3 as foaming agent having narrow particle size (1-32 μm) has been used. Cell size of the sample foamed increases with increasing particle size of blowing agent. By properly controlling the foaming temperature and zircon sand content of the melt, which is different for alloy and composites, it is possible to optimize the condition to get uniform size foams having high porosity. The increase in foaming temperature and gas flow rate enlarge the cell size and decrease the cell wall thickness due to the difference pressure between cell wall and node. AMCF- $^{15}\text{C}_{\text{fine}}$ shows better wear resistance in comparison to all AMFs and AMCFs at different applied loads.

CHAPTER 1

INTRODUCTION

Overview

Composites and foam materials exist in nature and are also man-made. A brief history of composites and foam materials and their classification on the basis of their constituents is given in this chapter. Closed cell foam is a new class of material, unfamiliar to most engineers. They are made by a range of novel processing techniques, many still under development, which are documented in this chapter. The main factors affecting the mechanical properties of the composite and composite foams along with brief introduction of different types of foams and their applications is also given in the end of the chapter.

1.1 A Brief History of Composite Materials

In ancient times

Throughout history, humans have used different types composite materials. One of the earliest use of composite material was by the ancient Mesopotamians around 3400 B.C. They glued wood strips at different angles to make plywood. Archaeologists have found natural composite building materials that were in use in Egypt and Mesopotamia. The ancient builders and artisans used chaff/straw to reinforce mud bricks, pottery, and boats around 1500 B.C. The Mongols invented the first composite bows made from different type combination of wood, cattle tendons, bamboo, horns, bone and silk bonded with natural pine resin. The bows were small, very powerful, and extremely accurate. There are other numerous examples of both natural and man-made composite materials.

The 19th century was perhaps the most important decade for the composites industry. In 1947, automobile body fully fabricated by the composite was tested. There are numerous potential applications of composite materials over monolithic materials in different fields of engineering viz., construction, automotive, defence and aerospace sectors [1-3]. Appropriate combination of metals, non-metals and ceramic materials in different proportions provides the opportunity to fabricate composites for a wide range of applications. The physical and chemical properties of the constituent materials play vital role in the specified application. The advanced engineering materials have a wide range of applications in the field of energy conservation, national security, mechanical applications and recreational activities.

Earlier, composites were commonly used in advanced mechanical applications but nowadays, the role of composites in society has increased to a greater extent. Automobiles, ships, aircrafts etc. all preferably used components made of composites. The weight considerations of the composites make them a good substitute over the conventional materials for a large variety of mechanical applications [4]. Further, to meet the demand of low cost, light weight shielding materials have opened an area of research of foam materials also.

1.2 Composite

The material comprising of two or more components with distinguished physical or chemical properties and distinct boundaries between the components are referred as composite material. However, the individual components should remain separate and distinct within the finished

structure. Most of the composites have two constituent materials: a matrix (binder) and reinforcement. The continuous phase in the composite (or two phase alloy microstructure) in which a second phase is dispersed is known as matrix. Selection of the matrix for the composite depends on the desired properties for different engineering applications. For the structural applications aluminium, titanium and magnesium are used as a matrix to develop light weight composite materials. The discontinuous phase present in the composite is known as reinforcement. In general, the reinforcement is much stronger and stiffer than the matrix which provides better mechanical and chemical properties to composite.

1.3 Classifications of Composite Materials

1.3.1 On the Basis of Matrix

On the basis of matrix, composites are classified as metal matrix, polymer matrix, and ceramic matrix. Figure 1.1 shows the allocation of the composite materials into groups of various types of materials presenting their main basic properties.

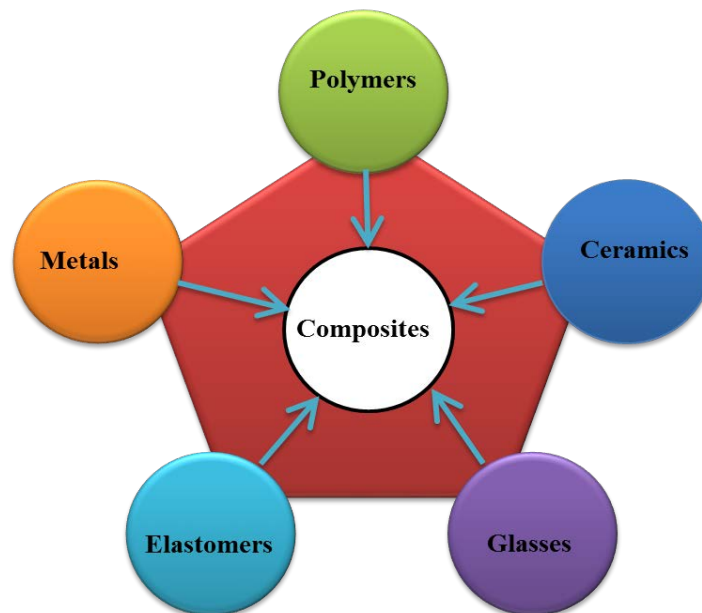


Figure 1.1: Classification of composite within the group of materials.

1.3.2 On the Basis of Reinforcement

On the basis of reinforcement composites can be classified as fibers, fabrics, particles, and whiskers [5]. Reinforcement facilitates the tailoring of the matrix properties specifically to meet the required application. However, in addition to structural properties reinforcement also improves

strength, thermal and electrical conductivity and wear resistance. Figure 1.2 shows the classification of the composites on the basis of the reinforcements.

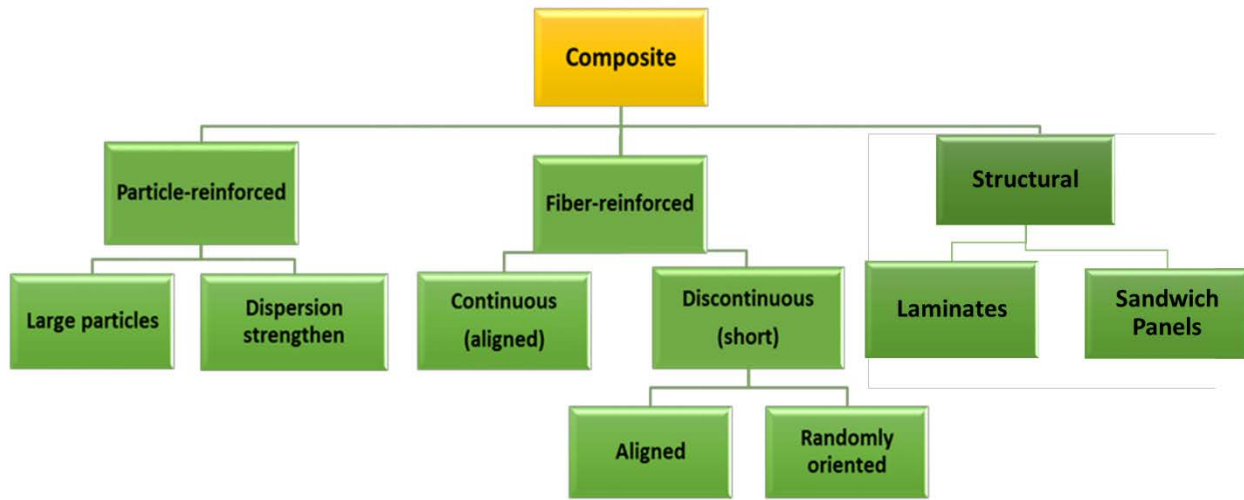


Figure 1.2: Classification of the composite on the basis of different reinforcement [5].

1.4 Production and Processing of AMCs

Aluminium matrix composites (AMCs) can be developed by varieties of methods. The selection of suitable process depends upon the matrix, shape and size of reinforcement, type of reinforcement (particles/fibers) and its application. By altering the processing parameters, it is possible to obtain different characteristic profiles, although the same composition and amounts of the components are involved. At industry level, AMCs are mostly fabricated by using metallurgical processes (e.g. Stir casting, Infiltration, Spray casting, In-situ processing etc.) or by powder metallurgical processes. At present melting metallurgy process route offers a greater importance such as large scale production, low cost and ease to fabrication of complex structures over the powder metallurgy process. Some widely used melting metallurgical processes for the development of composites are briefly discussed below:

1.4.1 Stir Casting Process

Stir casting process involves incorporation of ceramic particulate into liquid aluminium melt and allowing the mixture to solidify. There should be good wettability between the particulate reinforcement and the liquid aluminium alloy melt [6]. The most commercially used technique for the fabrication of composites is known as vortex technique or stir-casting technique. The vortex

technique involves the mixing of pre-treated ceramic particles into the vortex of molten alloy created by the rotating impeller.

1.4.2 Infiltration Process

Liquid aluminium alloy is injected/infiltrated into the interstices of the porous preforms of continuous whisker/particle fibre/short fibre to produce AMCs [7]. The infiltration can take place with or without the application of pressure or vacuum which depends on the nature of reinforcement and its volume fraction.

1.4.3 Spray Deposition

Spray deposition techniques fall into two distinct classes, depending upon whether a droplet/stream is produced from a molten bath (Osprey process) or by continuous feeding of cold metal into a zone of rapid heat injection (thermal spray process). The spray process has been extensively popular for the production of AMCs by injecting whisker/ceramic particle/short fibre/fibre into the spray [8].

1.4.4 In-Situ Processing

There are several different processes that would fall under this category including liquid-gas, liquid-solid, liquid-liquid and mixed salt reactions [9]. In these processes refractory reinforcement is created in the aluminium alloy matrix.

1.5 Application of Aluminium Matrix Composites (AMCs)

The mechanical behaviour of AMCs has been investigated thoroughly in various research works [1–8]. The increased mechanical properties, with respect to unreinforced aluminium, constitute in fact the main driving force for the applications of these materials in different engineering fields. Discontinuously reinforced aluminium composites have competitive price levels, which makes them very attractive for different applications. A key-point for the commercial success of these materials is related to their good mechanical behaviour at high temperatures. The toughness and the impact strength as functions of the different processing parameters have been deeply analyzed and reviewed by several researchers [10-13].

1.5.1 Limitation of AMCs and Development of Aluminium Metal Foam

Apart from the tailored properties of AMCs, now a day's requirement of higher impact energy, higher absorption capacity along with lower thermal and electrical conductive properties in different engineering applications limits the intake of AMCs.

In recent times, the demand of lightweight material has rapidly increased due to the advanced development of automobile, defence area and transportation industries. As a rule, the growing demands for active and passive safety of vehicles, particularly in the automotive industry, lead to an increase of vehicle weight, which is in contradiction to further demands viz. lower fuel consumption. For this reason, these exceptionally lightweight materials possess unique combinations of properties. The increasing demands of lightweight materials with low coefficient of thermal expansion, high impact energy absorbers and better wear resistance have led to the development of metal foam [14-17].

1.6 Introduction: Foams

Foam is the uniform dispersion of one phase (gas) in a second phase (solid/ liquid) [15]. Metal foams are very interesting materials as their manufacturing involves the simultaneous occurrence of solid, liquid and gaseous phases. The morphology of the solidified foam is quite complex. Cellular metals or metallic foams are a class of material in which pores are intentionally integrated in their structure. The terminology cellular metals or metallic foams is referred to the material having large volume of porosities [15]. The porous metals are fabricated using foaming technique, they are generally termed as foamed metal or metallic foams. The highly porous materials are called as metal sponge. Metallic foam has a complex interconnected porosity which cannot be segmented into definite cells [17]. The combination of properties viz. mechanical strength, stiffness and energy absorption achieved by porous metal and metallic foams are superior to dense polymers/ metals and ceramics/polymer and ceramic foams. As compared to polymer foam, metallic foams have better thermal and electrical conductivity and can sustain their mechanical properties at elevated temperatures. They are usually stable in harsh environments as compared to polymer foams. Metallic foams have the ability to deform plastically and absorb energy [15]. If these have open porosity, they are permeable and can have very high specific surface areas, characteristics required for flow-through applications or when surface exchange are involved. Solid foams possess unique combinations of physical and mechanical properties. For this reason, the last decade has seen an increase in demand of these materials. Figure 1.3 presents the classification of foam into different group of materials. The classification of different types of metallic foams has been done as follows:

- **Cellular Metal:** Space is divided into discrete cells. The boundaries of these cells are made of solid metal, the interior are voids. Ideally, the two individual cells are separated from each other by metal but often this restriction is relaxed.
- **Porous Metal:** The metal contains a multitude of pores, i.e. closed, curved gas voids with a smooth surface.
- **Metal Foam:** Foams are special cases of porous metals. Solid foam is created from a liquid foam in which gas bubbles are finely dispersed in a liquid.
- **Metal Sponge:** Space is occupied by pieces of metal that form a continuous network and co-exist with a network of unfilled space which is also interconnected.



Figure 1.3: Classification of foam within the group of materials [17].

1.6.1 Classification of Foam on the Basis of Cell Structure

On the basis of the cells structures, foams are classified as open cell and closed-cell foam structures [15]. Closed-celled foams do not have interconnected cell openings, so gas is trapped inside the cell. Open-celled foams have interconnected cells, which permit the passage of a gas or fluid through the void space from one cell to the next. Figure 1.4(a-b) presents the closed and open-cell foam structure.

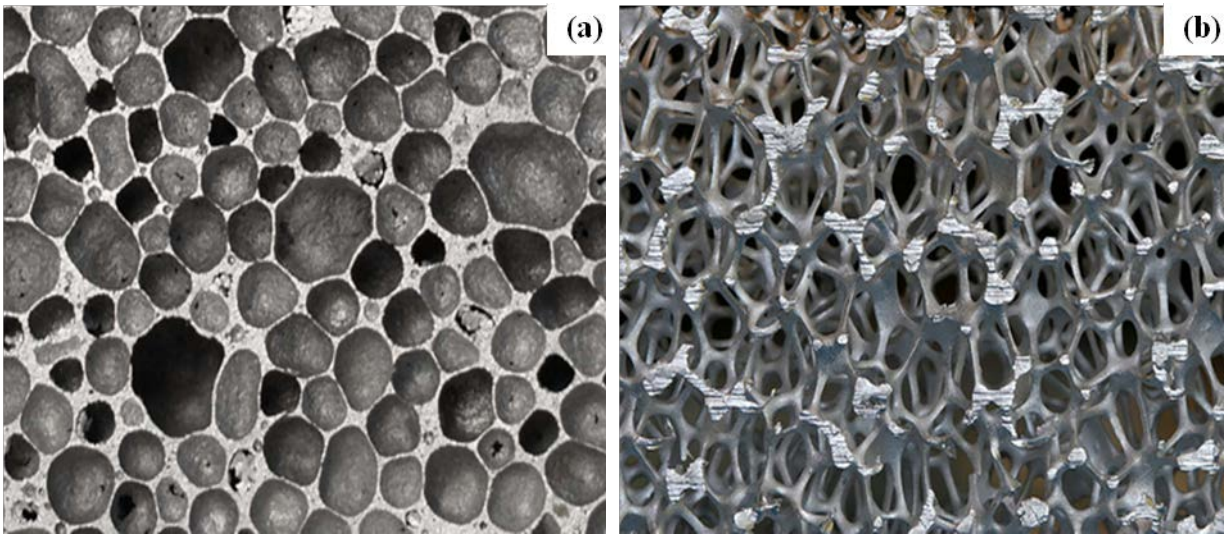


Figure 1.4: Classification of foam on the basis of cell structure (a) closed-cell and (b) open cell foam structures [15].

1.6.2 Closed-Cell Aluminium Matrix Composite Foams (AMCFs)

The past few decades has seen an increase in the development and use of AMCF as a new engineering material [16]. The development of the technologies for the production of AMCF has increased their use in a wide range of applications, including structural sandwich panels and energy absorption devices [17]. Both physical and mechanical properties of AMCF are not only controlled by their composition, they are also affected significantly by cell structure. The cell structure (cell size and cell wall thickness) is controlled by the process variables such as the size and volume fraction of the solid particles, foaming temperature, rate of bubbles formation, and impeller design and impeller speed during the AMCF making process [18]. The morphology of the solid foam obtained by processing is very complex. AMCF are a new, as yet imperfectly characterized class of materials with low densities and novel physical, mechanical, thermal, electrical and acoustic properties [14, 18]. AMCF presents the potential of lightweight structures, for energy absorption, and thermal management and are cheap.

1.7 Manufacturing Methods of Closed-Cell Aluminium Composite Foams

The following methods are used for the fabrication of closed-cell aluminium composite foams viz., foaming liquid melts, metallic precursors, foaming of powder compacts and ingots containing blowing agents [18]. Various processing routes have their unique relative densities and cell structures. On the basis of the state of metal (solid, liquid, powder) used for the fabrications the processing route can further be classified into four “families” viz., liquid metal, solid metal in powdered metal vapour, and metal ion solution (Figure 1.5). There are two main manufacturing methods to develop aluminium foams. The direct foaming methods start with liquid aluminium metal containing uniform dispersion of gas/non-metallic particles and gas is injected to create foam [19]. Indirect foaming methods start from solid precursors which consist of continuous matrix phase with uniform distribution of blowing agent particles [20]. Upon melting this precursor expands and forms a foam.

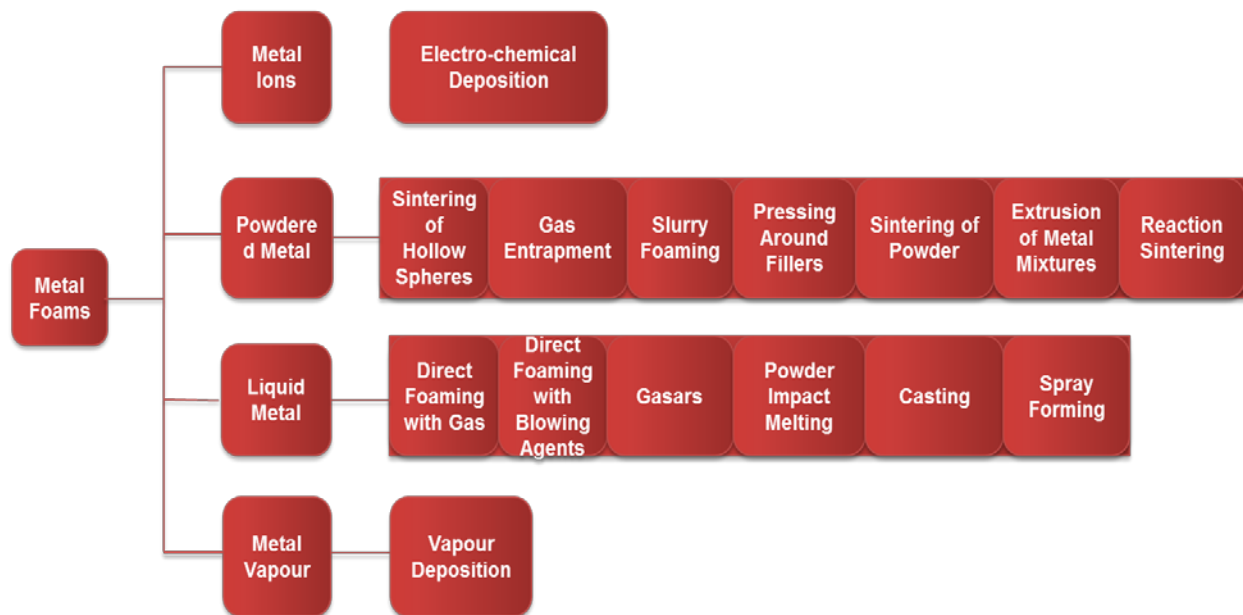


Figure 1.5: Overview of the various families of production methods for metal foams materials [18].

1.7.1 Direct Foaming of Metals

The following processing route directly creates the cellular structure foam from the liquid aluminium. The gas bubbles formed in the metallic melt have a tendency to quickly rise to the surface due to high buoyancy forces acting because of the high-density liquid [20]. The buoyancy forces can be restricted by increasing the viscosity of the molten metal. Although the foams

produced by this method are satisfactory but this method is not popular to produce the foam. Foaming of metallic melts is done directly by injecting gas into the liquid metal from an external source, or to cause an in-situ gas formation in the liquid by admixing gas-releasing blowing agents to the molten metal.

1.8. Factors Affecting the Mechanical Properties of the Closed-Cell Aluminium Composite Foams

Mechanical properties of the aluminium composite foams depend on the gas pressure inside the bubbles and the interaction of reinforcement with the matrix at micro-level of cell walls. Factors like interface, solidification, microstructure, wettability, pore size distribution and cell wall thickness, and distribution of reinforcement in the matrix play an important role in tailoring the properties of the composites.

1.8.1 Microstructure

The characteristics of aluminium composite foams are determined by their microstructure. The microstructure covers the structure of the matrix and the reinforced phase. The chemical composition, cell and cell wall size, node size, ligament length, texture, precipitation behaviour and lattice defects are of importance to the matrix [15-17]. The second phase is characterized by its volume percentage, its type, size, distribution and orientation.

1.8.2 Distribution of Reinforcement

The distribution of reinforcement significantly tailors the properties of the AMCF. However, shape, size and uniform distribution of reinforcement in composite enhances the mechanical properties significantly [21]. Particle-to-liquid density ratio is responsible for the distribution of reinforcement in the matrix [14]. Segregation and clustering of the particles during casting process are major problems, which restrict the uniform distribution of particles in the matrix.

1.8.3 Distribution of Blowing Agent

Distributions of the blowing agent particles also plays a vital role in the formation of uniform cell size of the aluminium composite foam [22]. Segregation and clustering of the blowing agent particles during casting process are major problems, which restrict the uniform distribution of particles in the matrix. Hence for the uniform distribution of particles in matrix it is necessary to get uniform cell size structures [16]. Decomposition of the blowing agent to generate gas in the molten metal plays an essential part in the fabrication of good quality AMCF.

1.8.4 Interface

Chemical interactions and reactions between the matrix and the reinforcement component determine the interface adhesion. This can affect the mechanical properties significantly. The higher strength associated with good interfacial bonding, reduces the ductility of cell wall thereby limiting its applications.

1.8.5 Wettability

The wettability of reinforcement with a metal melt is shown by the edge angle adjustment of a molten droplet on a solid base. The degree of wettability as given below:

$$\gamma_{SA} - \gamma_{LS} = \gamma_{LA} \cos\theta. \quad (1.1)$$

Where γ_{LA} is the surface energy of the liquid phase, γ_{SA} the surface energy of the solid phase, γ_{LS} the interface energy between the liquid and solid phases and θ is the edge angle.

At an angle of $\theta > \pi/2$ a nonwetable system is described and for an angle limit of $\theta < \pi/2$ it is a wettable system. With decreasing angle, the wettability improves [23].

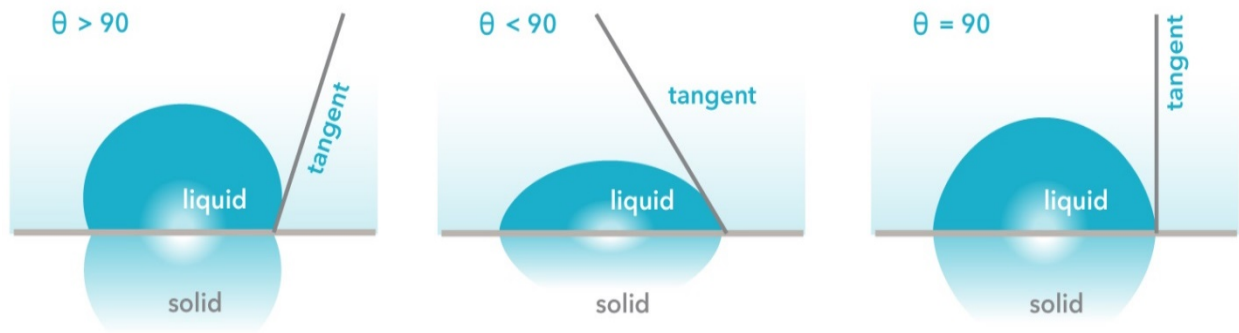


Figure 1.6: Edge angle adjustment of a melt drop on a solid base for various values of the interface energy [30].

1.8.6 Solidification

Phase transition of the material from liquid to solid phase is obtained by lowering the temperature below the melting point of the solid. Solidification affects the characteristics of the aluminium composite foams significantly. Solidification is most widely used for shaping of the foam materials to desired product, as it controls the microstructural and interfacial interaction of the matrix and reinforcement.

1.9 Properties of Closed-Cell Aluminium Composite Foams

1.9.1 Structure

The properties of closed-cell aluminium composite foam materials highly rely on the structure of the material. Because it has complex architectures and microstructures, the measurement and parameterization of their structural characteristics still represent important challenges. Besides density and cell size, the properties of the material are strongly affected by many structural parameters such as the shape and size of the cells, distribution, density, surface area, etc. [15-17, 22].

1.9.2 Mechanical Properties

At a constant peak stress, closed-cell aluminium foams can absorb very high amount of impact energy. So, these are suitable for crash (impact) and blast-mitigation applications [24]. Good stiffness of these closed cell foams makes them suitable for lightweight structural applications. Mechanical properties are strongly influenced by the cell size variation [15].

1.9.3 Damping and Acoustic Properties

Metal foams act as excellent dampers for vibration and also for sound absorption. It is also observed that foams have exceptionally higher sound damping capability in the mid-frequency range.

1.9.4 Electromagnetic Wave Shielding

Electromagnetic wave shielding is applied to shield electronic devices and room interiors from the negative impact of electromagnetic waves. The ability to reflect the electromagnetic energy is referred to as shield effectiveness.

1.10 Applications of Closed-Cell Aluminium Composite Foams

Aluminium composite foam materials are widely used in different applications. The overall composite foam materials market is mainly divided in the following major commercial segments to cover its thousands of products.

- **Aircraft/Military**

Military and commercial aircrafts, components for aerospace, blast mitigation and related applications.

- **Automotive/Transportation**

The largest of the markets, products include parts for automobiles, trucks, rail and farm applications.

- **Civil Infrastructure**

A relatively new market for aluminium composite foams, these applications include the repair and replacement of civil infrastructure including buildings, roads block quote bridges, piling, etc.

- **Construction**

Includes materials for the building, offices and architectural components. Products include swimming pools, bathroom fixtures, wall panels, roofing block quote architectural cladding.

- **Electrical**

This encompassing market includes components for both electrical and electronic applications such as pole line hardware, substation equipment, microwave block quote antennas, etc.

- **Marine**

Products for naval, commercial and pleasure boats/ ships.

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CHAPTER 2

LITERATURE REVIEW

Overview

In this chapter, the work done by different researchers for fabrication of reinforced aluminium matrix composites and composite foams along with their mechanical properties has been presented. Since the present work pertains on development of composites by stir casting technique so the focus of this chapter is to present the study about the development of aluminium matrix composites and its foams by stir casting process. Different physical, chemical, mechanical and tribological properties of composite and composite foams have also been given. The chapter also contains the study about the role of ceramic particles in the Al-alloy composites and foam on the mechanical properties and blast mitigation.

The versatility of aluminium and its alloys makes it the most extensively used metal after steel. Aluminium being one of the lightest engineering metals has superior strength to weight ratio compared to that of steel. Typically, different alloying elements can be added to aluminium viz., magnesium, silicon, copper, manganese, zinc, etc. The function of these alloying elements is to provide strength needed for various applications. High strength to weight ratio makes aluminium and its alloys as preferred choice for aerospace, defence and other industries. Now a day's researchers have shifted their interest towards aluminium matrix composites (AMCs) with hard ceramic particles as reinforcement in the aluminium/aluminium alloy matrix. Badia et al. [1] worked on particle reinforced metal matrix composite. They injected nickel-coated graphite powders in aluminium alloys in presence of argon gas into a molten bath of the alloy. Ray [2] was the first to use stir casting technique to develop Al-Al₂O₃ composites by incorporating Al₂O₃ particles in aluminium matrix. In mid-sixties the most commonly used metals for the fabrications of MMCs were Mg, Ti, Cu, and Al. These are light in weight but their use is limited because of their low melting point and higher reactivity with oxygen to form oxides. Although, titanium alloys have high melting point and higher strength/weight ratio but manufacturing process is complicated because of their affinity towards hydrogen, oxygen and nitrogen. Aluminium alloys are most widely used as metal matrix for development of AMCs because of low cost, low density, high strength, high toughness and resistance to corrosion. Silicon is an important alloying element, added to aluminium to improve its mechanical properties such as tensile strength, hardness and wear resistance. It also reduces thermal expansion and increases fluidity of the molten alloy which enables thin sections to be cast. Magnesium is usually added to improve the wettability, Mg acts as a wetting agent in order to enhance wettability between the ceramic particles and matrix alloy. Other elements such as titanium, nickel, strontium and phosphorus are added to refine the microstructure [3].

2.1 Al-Si Alloys as Matrix in AMCs

Al-Si alloys have excellent corrosion resistance, high specific strength, excellent castability and good wear resistance properties. Commercial grade Al-Si alloys are selected for development of AMCs because they offer good mechanical properties, easy availability and many of them are suitable for heat treatment. Applications of Al-Si alloys based AMCs are in the areas of defence, automotive, aerospace and light vehicles as has been well documented by Babalola [4]. The silicon content in commercial cast Al-Si alloys is in the range of 5-23wt%. Depending on the Si concentration in weight percentage, the Al-Si alloy systems are divided into three major categories as hypoeutectic (Si < 12.6%), eutectic (Si = 12.6%) and

hypereutectic ($\text{Si} > 12.6\%$). Phase diagram of the Al-Si binary alloy is shown in Fig. 2.1. There is only one invariant reaction in this diagram, namely



Where, L is the liquid phase, α is predominantly aluminium, and β is predominantly silicon. The eutectic reaction takes place at 577°C and at a silicon level of 12.6%. Binary Al-Si alloys, near to the eutectic composition exhibit acicular or lamellar eutectic silicon which is in the form of large plates with sharp sides and edges. For Al-Si alloys, the eutectic composition is a structure of $\alpha + \text{Si}$ rather than $\alpha + \beta$.

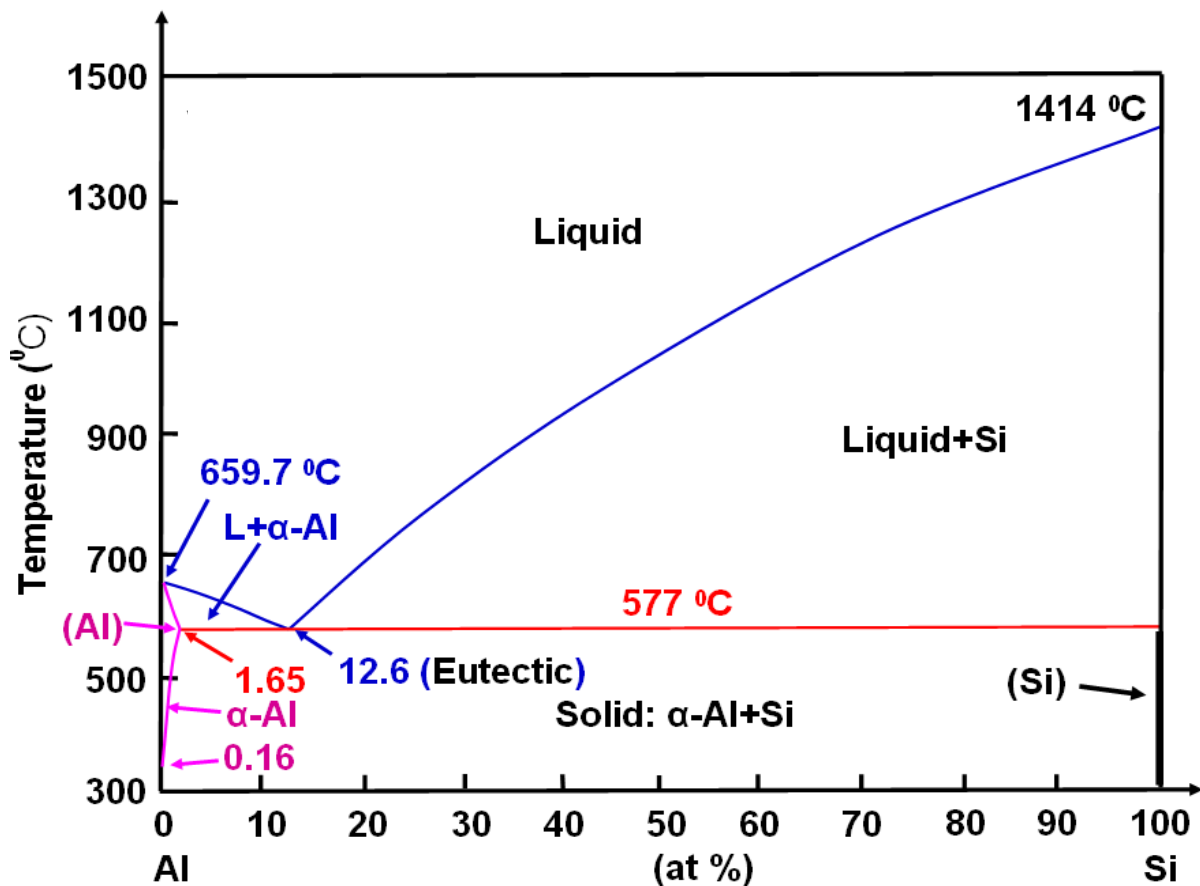


Figure 2.1: Aluminium-Silicon binary phase diagram [5].

As cast eutectic alloys reveal a coarse acicular silicon structure. The coarse silicon flakes being brittle in nature, significantly lowers the strength and ductility of the Al-Si alloy. During solidification, the primary aluminium grows in dendrites and silicon phase grows in angular primary particles. At the eutectic point, the Al-Si phases nucleate and grow until the solidification is complete. While, hypoeutectic alloys largely consist of soft and ductile primary aluminium phase and hypereutectic alloys usually contain coarse angular primary silicon particles [6]. Al-Si alloys having near-eutectic composition have low melting point which

makes them cheaper to cast. The brittle nature of Si flakes in near eutectic composition of Al-Si alloy can be modified according to the requirement. Hernández-Méndez et al. [7] suggested that the presence of nickel (Ni) in Al-Si alloy makes an intermetallic compound named as Al_3Ni which is responsible for the improvement in the hardness, compression and flexion resistances of Al-Si alloy. Kaur et al. [8] in their work found that the addition of Sn in Al-Si alloys provides lubrication for rubbing surfaces which helps to reduce the friction in the tribological applications. Abedi and Emamy [9] in their work found that addition of Mn improves the tensile strength, corrosion resistance as well as significantly improves the low-cycle fatigue resistance in the Al-Si alloys.

2.2 Reinforcement of Ceramic Particles Used in AMCs

Some of the desired properties of the composites include low density, compatibility with matrix alloy, chemical stability, thermal stability, high compression and tensile strength with economic efficiency in production. Aluminium and its alloy being light in weight provide a wide range of applications in the field of architecture, transportation and packaging. However, in comparison to ferrous alloys, Al-Si alloys possess inferior strength, rigidity and wear resistance. This has promoted the researchers to improve the properties of Al-Si by using these as matrix in AMCs [10]. Reinforcement in Al-Si alloys is of great interest, due to large possibility to change the properties of materials with ease manufacturing process. The reinforcement phase in Al alloy matrix is generally hard ceramic particles such as SiC, TiC, B_4C , Al_2O_3 and ZrSiO_4 [11–15]. The volume fraction of reinforced particles or whiskers is generally within the range of 10-30%. Easy availability, low cost, independence of mechanical properties from particulate orientation and production via a wide range of manufacturing routes are the main features for the ceramic particles to be used as reinforcement phase in AMCs. Addition of hard ceramic particles in the AMCs improves their hardness, Young's modulus and abrasion wear resistance; however, it also results in deterioration of their plastic properties.

2.3 Manufacturing Methods for AMCs

Two common methods to manufacture aluminium MMCs are powder metallurgy and liquid metal processing. Casting method is most preferred method for production of aluminium AMCs. Stir-casting technique is currently the simplest and most commercial method of production of AMCs [16]. This process involves mechanical mixing of the reinforcement particulate into a molten metal bath followed by transferring the mixture directly to a shaped mould prior to complete the solidification. In this process, the crucial thing is to create good

wetting between the particulate reinforcement and the molten metal [17]. Microstructural inhomogeneity can cause notably particle agglomeration and sedimentation in the melt and subsequently during solidification. Inhomogeneity in reinforcement distribution in these cast composites could also be a problem as a result of interaction between suspended ceramic particles and moving solid-liquid interface during solidification [18]. This process has major advantage that the production cost of AMCs is very low. One very crucial issue to consider in selection of the matrix material involves the natural dichotomy between wettability of the reinforcement and matrix [17]. Good load transfer rate (matrix to reinforcement) is dependent upon a strong adherent interface [17,18]. A strong interface requires adequate wetting of the reinforced particulate by the matrix phase. However, wettability and aggressive reactivity are both favored by strong chemical bonding between the matrix and reinforcement. Adjusting the chemical composition to accomplish this delicate compromise is difficult as many subtleties are involved.

Since, the present work is based on the development of composite reinforced with minerals, so the literature pertaining to minerals reinforced composite have been presented here. Literature related to mineral reinforced composites in concise form has been discussed below in Table 2.1.

Table 2.1: Tabulated summary of outcomes of various investigators on the development of minerals reinforced AMCs.

Investigator [Year]	Materials [Matrix/Reinfor cement]	Process	Properties /Parameter Investigated	Outcomes
Singh et al. [19] in 2001	Al-Si alloy (BS: LM6) with Sillimanite (100- 170µm)	Stir casting	Hardness/ Microstructure/ Wear resistance	• Micro-hardness of LM6 alloy matrix increased from 57 to 85HV due to the uniform dispersion of sillimanite particles in the matrix. • Ultimate tensile strength and percentage elongation of LM6 alloy composite decreased marginally. • The microstructure characterization revealed a uniform distribution of sillimanite particles with good mechanical bonding between the matrix alloy. • Micro-hardness and abrasive wear resistance of the composite improved significantly over the matrix alloy.
Singh et al. [20] in 2002	Al-Si alloy (BS: LM6) with Sillimanite (50- 150µm)	Stir casting	Hardness/ Microstructure/ Wear resistance	• The microstructure of LM6 alloy composite showed reasonably uniform distribution of sillimanite particles in the matrix alloy with good interfacial bonding. • Wear resistance of the LM6 alloy composite was superior to that of matrix alloy for finer size abrasives, whereas the trend reversed for coarser size abrasives. The coarser abrasives are responsible for higher wear rate than the alloy beyond a critical applied load. • Wear rate decreased with increase of sliding distance. This may be due to work hardening of wear surface, clogging, attrition and shelling of abrasive particles.
Chaudhary et al. [21] in 2005	Al-2Mg alloy with Rutile TiO ₂	Stir and spray casting	Thermal conductivity/ density	• The density of spray formed composites was greater than stir cast composites. • Thermal conductivity of spray formed Al-2Mg-TiO₂ composite was greater than Al-2Mg alloy. The specific heat of spray formed composite was greater than the matrix alloy, while the thermal diffusivity of the former (composite) was lower than the latter (alloy).

Kumar et al. [22] in 2006	Zinc-Aluminium (ZA-27) alloy with Garnet (50-70µm)	Stir casting	Wear/ friction coefficient	- The reinforced garnet particles exhibited reduced wear rate than the unreinforced alloy. The wear rate decreased with increase in garnet content. - The dry sliding wear rate of composites as well as the matrix alloy increased with the increase in speed of the disc and load applied. - The friction coefficient of zinc-AMC decreases with increase in reinforcement from 0 - 20 wt.%.
Abdizadeha et al. [23] in 2008	(A356.1) with Zircon ($ZrSiO_4$) and titanium diboride (TiB_2)	Stir casting	Microhardness/ tensile strength	- The improvement of hardness and tensile strength of aluminium by incorporation of $ZrSiO_4$ and TiB_2 particles is due to the high strengthening and work-hardening at low strain in the composite materials. - The best property values for Al-5% $ZrSiO_4$ are achieved at 750 °C and for Al-5% TiB_2 at 950 °C because $ZrSiO_4$ has a lower thermal expansion than of TiB_2. - The wettability of TiB_2 with aluminium is better than of $ZrSiO_4$, opportunity for a larger fraction of TiB_2 particulates to be incorporated in the matrix, the value of microhardness and tensile strength is higher.
Niyomwas [24] in 2010	Al ingot (A356 brand) with Fe_2TiO_3 (ilmenite)	In-situ process.	Interface analysis	- Titanium diboride alumina-iron aluminide composites were in-situ produced from ilmenite-boron dioxide-aluminium mixtures taken in mole ratios of 1:1:5, 1:1:6, and 1:1:7. At a lower molar ratio of Al, the product contained $FeAl_3$ with a small mixture of unreacted $FeTiO_3$. - The reactions went to the end and yielded the TiB_2-Al_2O_3-$FeAl_3$-Fe_3Al and TiB_2-Al_2O_3-Fe_3Al composites, respectively. The products exhibited good inter-particle cohesion within agglomerated particles of Fe-TiB_2, well mixed with surrounding Al_2O_3.
Miranda et al. [25] in 2015	Al-Si alloy with Ti and SiC	Powder metallurgy	UTS/wear	- Al-Si-11.25%Ti-5% SiC hybrid composite exhibited the highest UTS while retaining a good level of ductility, unachievable by using this reinforcement content exclusively with SiC. - Reinforcing Al-Si alloy with Ti_p led to a wear behavior improvement. Al-Si-11.25%Ti-5% SiC hybrid composite show the 32% reduction in wear rate when compared to Al-Si alloy.
Prasad et al. [26] in 2015	LM13 with Garnet and carbon	Stir casting	UTS properties	- UTS properties of the composite increases as garnet amount is increased up to 9 wt.%, beyond which it drops again. - A dispersion amount of 9 wt% Garnet + 3 wt% carbon with copper chill was found to increase the mechanical properties.

Zhang et al. [27] in 2015	Al2014 aluminium alloy with Nano-SiC particles	Stir casting	Microstructure/ Tensile properties	- Aluminium composites exhibited refined α-Al grains which is reasonable to uniform distribution of nano-SiC particles. - Uniform distribution of nano-SiC particles in matrix is reasonable for good interface bonding and high dislocation density. - The superior tensile properties of the nano-SiC particles composites were observed.
Arora et al. [28] in 2015	LM13 aluminium alloy with rutile	Stir casting	Microstructure/ Microhardness/ Wear	- The hardness of the composite increased with the increase in reinforcement content. - High temperature wear study indicates a transition in wear mode from mild to severe at temperature above 150 °C for all the developed composites. - The analysis of wear track and debris indicates that the formation of MML is responsible for the reduction in wear rate below the critical transition temperature (150 °C). Above this temperature, the removal of MML followed by plastic deformation at high temperature is the main cause behind the high wear rate.
Arora et al. [29] in 2015	LM13 aluminium alloy with rutile dual size particles	Stir casting	Microstructure/ Microhardness/ Wear	- Rutile particles inclusion in the matrix modifies the microstructure and in turn improved the wear behavior of the composite. - Dual particle size composites proved to be a better wear resistant material under the high contact pressure as compared to single particle composites. - At a critical pressure, change in wear from mild to severe wear transition takes place which is confirmed from the morphology of the wear tracks and the wear debris.
Kumar and Rajadurai [30] in 2016	Atomized aluminium powder with SiC and TiO ₂	Powder metallurgy	Microstructure/ Wear	- Rutile increases the density and microhardness of the hybrid composites. The density and microhardness show remarkable improvement with the mass fraction of TiO₂ (rutile). - Al-SiC-TiO₂ hybrid composites display better wear resistance than the unreinforced Al-SiC composites and the base matrix. Al-15%SiC reinforced with 12% TiO₂ revealed the best wear resistance.

2.4 Introduction of Aluminium Matrix Composite Foams (AMCFs)

Benjamin Sosnick [31] was the first one to invent metallic foam at San Francisco, California. He developed a “sponge metal” using mercury as a foaming agent in molten aluminium. It was not until the 1990, however, that an intensified research effort began. This effort was successful in developing new technologies that have brought several porous structures to the marketplace. The list of metals and its alloys that have been foamed is numerous. Among them the most popular metals are aluminium, iron and titanium. According to Banhart and Baumeister [32] liquid and powder metallurgy are common techniques which are used to manufacture a larger quantities of porous castings. Porous castings form a subgroup of cellular metals, usually having polyhedral cells but cell boundary and shapes may vary in cases where directional solidification creates different type morphologies and are also called as metal foams [33]. The porous structures can be classified into two types based on their pore structure which is known as open cells or closed cells or a combination of the both. They may be either closed with membranes separating adjoining cells or open if there are no membranes across the faces of the cells so that the voids are interconnected. Babcsan et al. [34] have stated that solid foams originating from liquid are closed. The characteristic properties of foam are defined by the cells structures with relative density. Open cell can be thought of as a structure network of interconnected solid struts. The open cell will allow fluid media to pass through it [35]. Khayargoli et al. [36] reported that open pore generally has a cellular structure made up of 3-dimensional interconnected network of solid plates that form the edges and faces of the cells. Closed cell is made up of a network of adjacent sealed pores, all sharing walls with each other. A fluid media cannot pass through closed cell. A combination of open cell and closed cell composition is technologically possible [37].

2.5 Manufacturing Methods for AMCFs:

Aluminium matrix foam (AMF) and aluminium matrix composite foam (AMCF) generally arise by nucleation and subsequent growth of gas bubbles in a liquid or semi-liquid metal. The distribution of gas bubbles is therefore pretty random. The nature of gas bubbles is initially closed although some cracks or openings can be found in the cell walls during the solidification and cooling. These are the main features of this kind of porous materials. Metallic foams can be mainly prepared by direct foaming of the liquid or by powder metallurgical (PM) techniques. Molten metal is foamed by the addition of gas blowing agent in the liquid alloy [38, 39]. Metallic hydrides such as Mg, Ti, Zr releasing hydrogen or carbonates releasing H_2/CO_2 are

generally used as blowing agents. The presence of hydrogen, or carbonates releasing gas in the molten metal requires considerably higher viscosity, that is achieved by additional alloying, e.g. with B, Na, Ca or by addition of ceramic particles [40]. Though the process of direct foaming of the molten metal is economically most efficient but it is difficult to control the structure. Moreover, the complex shapes of cells cannot be generated under controlled fabrication parameters. As the molten foam cannot be cast yet, the direct foaming fabricating method is limited to the production of foam blocks at large scale that can be machined to simple shape components. Developed aluminium foam in this way exhibits closed cell structure with the total porosity in the range ~80-95 % and is being used mostly in combination with other porous materials for many purpose such as absorbers in blast or crash protection, damping and insulating [40]. Srivastava and Sahoo [41] dispersed gas as small bubbles in a melt by a rotor impeller. The walls of the bubbles created are stabilized by refractory particles, avoiding coalescence between them. Many researchers used different manufacturing processes. Some of them are summarized in Table 2.2.

Table 2.2: Tabulated summary of outcomes of various investigators on the manufacturing methods of AMF and AMCF.

Investigator [Year]	Materials [Matrix/Reinforcement/Blowing agents]	Process	Properties /Parameter Investigated	Outcomes
Asholt [42] in (1999)	AA336 Al chips/NaCl powder	Sintering-Dissolution Process (SDP)	Porosity, Compressive, Yield stress/ elastic modulus	- The fabricating of aluminium foams from turning scraps through the SDP was confirmed. - Melting of AA336 chips is essential for a continuous and strong network structure to form in the preform. - An increase in the wt.% of chip of Al/NaCl mixture increased the strength of the cell walls by decreasing the number of cells. This increased the yield stress and the deformation plateau of the produced foams.
Thomas et al. [43] in (1997)	Metal/Blowing agent	Melt gas injection (air bubbling) Pure	Bubble size/ Porosity	- Gas-injection processes are easiest to implement with Al-alloys because they have a low density and do not excessively oxidize when the melt is exposed to air or other gases containing oxygen. - A variety of gases can be used to create bubbles within liquid aluminium. Bubbles formed by this process float to the melt surface, drain, and then begin to solidify.
Miyoshi et al. [44] in (1998)	Metal/Blowing agent	Gas-releasing particle decomposition in the melt metal	Bubbles/ Viscosity	- Metal alloys can be foamed by mixing into them a foaming agent that releases gas when heated. The widely used foaming agent TiH_2 begins to decompose into Ti and gaseous H_2. TiH_2 particles added to an aluminium melt, large volumes of hydrogen gas are rapidly produced, creating bubbles that can lead to a closed-cell foam. - Foam drainage is sufficiently slow, which requires a high melt viscosity. The Shinko Wire Company has developed an aluminium foam trade named Alporas using this approach.

Yu and Eifert [45] in (1998)	Aluminium/ blowing agent	Particle decomposition in semisolids	Bubble formation	• Foaming agents can be introduced into metals in the solid state by mixing and consolidating powders. • The possibility of creating a foam by dispersing the blowing agent in solid aluminium using powder metallurgy processes and then raising the temperature sufficiently to cause gas release and partial or full melting of the metal, allowing gas bubble growth. Cooling then stabilizes the foam.
Stanzick [46] in (2002)	AlSi6Cu4/SiC TiH ₂ particles	Expanding foamable precursors	Cell morphology	• AlSi6Cu4/SiC foams were developed by expanding foamable precursors containing a gas releasing blowing agent in a dense metal matrix. • The difference in cell formation between the two processing technique was studied. Rupture of cell walls during foam expansion could be visualized. • Defects in the precursor material induce the local void formation but have little influence on the morphology of the fully expanded foam in the cases studied.
Ozan and Bilhan [47] in (2008)	A6061/ TiH ₂ powder	Hot powder extrusion	Porosity/ Compressive strength	• Aluminium foams directly from powder for automotive parts, the combined process of hot powder extrusion and foaming was proposed. • The A6061 aluminium powder mixed with TiH₂ powder as a foaming agent was extruded into the die heated upto melting temperature and the aluminium foam was made. • The metallic reinforcements implemented into the foam surface significantly improve the strength, plasticity, energy absorption capacity, fracture toughness and damage tolerance of foamed part with only a little weight increase. • The reinforcements can be effectively used also for improving the bending stiffness when the thickness of the foam is limited. • They ought to be placed and oriented in the vicinity of foam surface according to the way of expected loading.
Yang et al. [48] in (2008)	AZ91/AM60/ CaCO ₃ powder	Melt foaming method	Decomposition rate of CaCO ₃	• The blowing gas to foam the melt is not CO₂ but CO, which comes from liquid-solid reaction between Mg melt. • The reaction temperature is more than 100°C lower than CaCO₃ decomposition, which makes Mg alloy melts foam into cellular structure much more easily foamed in the temperature range from 690°C to 750°C. • As is the case with dense metals, the strain rate sensitivity of the foam was not a constant value, but found to be dependent on the strain and incremental change in strain rate.

Mondel et al. [49] in 2014	Zinc aluminium alloy (ZA27)/SiC/ CaH ₂	Stir-casting technique	Compressive / plateau stress/ energy absorption	• ZA27-SiC foam is characterized in terms of its micro-architectural characteristics and deformation responses under compressive loading. • The density of the developed ZA27-SiC foam ranges from 0.25 gm/cc to 0.45 gm/cc due to the variation in amount of CaH₂. • The plateau stress and energy absorption of these foams follow power law relationship with relative density. Wherein, the densification strain follows a linear relationship with the relative density.
Zhang et al. [50] (2015)	Aluminium/ Calcium granules/TiH ₂ powders	Modified melt foaming method	Compressive property/ Multi-walled carbon/ Nanotubes	• Closed-cell aluminium foams with different contents of WCNTs were fabricated by using a modified melt foaming method. • The yield strength, structural stiffness and energy absorption capacity of the foams with MWCNTs content of 0.5% were optimal under the experimental conditions. • With increasing MWCNT content the yield strength, structural stiffness and energy absorption capacity of the foams increased first and then decreased.

2.6 The Effect of Processing Parameter on Cell Geometry and Mechanical Behavior of AMCFs

According to Banhart and Baumeister [32] blowing agent, stirring speed and temperature are the important process parameters that influence the cell formation in the casting. The shape and size of the cells depend directly on the process parameters [51]. Du et al. [52] pointed out that the pouring temperature of the molten metal should be maintained such that it fills all the cavities or voids before solidification starts. Shiomi et al. [53] worked on the fabrication of aluminium foams from powder by hot extrusion and foaming. As received aluminium powder A6061 was foamed with TiH_2 at a temperature of 420 °C. The die temperature was kept at 720 and 760 °C. It was observed that the relative density of the aluminium foam was 0.22. The energy absorbing capacity of the pipe increased with the aluminium foam filled by the proposed method. Mondal et al. [54] used cenosphere as space holder to make Ti-cenosphere syntactic foam through powder metallurgy route. Microstructural characteristics, density vis-a-vis porosity, compressive deformation behavior and modulus of elasticity of the sintered samples were examined. It was noted that the porosity varies in the range of 51-70% depending on the pressure applied during cold compaction. A numerical model has been proposed to correlate density of foam with the extent of cenosphere crushing, density of cenosphere and density of titanium. Mukherjee et al. [55] worked on the effect of cooling rate on the structure and properties of closed-cell aluminium foams. Al foams ($\text{AlSi}_6\text{Cu}_4+0.5\text{wt.}\% \text{TiH}_2$) were prepared by powder processing from elemental powders of Al, Si and Cu and the blowing agent titanium hydride. Powder blending method was used for the fabrication of the foam. Foaming was conducted inside a furnace and the entire process was monitored in-situ by means of X-ray radioscopy. It was observed that the variation in cooling rate was a good strategy for enhancing the strength of aluminium foams through modification of the macro and micro-structure. Among the three cooling rates studied, the medium one produces the best result in terms of a more uniform cell size distribution and the presence of a greater number of smaller cells. However, a further increase in cooling rate lead to adverse effects and the structure deteriorated. The micro-porosity and grain size in the cell walls decreased with increase in cooling rate. The effects of manufacturing processing parameters on the cell geometry and mechanical behavior of AMCFs are discussed briefly in Table 2.3.

Table 2.3: Tabulated summary of outcomes of the effect of processing parameters on cell geometry and mechanical properties of AMF and AMCFs.

Investigator [Year]	Materials [Matrix/Reinforcement/ Blowing agents]	Process	Properties/Parameter Investigated	Outcomes
Gui et al. [56]in (2000)	A356/SiC /TiH ₂ powders	Stir casting	Holding temperature/ Stirring speed/Size of the impeller/Position of the impeller in the melt	• The tensile stress-strain curve shows a quite low fracture elongation and no distinct plastic deformation. • The compressive stress-strain curves of the composite foams demonstrate three distinct regions: the linear elastic region, the crushing collapse region and the densification region. • The relative density of the composite foams exhibits a significant effect on the damping capacity, while its influence on the dynamic modulus of the composite foam is very small.
Paul and Ramamurty [57] in (2000)	Aluminium /TiH ₂ powders	As cast/ Heat treatment	Plastic strength /Energy absorbed	• The plastic strength and the energy absorbed increased with increasing strain rate. • The plastic strength was found to increase bilinearly with the logarithm of strain rate, whereas dense metals tend to show only a linear response. • In dense metals, the strain rate sensitivity of the foam was not a constant value, but found to be dependent on the strain and incremental change in strain rate.
Stanzick et al. [46] in (2002)	AlSi6Cu4/SiC/ TiH ₂ particles	Expanding foamable precursors	Foam morphology / Defects	• AlSi6Cu4/SiC foams were developed by expanding foamable precursors containing a gas releasing blowing agent in a dense metal matrix. • The difference in cell formation between the two processing technique was studied. Rupture of cell walls during foam expansion could be visualized. • Defects in the precursor material induce the local void formation but have little influence on the morphology of the fully expanded foam in the cases studied.

Mondal et al. [58] in (2009)	Aluminium /cenosphere /CaH ₂	Stir casting	Compression test/Plateau stress/Density	• The stress-strain curve for all tested foam samples had four characteristics: an initial linear elastic region, a yield point, a plateau region and densification. • The densification strain was found to be invariant to the relative density and strain rate. • The plateau stress was highly dependent upon the relative density and marginally upon the strain rate. • Plateau stress was found to be insensitive to strain rate. Thus, the effect of strain rate on the compressive deformation response for light aluminium foam is insignificant.
Da-wu et al. [59] in (2011)	Pure aluminium foamed with ZrH ₂ , and Ca	Stir casting	Cell structure/ Compression tests/ Stirring time/Holding time	• Ca addition, ZrH₂ content, processing temperature, stirring time and holding time led to the production of closed cell aluminium foam with a uniform cell structure and high porosity. • The average cell size and porosity changed a little with 0.6%-1.2% ZrH₂, while the average cell size and porosity increased with increase in foaming temperature and shows essentially linear relationship with Ca addition. • Aluminium foam with average pore diameter of 1.2 mm and uniform distribution was obtained under the optimum parameters with the addition of 1.0% ZrH₂ and 2.5% Ca, at temperature of 680 °C and stirring time and holding time of 1.5 min and 2.5 min, respectively. • Intermetallic compounds ZrAl₃ and Al₂O₃ increased viscosity of the melt.
Sarajan et al. [60] in (2011)	Al-Si alloy/ TiH ₂	Melt foaming method	Cell structure/ Porosity	• The existence of the aluminium powder in the melt increased the viscosity. With an increase in the time, the number of cells increased. • The cell size and the total porosity volume increased with an increase in TiH₂, H₂ gas is completely absorbed by the melt and largely increased the foamed. • Properly control over viscosity and solidification a good uniformity of the cell structure of the Al-6wt.%Si foams can be achieved.

Byakova et al. [61] in (2014)	Al-Zn-Mg alloy foamed with CaCO_3	Alporas-like route	Sound Absorption Ability	- Enhanced sound absorption ability of Al-foam was provided due to opening the closed cell structure by micrometer cracks lengthways. - A number of interconnected cells reduced the relative density down to critical value, as the cell wall thickness became comparable with dimension of Al dendrites the global opening of the cell structure took place. - This favors greater dissipation of sound waves thus, ensuring a significant increase of sound absorption over the entire range of frequencies.
Byakova et al. [62] in (2014)	Pure aluminium, Al-7Si, Al-5Mg and Al-Zn-Mg alloy foamed with TiH_2 and CaCO_3	Alporas like route	Closed-cell structure/ Compressive test	- No discrepancy between experimental values of compressive strengths for Al-foams comprising ductile cell wall constituents and those prescribed by theoretical models for closed-cell structure was found. - The presence of low ductile and/or brittle eutectic domains and contaminations including particles/layers of Al_3Ti, residues of partially reacted TiH_2, and Ca bearing compounds, results in reducing the compressive strength to values close to or even below those of open-cell foams of the same relative density.
Kumar et al. [63] in (2014)	Al6061 alloy foamed with CaCO_3	Stir casting	Density/Porosity/ Micro hardness/Wear Test / Corrosion Test	- Closed cell as well as open cell foam was economically produced in the research laboratory. 21.771 and 19.18 % porosity was obtained for open and closed cell aluminium foam respectively. TiH_2 powder was successfully replaced by commercial CaCO_3 powder in closed cell aluminium sponge production. The properties of aluminium sponge significantly depend on its porosity, which means that a desired property can be tailored by the sponge density. - Co-efficient of friction of the foam is considerably low, this makes them fit for automotive applications. - The Corrosion and wear rate of produced Aluminium sponge is low.
Movahedi et al. [64] in (2014)	A356 aluminium alloy foamed with TiH_2	Stir casting	Compression test/Relative density/Absorbed energy	The microstructure of closed cell A356 foam was strongly affected by increase in the molten liquid temperature during the foaming process.

				• The increase in the process temperature to more than 675 °C led to production of closed cell Aluminium foams with polygonal and disordered shapes. • The mechanical properties of closed cell A356 foams were also influenced by the foaming temperature, which plays an important role in the final A356 foam density.
Sahu et al. [65] in (2014)	ZA27/SiC	Stir casting	Compression test/ Energy absorption	• The presence of a few coarse and fine cells caused the large variation in the compressive stress-strain curves. • At constant temperature plateau stress increased with increase in the strain rate. Whereas, plateau stress increased with increase in relative density but decreased with increase in temperature. Marginal change was observed with cell size. • Energy absorption increased with increase in relative density but decreased with increase in temperature. Energy absorption decreased significantly with the increase in cell sizes.
Uzun and Turker [66] in (2014)	Alumix 231 powder foamed with TiH ₂	Powder metallurgy	Volume expansion/ Density/ Porosity	• With the increase in the deformation rate and foaming temperature, there was an increase in the volume expansion rate of foam and decrease in the maximum foaming duration. • There was a decrease in the foaming duration with the increase in the foaming temperature.
Haidar et al. [67] in (2015)	LM6 Aluminium alloy with SiC/ TiH ₂	Stir casting	Electrical conductivity	• The relative density has a significant effect on the electrical conductivity of this material. • The pre-treatment of TiH₂, changed the electrical conductivity abruptly, the best results were obtained when TiH₂ was pre-treated at 480 °C for 60 minutes, before using it. Hence, the pores produced in the foam at the given temperature were more homogenous.
Gawdzińska et al. [68] in (2016)	Al-Si alloy with SiC	Blowing gas in the liquid	Strength	• Different pore sizes and average porosity had no effect on the strength of the foam samples. • Lower total porosity higher stress is necessary for strain to occur. • The amplitude of the fluctuations was related to foam density. The greater the density, the larger fluctuations of stress occurs during strain.

2.7 Structural Properties

There are several structural parameters of these foams, such as cell number, cell-size distribution, average size, shape and geometry of the cells, thickness, intersections and defects in the cell-walls and thickness, defects and cracks of the external dense surface for describing the cellular architecture of the foams. The properties of these foams are influenced by these morphological features [69,70].

Progress has been made in understanding the relationship between properties and morphology. Although this exact interrelationship is not yet sufficiently known, one usually assumes that the properties are improved when all the individual cells of foam have similar size and spherical shape. This has not really been verified experimentally. There is no doubt that the density of metal foam and the matrix alloy properties influence the modulus and strength of the foam. All studies indicate that the real properties are inferior than the theoretically expected due to structural defects. This demands a better pore control and reduction in structural defects. Density variation and imperfections yield a large scatter of measured properties, which is detrimental for the metal foams reliability [71]. Wiggled or missing cell-walls reduce strength, and in turn, result in a reduced deformation energy absorbed under compression [72]. Mechanical studies demonstrate that selective deformation of the weakest region of the foam structure leads to crush-band formation [73]. Cell morphology and interconnection could also affect thermal and acoustic properties [74].

2.8 Blast Mitigation

Explosives create a pressure wave of approximately triangular profile, known as a ‘blast’ [75]. Protection against blast is an important application in defence. The blast imparts an impulse, J_i per unit area, which is equal to the integral of the pressure with respect to time as shown in equation (2.2):

$$J_i = \int p \, dt \quad (2.2)$$

The pressure-time transient of the blast wave depends upon the orientation of the structure with respect to the pressure wave. The blast wave is reflected by a rigid structure, in design, it is conservative to assume that the structure normal to the incident blast wave fully reflects the blast.

Miyoshi et al. [76] worked on the impact energy absorbing capacity of metallic foam during the crash testing of the vehicle. In this study, the impact energy absorbing capacity of close-cell foam structure was enhanced by increasing the aspect ratio (ratio of cell-wall thickness to

cell-edge length) with the reduction of cell size. Shim et al. [77] observed that foams have low density and are attractive materials to mitigate high-speed pressure by blast loads due to high-energy absorption capabilities. Thickness, density and skin plate properties of the panel are the most important parameters to estimate the transmitted pressure to protective structures. The pressure of close range blast loading is not uniform. Numerical simulations considering the above parameters provided basic design guidelines for the protective structures with sacrificial foam panels. Properly designed panels for the required blast loads can control the transmitted pressure. Zhu et al. [78] studied the deformation and failure of blast-loaded metallic sandwich panels. Metallic sandwich panels with a cellular core such as honeycomb have the capability of dissipating considerable energy by large plastic deformation under impact/blast loading. To investigate the structural response of sandwich panels under the blast wave, numerous experiments were conducted. Finite element simulations were designed to investigate the effects on the structural response of face-sheet and core configurations, i.e. face-sheet thickness, cell size and foil thickness of the honeycomb, and mass of charge. The experimental data were then compared with the simulation data and the results show a good agreement between the experimental and computational studies. Zhu and Lu [79] studied the blast and impact test of metallic and sandwich structures materials. The energy absorption behavior of structures has been presented comprehensively in a monograph by Luo and Yu [80]. Rajendran et al. [81] investigated the foam materials as an energy absorber for nuclear transportation cask. Closed cell foam was investigated for impact energy absorption characteristics and undergone deflection in foam due to the impact. Compression test was carried out on a foam cylinder to obtain the representative stress-strain diagram and energy-deflection diagram. Mondel et al. [82] studied the moduli of porous materials extensively both analytically as well as experimentally. Several empirical and analytical equations were proposed to express the effective moduli of porous solids. In this investigation, a few of these relations were checked for experimental validity in the case of closed-cell aluminium foams. Mondel et al. [83] studied the deformation response and energy absorption characteristics of closed cell aluminium-fly ash particle composite foam. The investigated foams were made by melt route using CaH_2 as foaming agent. The influence of strain rate on the deformation responses is found to be very marginal; within the domain considered in the present study, and the strain rate sensitivity was measured to be very low and varying in the range of 0.02- 0.04 when the foam relative density is greater than 0.1. The plateau stress, and energy absorption increased significantly following power law relationship. The densification strain was almost invariant to the relative density. For the foam with relative density less than 0.1, the strain rate sensitivity was found to be

negative. Yuen et al. [72] performed experiments and numerical simulation of “V” shape plates with different included angles subjected to localized blast load. Hopkinson Crazz blast scale law was used to determine the range of masses of explosive used for TM-57 anti-tank mine. The results showed that smaller inclusive angles deflect more blast energy resulting in lower mid-point plate deflection. The predictions show encouraging correlation with experimental data for the displacement profile. Spranghers et al. [84] studied the numerical simulation followed by experimental validation of the dynamic response of aluminium plates under free air explosions. Finite element analysis was carried out to study the influence of different parameters viz. element type, element size and integration method with respect to the accuracy. It is shown that for the modeling of the deformation of the investigated thin plate, the use of shell elements is allowed as the transverse shear strains appear to be sufficiently small. Bonsmann and Fourney [85] worked on the blast mitigation and studied the effect of accelerations of mitigation on blast loading utilizing polymeric-coated metallic thin-walled cylinders. Test plates of different shape and size were used for the study. Simple plate geometries were employed to investigate various mitigation parameters. Mitigation through crushing of thin-walled cylinders and the effect applying polymeric coatings to these dynamically loaded structures was studied. It was found that vast benefits are realized from using thin-walled cylinders to mitigate high acceleration values experienced from a blast load. Yin et al. [86] worked on shock mitigation mechanism of cellular cladding and optimized design of double-layer cladding (DLC). It was observed that the actual incident wave impinged on the cladding and interface pressure at the wet face was highly dependent upon the core crushing, fluid-structure interactions and cavitation phenomenon. The actual incident wave was enhanced due to the collapse of cavitation bubbles. The high pressure loading incident on the cladding was converted into a lower magnitude load for much longer duration beneficial for shock mitigation. DLC could absorb more energy and reduce the total foam thickness when properly designed. Luo et al. [87] studied the blast mitigation effect of water filled plastic wall. The maximum overpressure (reflected peak) reached up to 94.53%, while the minimum overpressure (reflected peak) was 36.3%. The scaled distance range was between 1.71 to 3.42 m/kg^{1/3}. The effects of the scaled gauge height, water/charge scaled distance (distance between explosive charge and water wall), water wall scaled height and water/structure scaled distance (distance between water wall and structure) were systematically investigated and compared with the usual rigid anti-blast wall. It was concluded that these parameters affect the mitigation effects of plastic water wall on blast loadings significantly, which was basically consistent to the trend of usual rigid anti-blast wall.

2.9 Plan of Work

2.9.1 Gaps in Study

As discussed above, many researchers have studied about the development of porous aluminium foam materials. However, after analyzing the entire work some gaps in the study exist, which are as follows:

- Limited work is available to develop of porous aluminium foam by stir casting process using ZrH_2 , CaCO_3 and MgCO_3 as a foaming agent.
- Limited data is available on the effect of zircon sand as reinforcement in aluminium foam.
- Limited data is available about the effect of size and amount of zircon sand reinforcement on mechanical properties of aluminium foam.
- To the best of our knowledge, no work is available on the study about blast mitigation test of zircon sand particle reinforced aluminium foam composite.

2.9.2 Objectives

During the research work, our main objectives will be:

- To prepare aluminium matrix composite (AMCs), aluminium matrix foam (AMFs) and aluminium matrix composite foam (AMCFs) materials by stir casting process using CaCO_3 as foaming agent.
- To prepare AMCs and AMCFs with addition of zircon sand particles in different amount with different particles size range.
- To study the microstructural, hardness and wear properties of AMCs, AMFs and AMCFs of prepared materials.
- To test the prepared AMFs and AMCFs for the blast mitigation.

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CHAPTER 3

EXPERIMENTAL DETAILS

Overview:

This chapter describes the details of the raw materials used for the development of the aluminum matrix composite (AMCs) and its composite foam (AMCF). Detailed procedure for the development of the AMCs, AMCF and their characterizations is given in this chapter. Different characterization techniques such as X-ray diffraction (XRD), Optical microscopy, Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), Vickers hardness testing, Wear test with pin-on-disc machine along with their operating parameters and Blast mitigation tests are also discussed. A flow chart of the methodology used is also given in this chapter.

3.1 Raw Materials

In the present work, for the fabrication of aluminum matrix composites (AMCs) and aluminum matrix composite foam (AMCF), we choose LM13 alloy (an alloy of aluminum) as a matrix, zircon sand mineral (ZrSiO_4) particles as reinforcement and CaCO_3 as blowing agent. LM13 alloy is selected due to light weight with good corrosion and wear resistance as well as good machinability. On the other hand, high hardness, high modulus of elasticity with good thermal stability is the reasons for the selection of zircon sand as reinforcement. Commercial grade LM13 alloy was purchased from Emmes Metals Private Limited, Mumbai (India) and zircon sand was supplied by Indian Rare Earths Limited, Orissa, India. Fine grade (1-25 μm) CaCO_3 was procured from S D Fine-Chem Limited, Mumbai (India) which is used as blowing agent. Zircon sand (ZrSiO_4) of particle size range (1-32 μm as a fine grade) and (106-125 μm , as a coarse grade) were used as reinforcement. The distribution of particles within the selected size range for zircon sand and blowing agent (CaCO_3) was further analyzed with the help of CAMSIZER XT. The detailed chemical composition of the LM13 alloy and zircon sand is given in Table 3.1 and 3.2 respectively.

Table 3.1: Chemical composition of the LM13 alloy

Elements	Si	Fe	Cu	Mn	Mg	Zn	Ni	Al
Chemical Analysis (Wt.%)	12.0	0.4	1.2	0.4	4.00	0.2	1.0	Bal.

Table 3.2: Chemical composition of zircon sand mineral (ZrSiO_4)

Elements	ZrO_2 (+ HfO_2)	SiO_2	TiO_2	Fe_2O_3
Wt% in Bulk	65.30	32.80	0.27	0.12

3.2 Development of AMCs and AMCFs

3.2.1 Development of AMCs

In this work, we developed AMCs and AMCF by stir casting process. For melting, a resistance furnace was used. The stirrer was attached at the top of the furnace. About 1000 gm of aluminum alloy (LM13) was melted at 750 °C in a graphite crucible of 3 kg capacity. The molten metal was mechanically stirred with graphite stirrer at 630 rpm to create the vortex in the melt. Before charging, zircon sand particles were heated at 400 °C to remove the moisture and other volatile elements. These preheated zircon sand particles were added inside the vortex with the help of funnel kept on top of vortex during the stirring of the molten metal. After the incorporation of the

reinforcement in the matrix, molten mass was further stirred for around 5 minutes to get the uniform distribution of zircon sand particles. Then, the molten mass was poured into cast iron mold of dimension: $12 \times 12 \times 4 \text{ cm}^3$ and allowed to cool in air.

3.2.2 Development of AMFs and AMCFs

Small pieces of LM13 alloy in desired quantity (1000 gm to 1500gm) were taken in a graphite crucible. The crucible was heated in a furnace upto 750°C at a rate of 5°C per minute and held at this for 10 minutes to ensure complete melting. After melting the slag from the top surface of the melt was removed. The melt was rotated with the help of three blade stirrer for 2 minutes. The rotation speed of impeller was increased upto 630 rpm slowly to create a vortex inside the melt. In the vortex different amount of blowing agent (CaCO_3) was charged and mixed properly with the help of stirrer for 3 minutes. After uniform mixing of blowing agent and LM13 alloy melt, the furnace temperature was increased upto 900°C and held for 5 minutes to allow CaCO_3 to decompose. The released gas forms bubbles and generate the LM13 alloy foam. Finally, the crucible was removed from furnace and cooled in air to get foam. For composite foam required amount of zircon sand was added first in the vortex. After rotating the melt for 5 minutes, the required amount of blowing agent (CaCO_3) was added in the vortex again. The entire melt was further rotated for 3 minutes and the temperature was raised to 900°C to facilitate the decomposition of CaCO_3 and held for about 5 minutes. Crucible containing developed LM13 alloy composite foam was removed from the furnace and cooled in air. Table 3.3 shows the processing parameters of developed AMCFs.

Table 3.3: Processing parameters for foaming of LM13 alloy

List of processing parameters for foaming	
Temperature at the time of addition of zircon sand	750°C
Temperature at the time of addition of blowing agent	750°C
Foaming temperature	850°C , 875°C , 900°C
Particle size of zircon sand	$1\text{-}32 \mu\text{m}$ and $106\text{-}125 \mu\text{m}$
Particle size of blowing agent	$1\text{-}25 \mu\text{m}$
No. of blades in stirrer	3
Blade angles of stirrer	45°
RPM of stirrer	Around 630
Height of the stirrer in the melt	$2/3$ inside the melt

Specimens from different parts of the alloy foam as well as composite foam of $12 \times 12 \times 50 \text{ mm}^3$ were cut by diamond cutter. Table 3.4 shows the summary of developed composites with varying

amount and size of zircon sand particles in aluminum alloy matrix. Experimental procedure followed for the development of AMCs and AMFCs is given in the flow charts shown in Fig. 3.1 & 3.2 respectively.

Table 3.4: Composites on basis of reinforcement with different amount and size of zircon sand particles.

Particle size (μm)	Amount of zircon sand particles (wt.%)					
	2.5%	5%	7.5%	10%	12.5%	15%
Fine (1-32 μm)	✓	✓	✓	✓	✓	✓
Coarse (106 -125 μm)	✓	✓	✓	✓	✓	✓

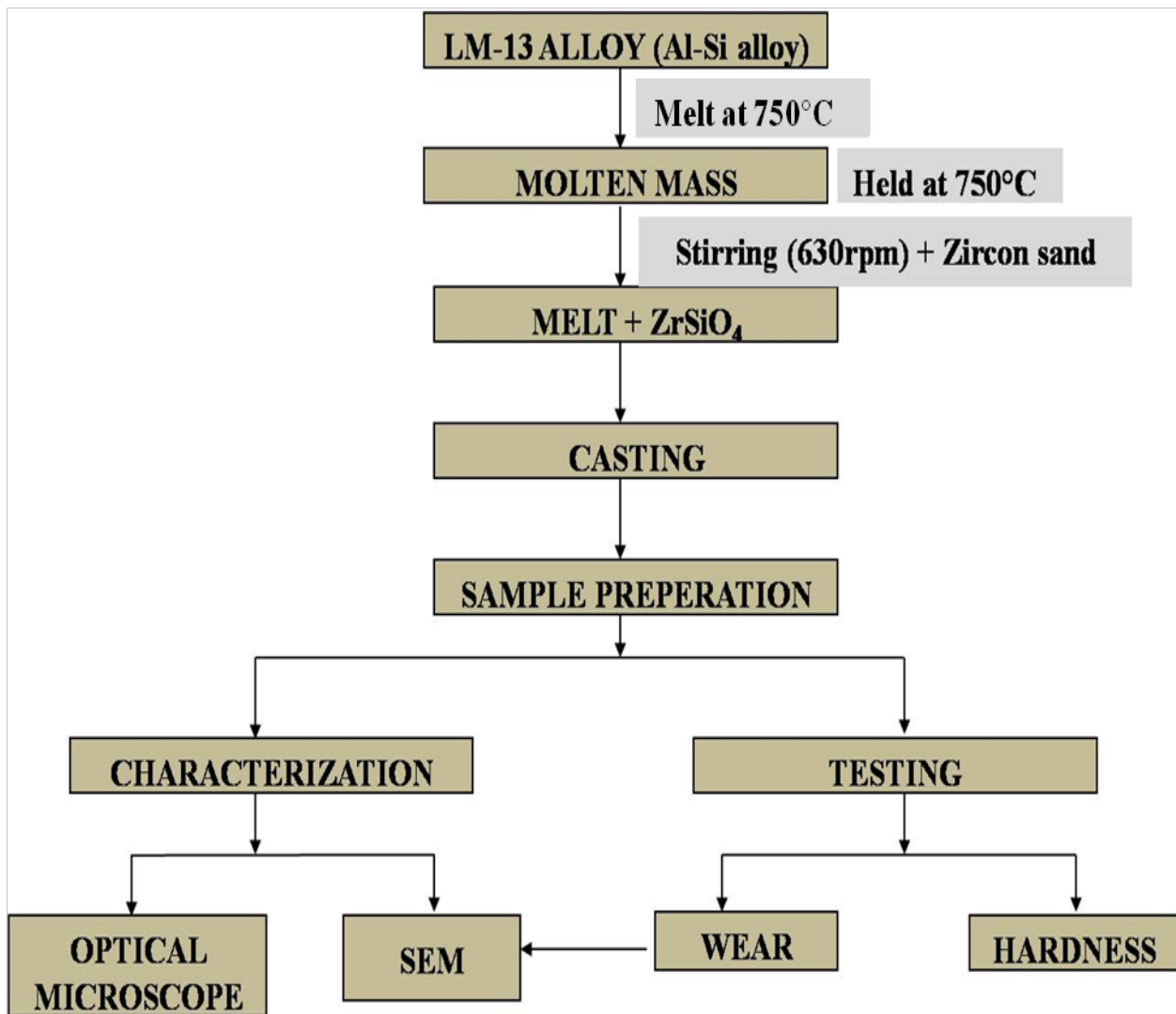


Figure 3.1: Flow chart of the methodology to develop the AMCs.

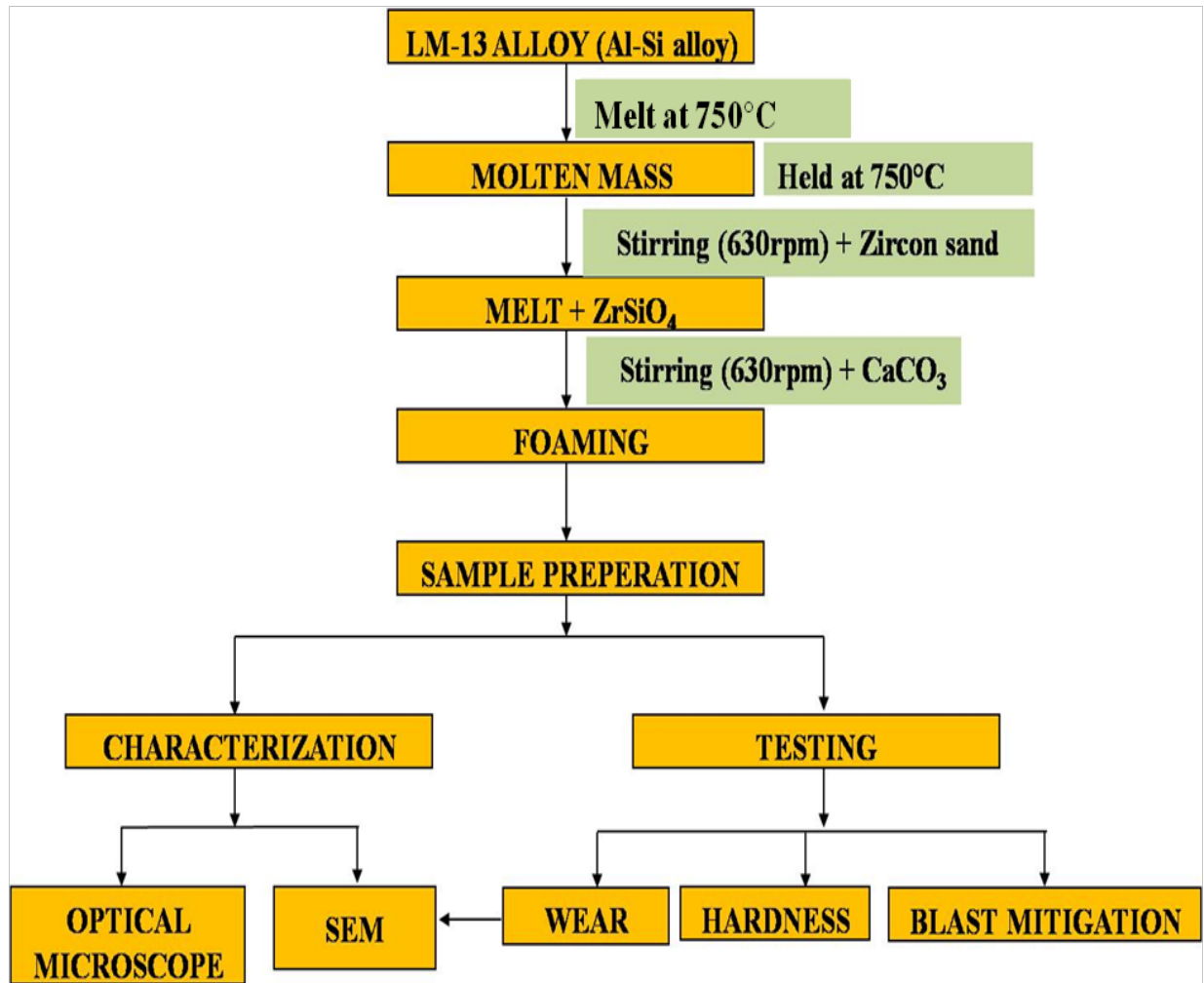


Figure: 3.2: Flow chart for the methodology of AMCF.

3.3 Materials Characterization

Composite and its foam samples were characterized using different techniques. Morphological, microhardness and wear tests were done by using Optical Microscope, Scanning Electron Microscope, Vickers hardness and dry sliding Wear test using pin-on-disc method respectively.

3.3.1. X-Ray Analysis

X-ray diffraction patterns of base alloy (LM13) and composites (LM13/Zr) were recorded by means of Panalytical X'pert PRO MPD, Netherland using Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$). The collected data was matched with reference data for identification of different phases present in the base alloy and composites.

3.3.2. Morphological Analysis

For the morphological analysis of composites and its foam, samples having dimensions 20×20 mm² were cut from the different areas of the cast samples. These samples were mechanically ground and polished followed by etching with Keller's reagent. The chemical composition of Keller's reagent is given in table 3.5. The surface morphology of each sample was studied with the help of optical microscope (Eclipse MA-100, Nikon) and scanning electron microscope (JOEL, JSM-6510LV) at different magnifications. Elemental analysis of the composite at different phases was done with SEM-EDS (Oxford INCA).

Table 3.5: Chemical composition of Keller's reagent.

Composition	Distilled Water	HNO ₃	HCl	HF
Vol. %	95ml	2.5ml	1.5ml	1ml

3.3.3 Hardness

Microhardness of the AMC, AMF and AMCF samples was measured by Vickers hardness test method respectively. Microhardness values at different phases were measured using Vickers hardness testing machine (model: MVK_HO, Mitutoyo, Japan). Each value of hardness is an average of the ten separate values taken from different places of the samples.

3.3.4 Wear Testing

Dry sliding wear test using pin-on-disc method was done to study of wear behavior of the materials. For wear tests a cylindrical pin of 8 mm in diameter was prepared from the cast material of the AMCs and AMCF samples. Tests were performed on wear testing machine (wear and friction monitor, TR-20 CH-400, Ducom Instruments, Bangalore India) under dry sliding conditions in ambient air at room temperature. Wear tests were conducted at different loads (9.8 N, 19.6 N, 29.4 N, 39.2 N and 49 N). All the samples of AMCs and AMCF were tested against EN32 steel disc having 65 HRC hardness. To get an average value of wear rate, each test was run three times. Before each test the track was cleaned with acetone. For each test a new track was used to get similar test conditions. Throughout the experiment a constant sliding velocity of 1.6 m s^{-1} was maintained. Sliding distance covered during the experiment was about 3000 meters. Wear rate was determined by measuring specimen height change using a linear variable displacement transducer (LVDT). In the entire study we have kept the worn out length of pin constant (2 mm) to have relative data. To study the wear behavior, wear rate was calculated by using the formula,

$[W \text{ (mm}^3\text{/m)} = \text{height change (mm)} \times \text{pin area (mm}^2\text{)} / \text{sliding distance (m)}]$. Cylindrical shaped pins with flat end were used as samples for wear testing. Test pins were ground to a smooth finish on 600 grit SiC paper. All the wear tests were conducted at room temperature. The worn surface regions (wear tracks) and collected debris after the dry sliding wear tests were also examined under scanning electron microscope.



Figure 3.3: Images of pin on disc setup for wear test (Ducom-TR-20CH-400).

3.3.5 Blast Testing for AMCFs

A special fixture was used for conducting the instrumented explosive trials on aluminum foam samples. The photograph of the trial setup and schematic are shown in Fig. 3.4 & Fig. 3.5 respectively. For blast test disc samples of 12 cm diameter and nearly 8 cm thickness were used in all tests.

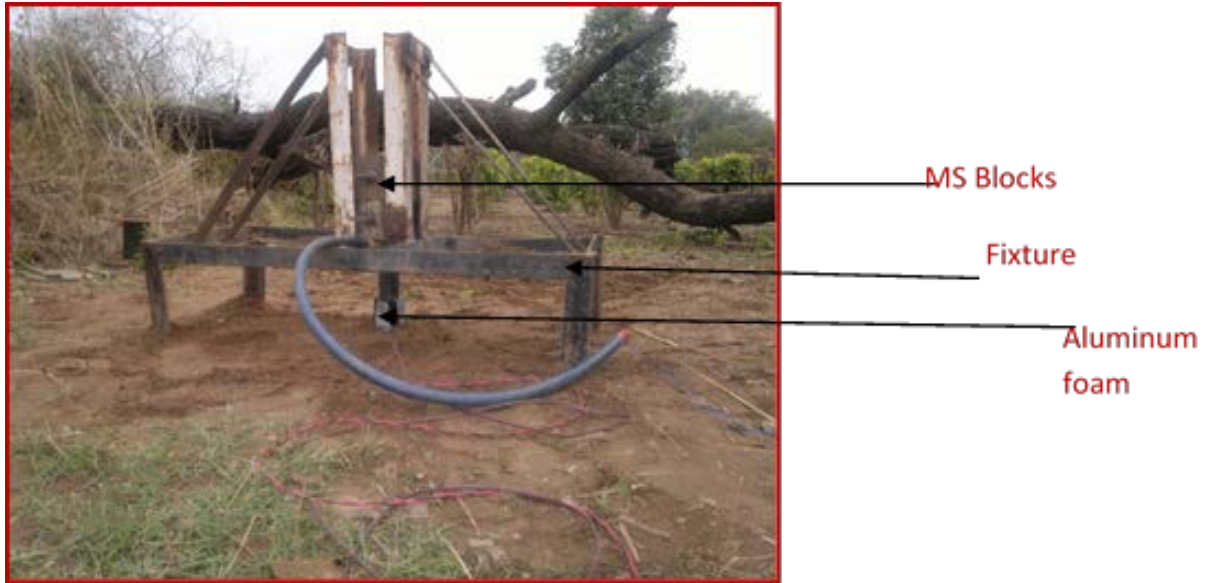


Figure 3.4: Photograph of the trial setup for blast testing of AMCF samples.

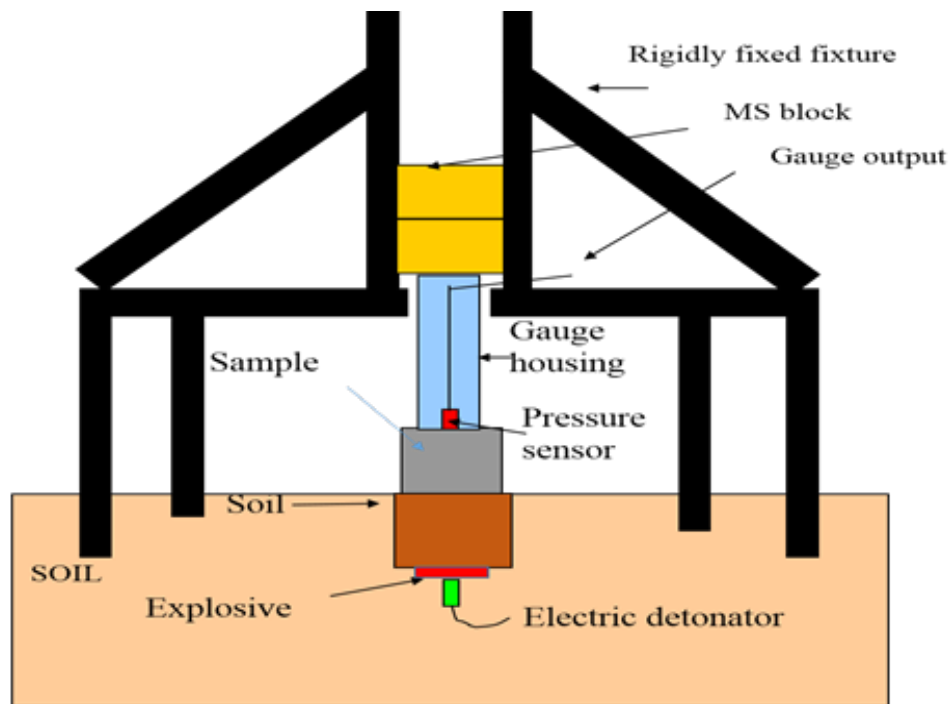


Figure 3.5: Schematic diagram of the trial setup for blast testing of AMCF samples.

Pressure sensors are used in research and development or in test laboratories. The generic pressure sensors, Kistler offers a vast range of customized solutions for pressure measurement in specific applications. Piezoelectric pressure sensors are suitable for measuring highly dynamic, dynamic

and quasi-static pressure curves or pulsations. A Kistler make shock sensor having range 10 kBar with sensitivity 1.16 pc/ Bar fixed in a mild steel housing was used for measuring transmitted pressure through the aluminized samples. The photograph of sensor is shown in Fig. 3.6



Figure 3.6: Kistler make sensor for pressure measurement in blast mitigation.

The output of sensor is conditioned by associated signal conditioner and brought to instrumentation shelter using specially designed low noise cable. The data was recorded on high speed Tektronix make oscilloscope. The trials were conducted using CE pellet having 35g of tetryl charge. The CE pellet was detonated using electric detonator no. 33. A soil over burden of 8cm was used in these trials. All blast mitigation tests were conducted at Terminal Ballistics Research Laboratory (TBRL), Defence Research and Development Organisation (DRDO), Located in Chandigarh, India.

CHAPTER 4

RESULTS AND DISCUSSION

(Microstructure, Microhardness and Wear Behavior Analysis)

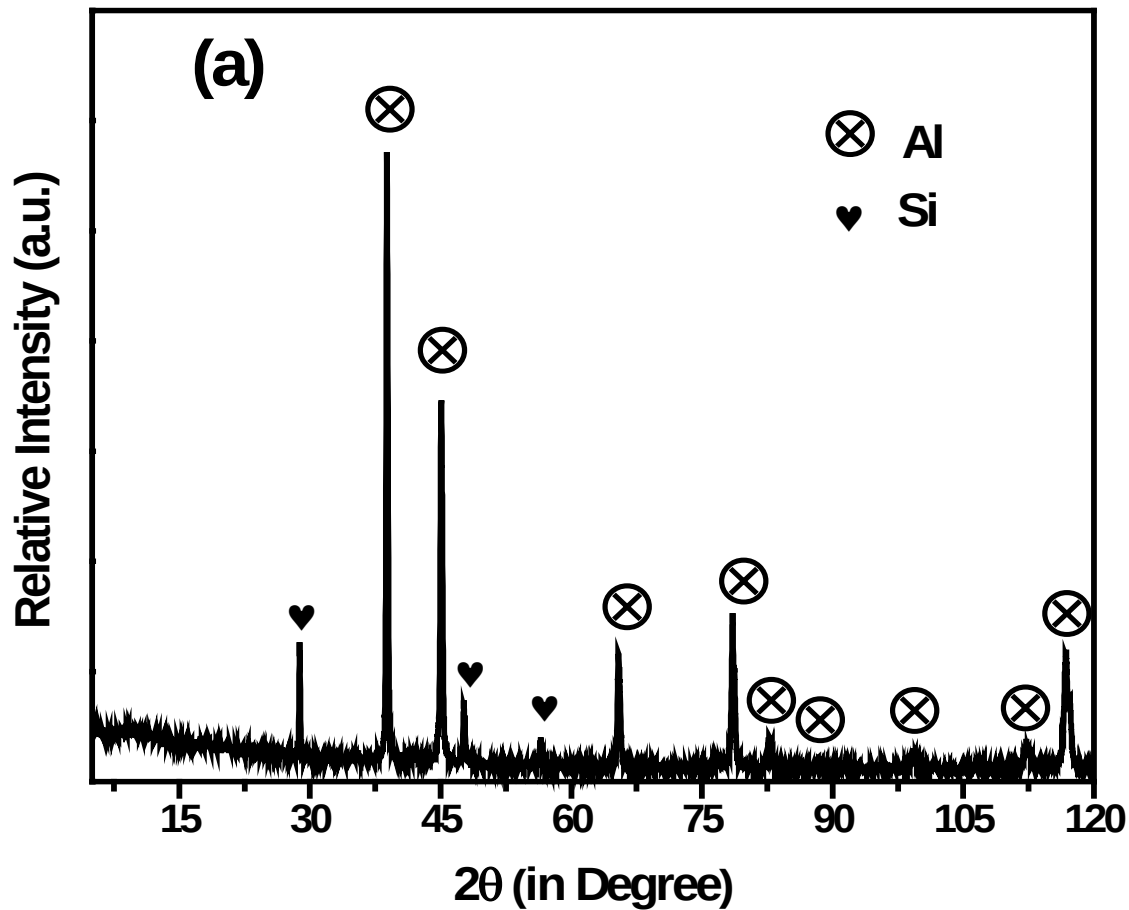
Overview:

In this section of results and discussions, the microstructure, microhardness and wear behavior of the developed AMCs, AMFs and AMCFs has been presented. The structural analysis of the developed AMCs, AMFs and AMCFs with reinforcement of different size zircon sand particles in the alloy matrix at different conditions has been done critically. Al-Si (LM13) alloy was used as the matrix material for the composites. Composites with reinforcement in between 2.5 to 15wt.% (average particle size range fine (1-32 μ m) and coarse size (106-125 μ m) were prepared. Optical microscopy and SEM were used to examine the distribution of reinforced particles in the matrix of AMCs and AMCFs. The results of microhardness and wear tests obtained from cast AMCs, AMFs and AMCFs is discussed in this chapter. The particle bonding with matrix was tested by measuring hardness at the interface. The results show that wear characteristics of the AMC-¹⁵C_{fine}, AMF-²F_{900 °C} and AMCF-¹⁵C_{fine} is better than other developed AMCs, AMFs and AMCFs. The operative wear mechanism has been established by analyzing the wear track and wear debris under scanning electron microscope.

As described in the previous chapter, LM13 alloy was used as matrix material, with zircon sand as reinforcement for AMC. Further, CaCO_3 were added as blowing agent for the development of AMFs and AMCFs. This section presents the systematic discussion about macro and micro-structural analysis of AMCs, AMFs and AMCFs respectively.

4.1 X-Ray Diffraction Analysis

The X-ray diffraction (XRD) patterns of base alloy (LM13) and composite ($\text{AMC-}^{15}\text{C}_{\text{fine}}$) are shown in Fig. 4.1(a &b), respectively. Figure 4.1a shows the presence of Al and Si in base alloy. The presence of zircon sand particles (ZrSiO_4), along with Al and Si elements is observed in the XRD pattern of composite (Fig. 4.1b). **Apart from these phases, Al_2SiO_5 (sillimanite) was also observed in the composite which could have been formed at the alloy-particle interface due to the reaction of zircon sand and LM13 base alloy during casting.**



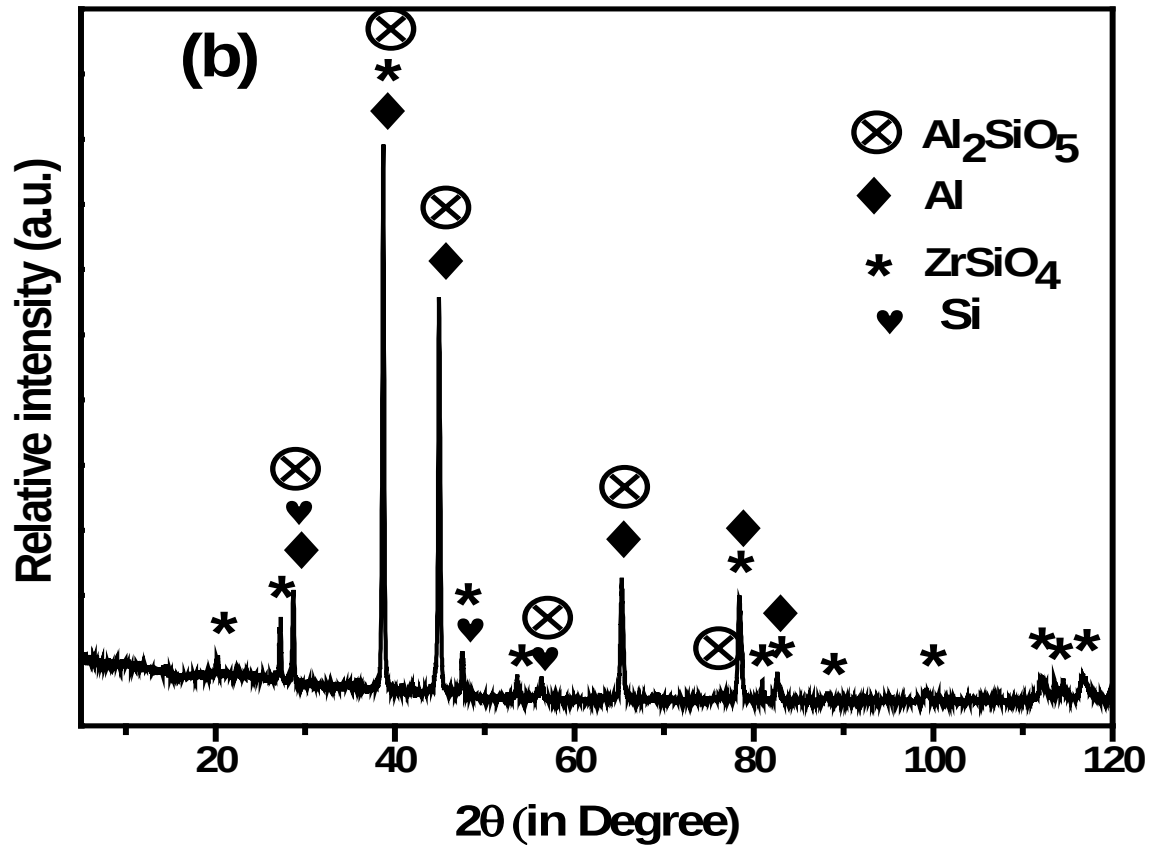


Figure 4.1: XRD patterns of (a) LM13 alloy and (b) AMC-¹⁵C_{fine}

4.2 Microstructure Analysis of AMCs

Figure 4.2 (a-b) shows the optical micrographs of the LM13 alloy at low (100X) and high (500X) magnifications respectively. The dendritic growth of primary α -Al phase along with eutectic phase in between the dendritic region (Fig. 4.2a) has been observed in LM13 base alloy which is more pronounced at higher magnification as shown in Fig. 4.2b. Presences of high silicon content in the LM13 alloy increases the corrosion and wear resistance and also improves casting and machining characteristics of the alloy [1]. When Al-Si alloy solidifies, the primary aluminum forms and grows as dendrites, whereas silicon solidifies as needle like structure. The Al-Si alloy usually has some other coexisting elements viz. copper, magnesium, manganese, zinc and iron which can form intermetallic phases during solidification. Size and volume fraction of the reinforced particles both influence the structure of composite to greater extent. Figures 4.3- 4.8 present the micrographs of composites reinforced with 2.5, 5, 7.5, 10, 12.5 and 15wt.% of zircon sand as particulates in the LM13 alloy matrix respectively. In the present chapter, the results of composites reinforced with fine particles (1-32 μm) and coarse (106-125 μm) are presented.

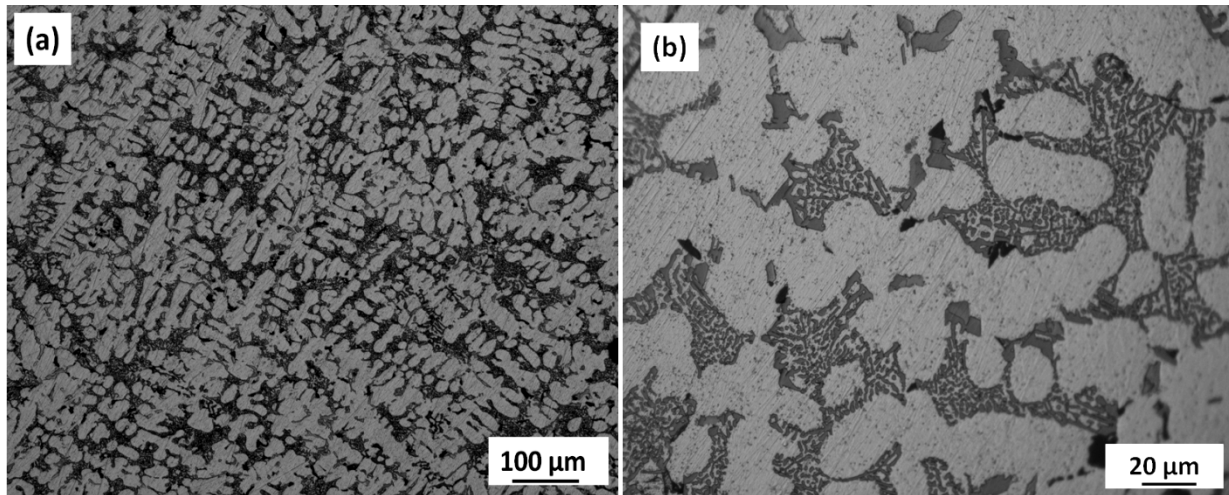


Figure 4.2: Optical micrographs of the LM13 alloy (a) showing dendritic structure and (b) showing inter dendritic region.

Figure 4.3(a-b) presents a fairly uniform distribution of reinforced particles in alloy matrix. Figure 4.3a presents micrographs of AMC-^{2.5}C_{fine}. The micrograph reveals nearly uniform distribution of reinforced particles in alloy matrix of composite. Homogeneous distribution of particles is achieved by the constant stirring action of the impeller which provides the normal shear strain and delays the particle settling tendency during stirring [1]. The change in surface morphology can be explained on the basis of the fact that during solidification ZrSiO₄ particles are pushed by the solid liquid interface of the melt which results the particles to occupy the space between the inter-dendritic regions.

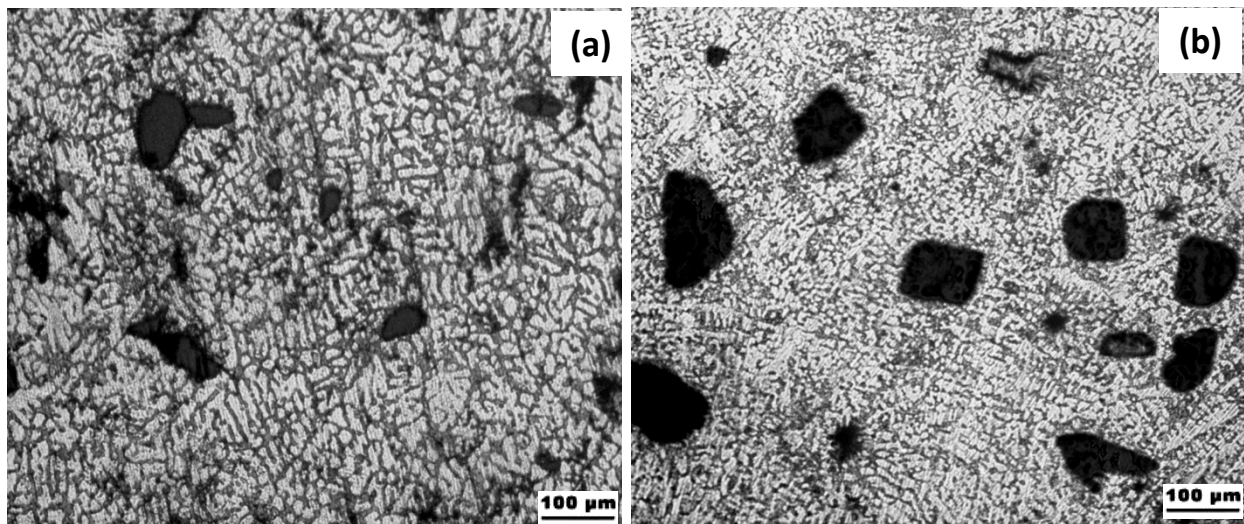


Figure 4.3: The optical micrographs of composite containing 2.5wt.% zircon sand (a) fine, and (b) coarse particles.

The acicular eutectic morphology of silicon is observed in the vicinity of the particles. The particles offer preferential sites for the effective nucleation of eutectic silicon due to difference in thermal conductivity of reinforced particles and the nucleated α -Al matrix. Delay in cooling in the vicinity of the particles accelerates the nucleation of α -Al away from the particles. The latent heat liberated during nucleation of α -Al phase rises the melt temperature. The growth of α -Al leads to the enrichment of Si in the remaining melt zone surrounding the particles.

Figure 4.3b presents the micrographs of 2.5wt.% coarse particles dispersed in the matrix. Coarse particles are more uniformly distributed as compared to the fine particles because large size particles experience high shear force during stirring. The dendritic morphology changes to cellular as reinforced particles restrict the solute transport processes by diffusion and flow [2-4]. The long dendritic growth is clearly observed in the particle depleted regions. Some fragmented dendrites and small broken particles can also be seen due to the stirring action of the impeller during mixing. Figure 4.4 (a-b) shows that the addition of 5wt.% ZrSiO_4 particles of fine and coarse size has further refined the microstructure of the composite. Figure 4.4a shows nearly homogeneous distribution of fine size ZrSiO_4 particles achieved in $\text{AMC-}^5\text{C}_{\text{fine}}$.

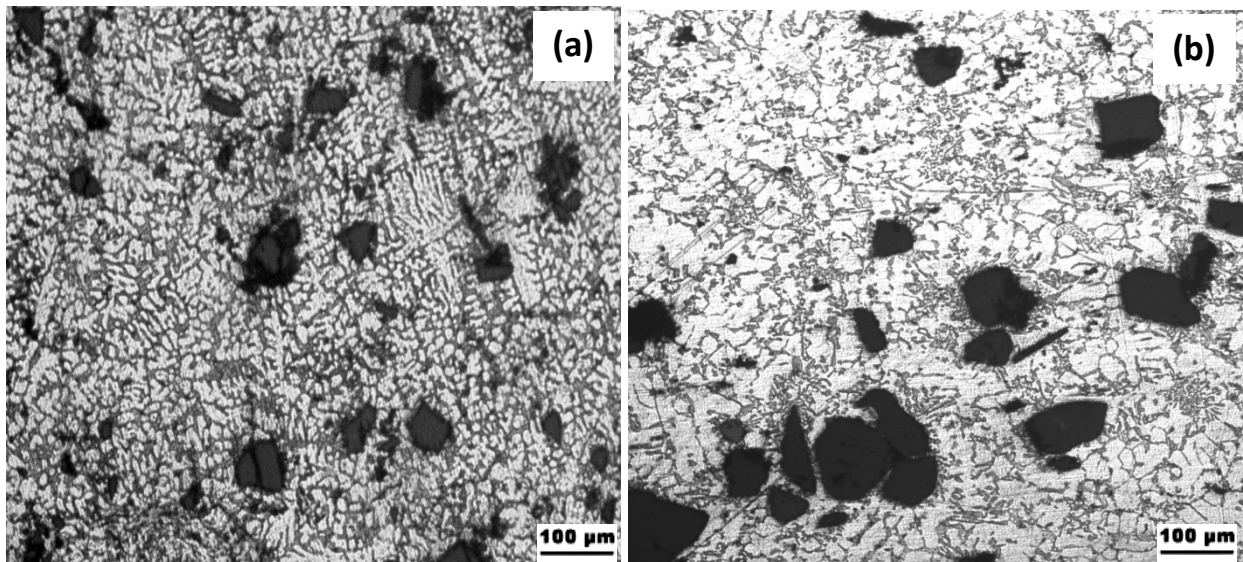


Figure 4.4: The optical micrographs of composite containing 5wt.% zircon sand (a) fine, and (b) coarse particles.

The surface morphology of $\text{AMC-}^5\text{C}_{\text{fine}}$ is changed by the presence of particles in matrix (Fig. 4.4a). The heterogeneous nucleation of silicon in the vicinity of the reinforced particles leads to a change in silicon morphology from needle shape to non-acicular. **This kind of globular or blunted type of Si morphology enhances the wear resistance and provides better mechanical**

properties [2]. Due to convection effect agglomeration of particles at certain places is seen as fine particles are pushed at faster rate during stirring.

Figure 4.4b shows that 5wt.% coarse size ZrSiO_4 particles are uniformly dispersed in the matrix than the fine particles because large size particles experience higher shear force during stirring. The dendritic morphology changes due to presence of ZrSiO_4 particles. During particle addition, local solidification of the melt occurs which is induced by the particles as there is a temperature difference between the particle and the melt. It was also found that the perturbation in the solute field due to the presence of particles can change the dendrite tip radius and the dendrite tip temperature. These effects give rise to a dendrite-cell transition as the density of particles is increased. Also the length of the dendrite is reduced [3]. The enrichment of Si phase is observed in the vicinity of the particles.

It can be noted from Fig. 4.5 (a-b) that addition of 7.5wt.% ZrSiO_4 particles of fine and coarse size has further refined the microstructure of the $\text{AMC-}^{7.5}\text{C}_{\text{fine}}$ and $\text{AMC-}^{7.5}\text{C}_{\text{coarse}}$. The floral pattern of the dendrites can be observed in some regions of the micrograph in the $\text{AMC-}^{7.5}\text{C}_{\text{fine}}$ as shown in the Fig. 4.5(a).

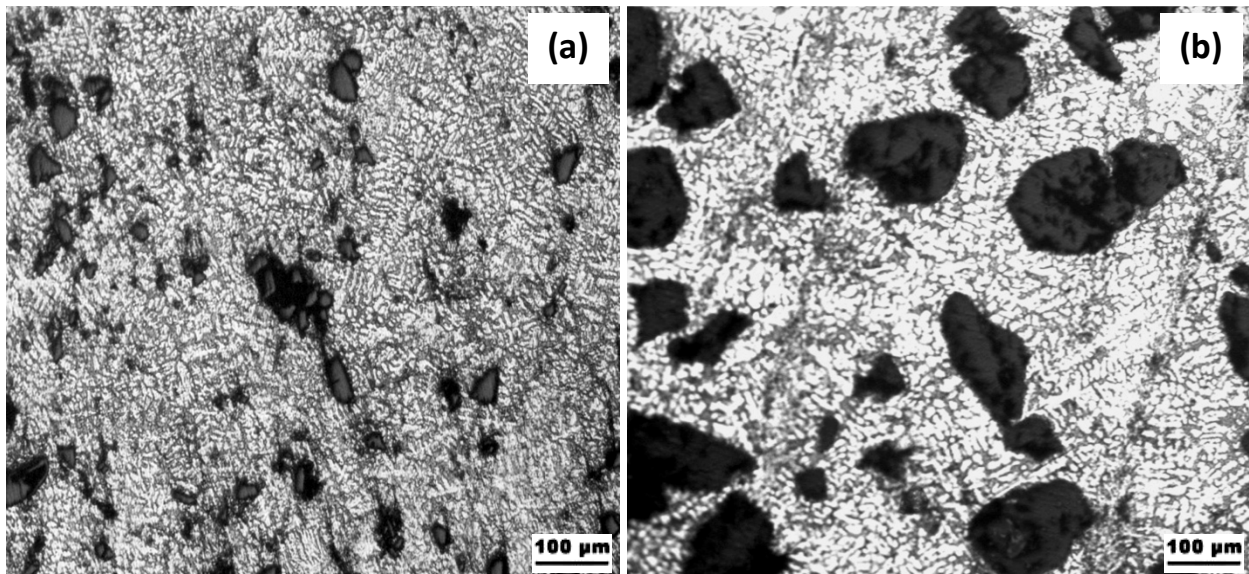


Figure 4.5: The optical micrographs of composite containing 7.5wt.% zircon sand (a) fine, and (b) coarse particles.

Accumulation of refined Si phase in the form of colonies can be seen around the particles. Some ZrSiO_4 particles are trapped in the inter-dendritic regions. Overall, the ZrSiO_4 particles are well dispersed in the matrix phase. The uniform distribution of ZrSiO_4 particles in the matrix of $\text{AMC-}^{7.5}\text{C}_{\text{coarse}}$ can be observed from Fig. 4.5b. Large dendrites are grown due to the increase in inter-

particle distance as compared to the fine particles. The termination of the dendritic growth on approaching the particle reveals that the presence of particles causes the hindrance to the dendritic growth. The restricted dendritic growth enhances the strength thus provides more hardness to the composite [3]. Even the fragmentation of dendrites during rotation gives rise to the growth of cellular structure. In certain areas, the segregation of the particles leads to the clustering. These overlapped segregated particles are loosely bonded to the matrix thus reduces its strength.

It can be noted from Fig. 4.6 (a-b) that the addition of 10 wt.% ZrSiO_4 particles of fine and coarse size has further refined the microstructure of the composites. Figure 4.6 shows the agglomeration of fine size particles in the matrix of $\text{AMC-}^{10}\text{C}_{\text{fine}}$.

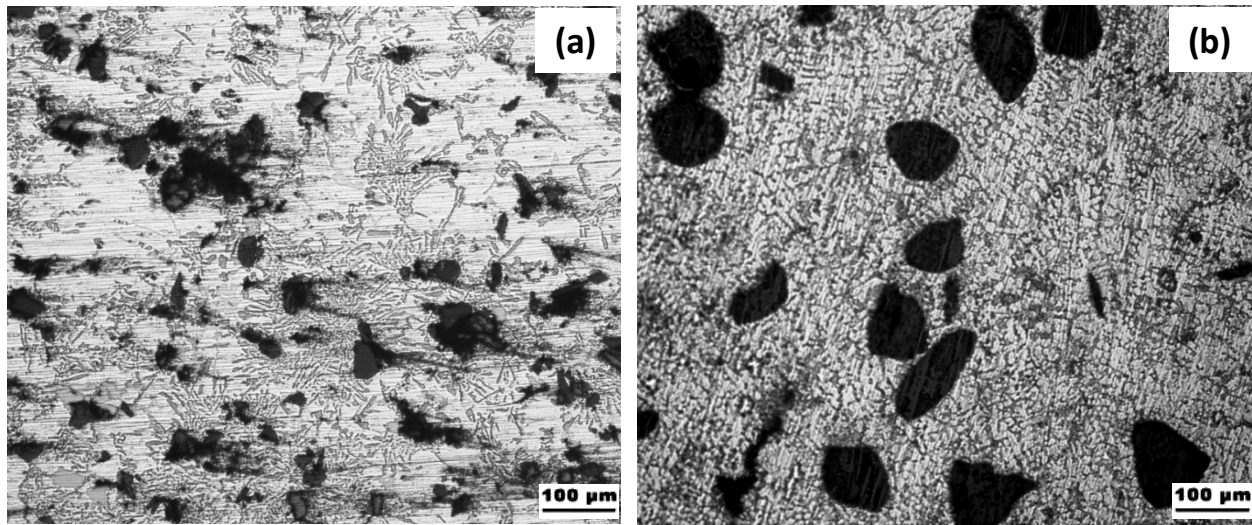


Figure 4.6: The optical micrographs of composite containing 10wt.% zircon sand (a) fine, and (b) coarse particles.

The nucleation of Si in the nearness of ZrSiO_4 particles leads to a change the morphology of silicon such as needle shaped to non-acicular in the matrix of $\text{AMC-}^{10}\text{C}_{\text{fine}}$. This kind of bulbous or blunted type of Si morphology with different tip structures enhances the wear resistance due to higher contact area and provides better mechanical properties. In the Figure 4.6b the primary $\alpha\text{-Al}$ dendrites growth increased due to increase of Si phase in the vicinity of the particles. The change in silicon morphology is more pronounced in the composite with coarse particles as compared to fine ZrSiO_4 reinforced particles. Delayed cooling of the large size ZrSiO_4 particles accelerates the modification of silicon phase. Even some Si particles are observed to be arrested in the dendritic regions during solidification. The optical micrographs of $\text{AMC-}^{12.5}\text{C}_{\text{fine}}$ and $\text{AMC-}^{12.5}\text{C}_{\text{coarse}}$ are shown in Fig. 4.7 (a-b) respectively. The uniform distribution of ZrSiO_4 reinforced particles with

some agglomeration in the matrix of $\text{AMC-}^{12.5}\text{C}_{\text{fine}}$ can be noted from the Fig. 4.7a. Addition of ZrSiO_4 particles increases the viscosity of the melt which reduces the degree of convection in molten composite hence slows down the solute transport process by diffusion and flow. Reinforced particles affect the thermal field (which is the mismatch of matrix and the particle) and solute field (by convection interrupting diffusion of solute silicon). Delayed rate near the particles and restricted diffusion within the melt changes the silicon morphology (such as leaves of the white spruce change into blunted type) which provides better strength to the material. This morphological transformation to the localized rapid cooling effect is produced by zircon sand particles due to large temperature difference in the melt around its vicinity.

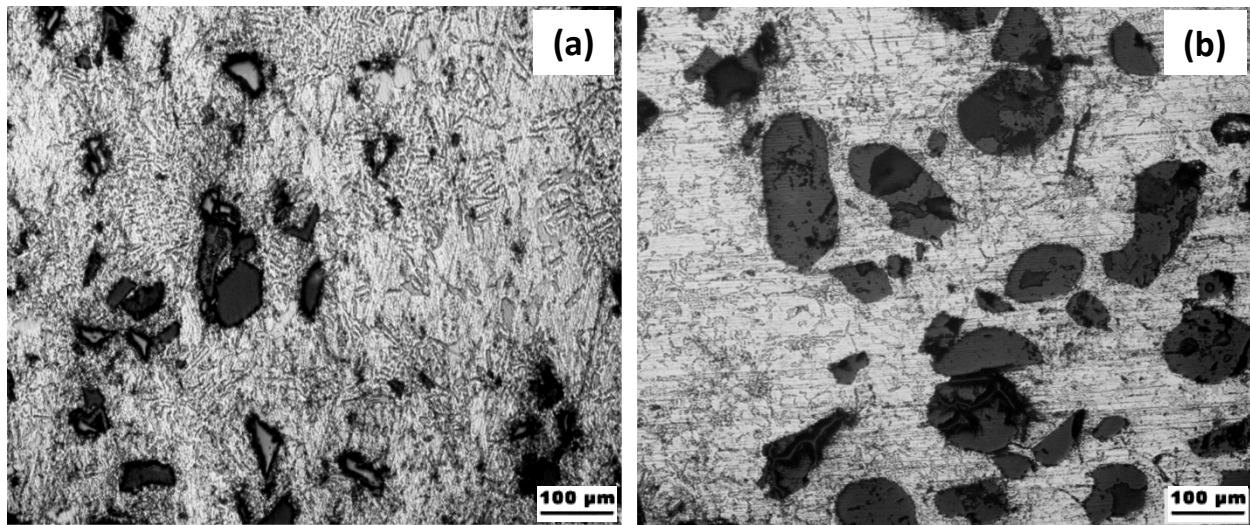


Figure 4.7: The optical micrographs of composite containing 12.5wt.% zircon sand (a) fine, and (b) coarse particles.

Coarse particle exhibits better distribution of particles in the matrix of $\text{AMC-}^{12.5}\text{C}_{\text{coarse}}$ as agglomeration of particles is not observed as shown in Fig 4.7b. However, dendritic growth can be seen in the particle depleted regions. Wettability (contact angle: 145°C) between the molten alloys with the ZrSiO_4 particles also plays an instrumental role in achieving the uniformity in distribution of particles [5]. During rotation the fragmentation of dendrites takes place leading to growth of cellular structure. Particle pushing and engulfing phenomenon during solidification is also correlated with the mutual wetting behavior among the solid, liquid, and particle phases. Figure 4.8 (a-b) shows the microstructure of $\text{AMC-}^{15}\text{C}_{\text{fine}}$ and $\text{AMC-}^{15}\text{C}_{\text{coarse}}$. The random distribution of the fine size particles can be seen in the LM13 alloy matrix. The occupancy of ZrSiO_4 particles in the eutectic zone provides more strength to the composite.

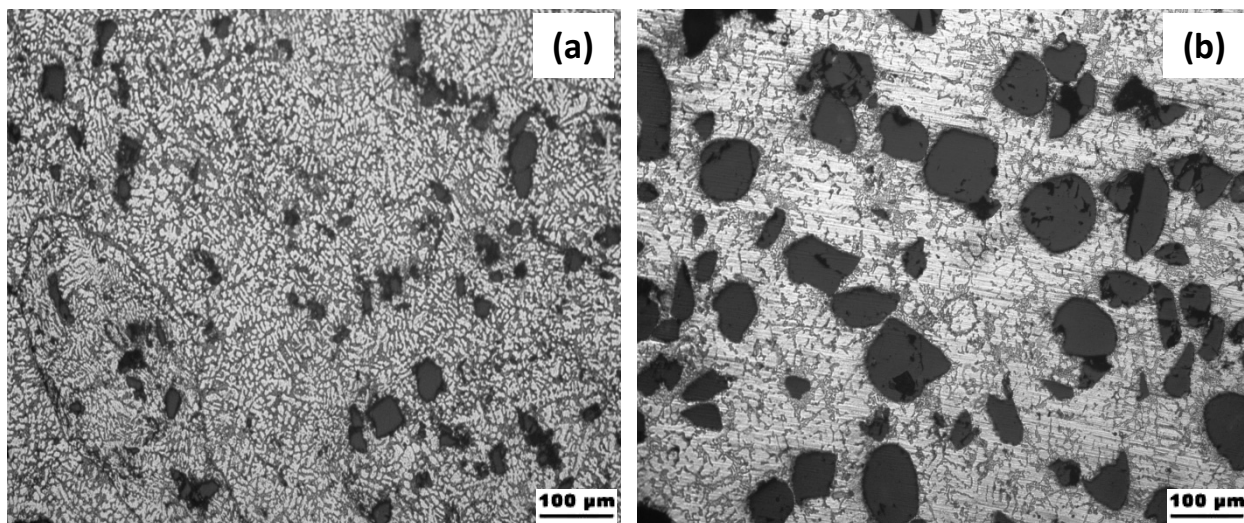


Figure 4.8: The optical micrographs of composite containing 15wt.% zircon sand (a) fine, and (b) coarse particles.

The thermal mismatch between the melt and the particle causes perturbation in the solute field due to the change in the dendritic tip shape and temperature. This effect gives rise to the dendritic cell transition [6]. The increase of ZrSiO_4 particles in the melt leads to the formation of small size dendrites in between the particle spacing due to the dendritic-cell transition. Homogeneous distribution of the particles with the addition of 15wt.% of coarse size ZrSiO_4 particles is shown in Fig. 4.8b. This can be attributed to the delayed solidification rate in the presence of increased number of coarse size particles which increases the dendritic arm spacing than the average size of the particles. No agglomeration, clustering and segregation of the ZrSiO_4 particles are seen. A network of silicon colonies is not that prominent as these refined silicon particles are trapped in the dendritic regions. Some black spots are seen on the coarse particles containing composite. This void space is created due to removal of the fine particles from the surface during the preparation and polishing of the sample. Even some small size fragmented particles are visible due to the fracturing during stirring. The limited dendritic growth due to the presence of ZrSiO_4 reinforced particles and dendritic fragmentation during stirring is clearly demonstrated in the micrograph. However, agglomeration of particles is also observed which is visible at certain places in Fig. (4.5a-4.8a). The possibility of agglomeration of particles is increased with increase in the amount of ZrSiO_4 particles.

From the studies of the micrographs of the base alloy and the composites it can be said that the addition of ZrSiO_4 ceramic particles acts as a barrier for the dendritic growth. **During solidification process, $\alpha\text{-Al}$ and Si phases are nucleated. Once the nucleation followed by**

growth starts, the melt temperature rises because of evolution of latent heat during nucleation of α -Al phase as alloy is hypoeutectic. Since, in the beginning both melt and reinforced particles have same temperature but after nucleation melt temperature rises and reinforced particles act as heat sink which facilitate the heat flow from molten mass towards the reinforced particles. Si being second phase finds its site for nucleation and is nucleated near the interface. Because of the fact that heat flow towards reinforced particles occurs from all directions so nucleated Si does not grow considerably and hence, we get refined Si structure. Thermal conductivity mismatch between the particle and matrix leads to the refinement of Si eutectic phase. Dendritic fragmentation takes place due to the high strain induced during stirring. The modification in the morphology is dependent on Si refinement, the dendritic growth and fragmentation during solidification [6]. The composite reinforced with fine particles have exhibited better morphology as compared to coarse particle reinforced composite. The increased viscosity of the melt with 15wt.% fine ZrSiO_4 particles reduces the degree of convection in the melt which promotes a small cell diameter leading to refined matrix structure. Reinforcement of ZrSiO_4 particles in the matrix provide hindrance to the dendritic growth and strengthen the composite by giving more hardness which may also improve the wear rate.

4.2.1 Effect of zircon content (with the same size) on the microstructure of the AMCs

Figures 4.3- 4.8 shows the micrographs of composites reinforced with 2.5, 5, 7.5, 10, 12.5 and 15wt.% of zircon sand with fine size particles (1-32 μm) (part 'a' of each figure) in the LM13 alloy matrix respectively. It indicates a good interfacial bonding between the particle and matrix along with cellular structure of primary α - aluminum. Figure 4.3a shows the micrograph of the composite where fragmented dendrites in the alloy matrix can be seen, though limited dendritic growth in the particle depleted region is also visible. This growth has occurred due to clustering of zircon sand. Fine size zircon sand particles are pushed or engulfed by advancing solid- liquid interface creating sufficient space inside the matrix, which leads to growth of dendrite [6]. Dendritic structure gets modified during casting, which is influenced by many factors such as dendrite fragmentation, restriction of dendritic growth by the particles. Homogeneous distribution of the particles with the addition of 2.5wt.% of fine size ZrSiO_4 particles is shown in Fig. 4.3a. The dendrite size is determined by the interplay or balance between the surface energy and temperature in the melt at interface. The amount of released latent heat at the solid-interface depends of amount on reinforced particles in composite. So, the amount of released latent heat is less at the interface of

LM13/2.5wt.% ZrSiO₄ as compared to LM13/15wt.% ZrSiO₄ for the temperature remains constant until the melt undergoes complete solidification. The dendrite size is large of LM13/2.5wt.% ZrSiO₄ composite as compared to LM13/15wt.% ZrSiO₄ composite. The amount of reinforcement is decides the nucleation and growth of dendrite and it influences the size of dendrite. Higher amounts of reinforced particle (15wt.% ZrSiO₄) gives the more chance of nucleation and thus reduces the size of the dendrites and inter dendrite region and lead to small grains.

4.2.2 Effect of zircon size (at the same content) on the microstructure of the AMCs

Figures (4.3- 4.8) present the micrographs of composites reinforced with 2.5 to 15wt.% of zircon sand with fine particles (1-32 μm) in the LM13 alloy matrix respectively. Some ZrSiO₄ particles are trapped in the inter-dendritic regions so the microstructure (Fig. 4.3a) of LM13/2.5wt.% ZrSiO₄ composite show the larger inter-dendritic regions as compared to fine size ZrSiO₄ particles. Larger size dendrites are grown due to the increase in inter-particle distance in the matrix as compared to the fine particles. The small inter particle distance in the matrix reduced the size of primary α -Al dendrite and grain size in inter dendrite region. Hence, fine equiaxed grain structure developed due to presence of fine size particles leads to several benefits, such as uniform distribution of second phases and micro-porosity, improved feeding ability [3], high yield strength, high wear resistance, improved machinability and excellent deep drawability of the products [4].

4.3 Microstructure Analysis of AMFs

4.3.1 Effect of foaming temperature on the cell size and density of AMFs

Aluminium foams have been developed at different temperatures. In the present work, the effect of holding temperature on cell size and their distribution have been investigated. It is seen that foaming temperature has a greater impact on the cell morphology of the AMF. With the increase in foaming temperature from 850 °C to 900 °C, the cell diameter varies and the distribution of cells observed is nearly homogeneous. However, there are few cells which collapse and form irregular spherical cells in the AMF. Some of the theoretical aspects related to this work is discussed below. **Figure 4.9a shows the TGA of CaCO₃ powder (1-25 μm) used in the present investigation. As the temperature increases between 600 °C to 800 °C, rapid weight change occur. The volatile material in the sample attempt to escape as decomposition begin to take place. It is clear from TGA graph of CaCO₃ that the blowing agent released more gas (Fig. 4.9a) with the increase in foaming temperature and results to an increase in the bubbles size.**

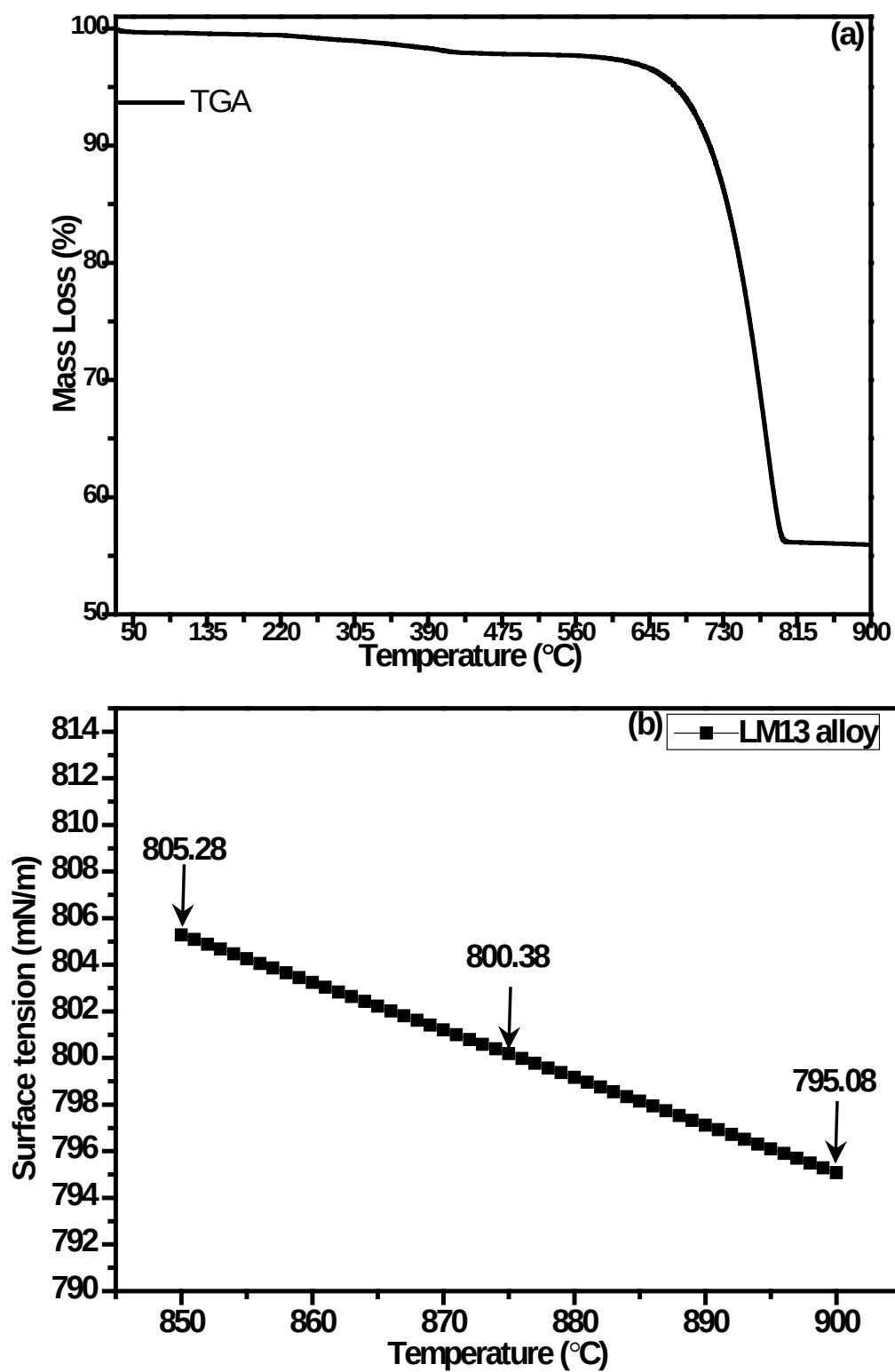


Figure 4.9: (a) TGA of CaCO_3 showing the decomposition of the powder sample and (b) effect of foaming temperature on surface tension of the LM13 alloy foams.

On the other hand, the pressure inside the bubble (P_{in}) increased with the increase in foaming temperature. It results in decreasing the additional pressure (P_c) which is caused by a decrease in surface tension (γ) of the liquid alloy so that gas bubbles can easily nucleate and grow. The variation in surface tension with temperature for liquid LM13 alloy has been calculated as [7]:

$$\gamma = 842 - 0.204 (T - T_m) \quad (4.1)$$

Where γ , T and T_m is the surface tension, foaming temperature, melting temperature of LM13 alloy respectively. This variation is shown in Fig. 4.9b. Similarly, viscosity of LM13 alloy melt with temperature was calculated using the Arrhenius relationship [7]:

$$\eta_L(T) = C \exp(E_a / RT) \quad (4.2)$$

Where η_L is the viscosity of the melt at temperature T , C is a coefficient factor determined by the kind of alloy. The activation energy of viscous flow is E_a , and R is the gas constant.

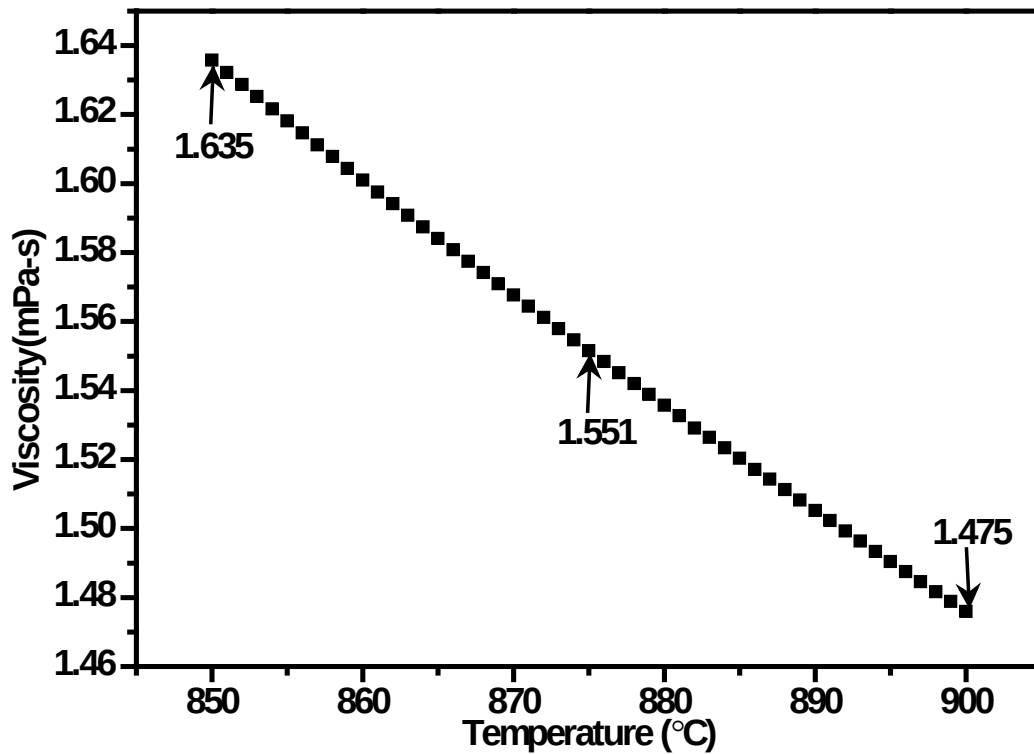


Figure 4.10: Effect of foaming temperature on the viscosity of the LM13 alloy foams.

The variation in viscosity corresponding to temperature is shown in Fig. 4.10. Addition of blowing agent in the molten LM13 alloy at the specific temperature causes the release of CO_2 gas and bubble nucleation in the liquid phase as a result of $CaCO_3$ decomposition. However, with the raising foaming temperature from 850 to 900 °C, the rate of decomposition $CaCO_3$ is increased

and nucleation of gas bubbles increased. The concentration of gas bubbles in the melt liquid is increased and the pressure inside the bubble is enhanced. In this case, because of the lower viscosity of molten aluminum at higher temperatures, the applied force of the trapped gas inside the cell will overcome the viscosity force [8]. This reveals a wide variation in cell structure at different foaming temperatures. As a result, the density, average cell size, cell wall thickness and average ligament length of AMF changed as the foaming temperature was increased. The amount of blowing agent also plays an important role in deciding the cell size of LM13 alloy foam. One way of foaming the melt is to add the blowing agent directly to the melt instead of injecting gas into it. Heat causes the blowing agent to decompose and release gases, which then propels the foaming process [9]. After the addition of blowing agent into the melt, it starts expanding slowly and gradually fills the foaming vessel. The foaming takes place at constant pressure. The decomposition reaction of CaCO_3 takes place on a definite boundary between the molten aluminum. This boundary moves towards the center of the compact and remains spherical in shape. The rate of the decomposition reaction of CaCO_3 is controlled by the transfer of heat (molten temperature LM13 alloy) to this reaction boundary. After cooling the vessel below the melting point of the alloy, the liquid foam turns into solid aluminum foam and can be taken out of the mold for further investigation.

In the liquid LM13 alloy, four different types of forces act on a gas bubble. The internal gas pressure (P_{in}) in the liquid LM13 alloy is responsible for increasing the size of the gas bubble which is shown in Fig.4.11a. At the same time metallostatic pressure (P_m), the atmospheric pressure (P_a) of the system, and the surface tension forces (P_{st}) are responsible for decreasing the size of gas bubbles at this equilibrium condition [10]:

$$P_{in} = P_m + P_a + P_{st} \quad (4.3)$$

The cell size of LM13 alloy foams is obtained by

$$r = 2 \gamma / P_{st} \quad (4.4)$$

where 'r' is the radius of the bubble, γ and P_{st} are the surface tension and the surface tension force, respectively. A general concept in favor of surface contaminants, such as the cell size of alloy foam is increased due to decrease in surface tension of the molten alloy with increase in foaming temperature, which is shown in Fig. 4.11b. It is possible that carbon dioxide gas interactions with liquid alloy may be responsible for the increment in the porosity when the addition of blowing agent in liquid alloy is done [11].

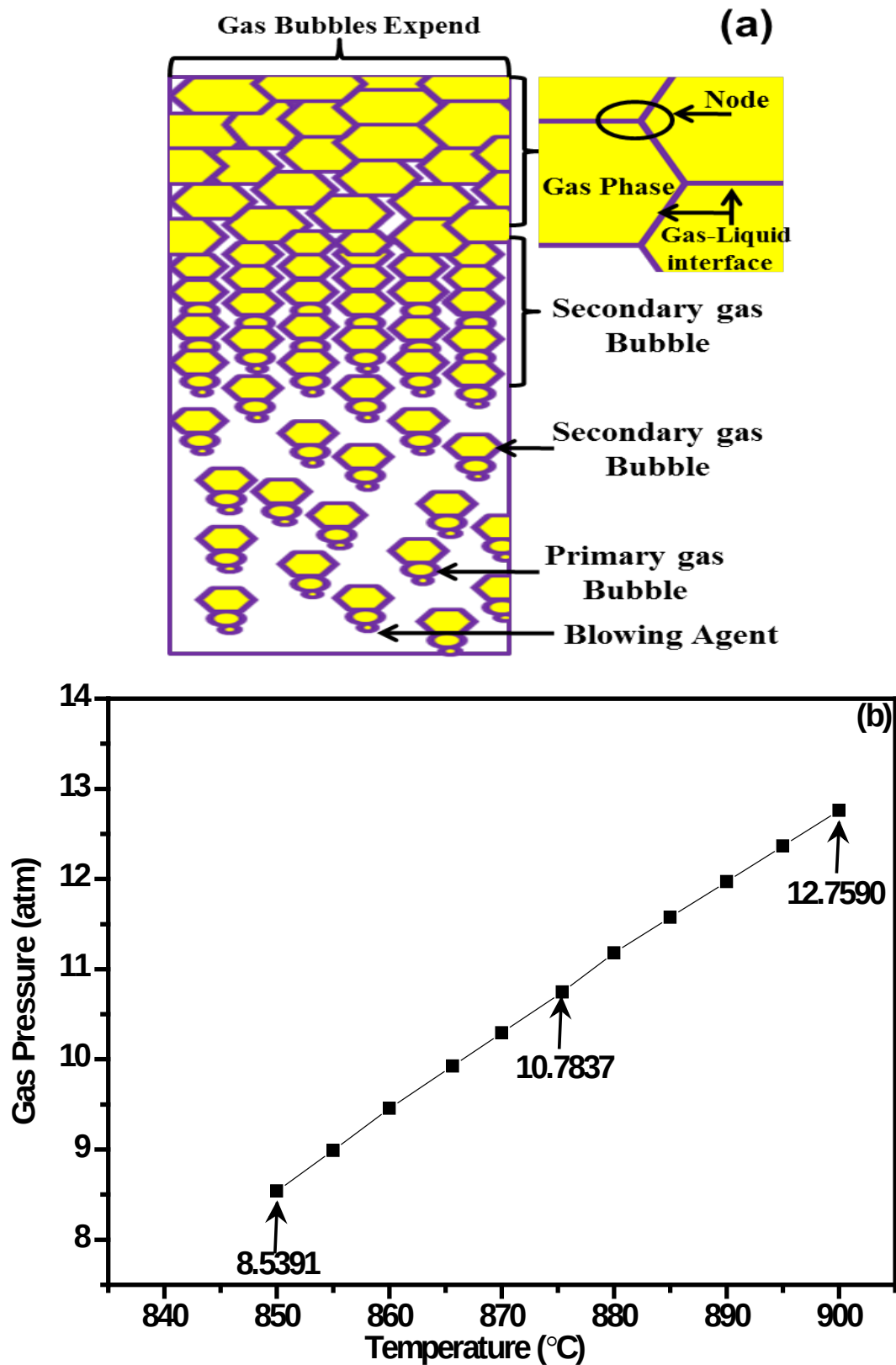


Figure 4.11: The effect of foaming temperature on the (a) formation of bubbles and (b) gas pressure inside bubble in the melt of LM13 alloy.

The above theoretical results of the comparison study are given on the basis of surface tension and viscosity of LM13 alloy foam. The surface tension and viscosity decreases with increasing foaming temperature of LM13 alloy foam. Experimentally it was observed that at 850 °C, the surface tension (805.28 mN/m) and viscosity (1.635 mPa-s) of the LM13 alloy are in perfect balance to produce the uniform cell size of LM13 alloy foam for 2wt.% blowing agent. When the temperature increases from 850 °C to 875 °C and 900 °C, the cell size of LM13 alloy foam observed is not uniform due to the decrease in surface tension and viscosity. The liquid film of molten LM13 alloy becomes thin due to lower surface tension and viscosity.

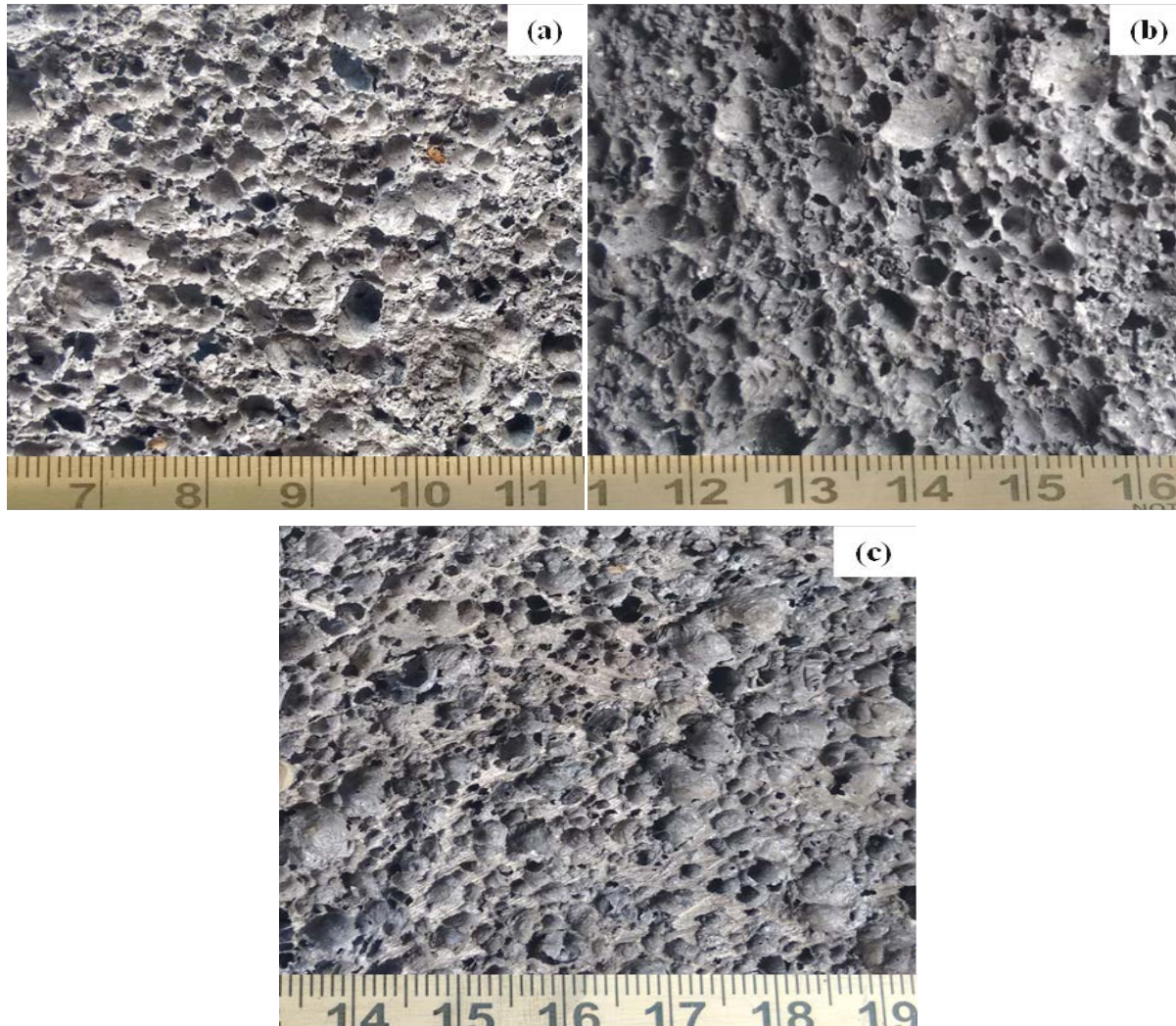
At 900 °C, bubbles collapse and generate the closely packed structure of the liquid alloy foam having thicker walls. All these observations are discussed while presenting the structural features. Experiments were preferred at different temperature viz. 850 °C, 875 °C and 900 °C with variation in blowing agent (CaCO_3) from 1.5 to 2.5wt % and reinforcement of zircon sand which varied from 2.5 to 15wt %. It was observed that the addition of higher amount of zircon sand particles in molten LM13 alloy increases the viscosity and decreases the cell size as temperature variation is not much. In order to have the uniformity in the text only critical parameters are discussed.

Figure 4.12 (a-c) presents the macrographs of AMF with 1.5wt.% blowing agent at 850 °C, 875 °C and 900 °C respectively. Figure 4.12a shows the structure of foam developed at 850 °C. It depicts nearly uniform cell size, a small size of the node with large ligament length. At this foaming temperature, there is nearly no bubble-free zone, coarsened cell structure, and unfavorable curvature in the alloy foam. CaCO_3 powder is easily decomposed into calcium oxide and carbon dioxide at 850 °C. At this temperature, the equilibrium presence of carbon dioxide gas increases due to the decrement in viscosity of the alloy which is sufficient enough for the foaming [12]. The released carbon dioxide gas accelerates and moves upwards, which causes bubble coalescence very rapidly. The uniformity of final bubble size and the total volume of porosity related to the carbon dioxide gas content in the melted alloy is determined by the growth rate between a bubble and solid-liquid interface [13].

Figure 4.12b shows the resultant morphology of regularly shaped cells as well as the overgrowth of some of the cells at 875 °C foaming temperature. As the temperature is increased the gas pressure also increases. Formation of these cells is due to the uniform growth as well as overgrowth of gas bubbles due to higher pressure. The cell diameter decreases steeply above 850 °C.

Table 4.1: Structural parameters of AMFs.

Sample ID	Holding temperature (°C)	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (μm)	Average Ligament Length (mm)
AMF- ^{1.5} F _{850°C}	850	0.95 ± 0.05	2.5 ± 0.10	1500 ± 100	0.65 ± 0.05
AMF- ^{1.5} F _{875°C}	875	0.65 ± 0.05	2.8 ± 0.10	1100 ± 100	0.50 ± 0.05
AMF- ^{1.5} F _{900°C}	900	0.80 ± 0.05	2.3 ± 0.10	1400 ± 100	0.55 ± 0.05

**Figure 4.12:** The effect of foaming temperature on the AMFs with 1.5wt.% blowing agent (a) 850 °C, (b) 875 °C and (c) 900 °C.

Foaming temperature directly affects the surface tension of melt and decomposition rate of the foaming agent. Such a high decomposition rate is not desired, because the excess gas released from the melt leads to the production of irregularly shaped cells and overgrowth of some cells of foamed aluminum [14].

Table 4.1 shows the change in the structural parameters of AMF developed with 1.5wt.% of blowing agent at different foaming temperatures. Further increment in the foaming temperature from 875 °C to 900 °C has led to a rapid generation of carbon dioxide gas. Moreover, the viscosity also decreased and liquid film becomes thin. Consequently, most of the air bubbles formed in the melt was larger in size and unstable. As they proceed upwards the top liquid pressure decreases and the bubble burst on reaching the melt surface [15]. The cell wall becomes weak and collapses with high pressure. The excess amount of released gas escaping through the melt results in low foaming efficiency [7, 8]. This results in a total collapse of the foamed structure and ultimately generates closely packed bubble like structure of the foam having thicker walls as shown in Fig. 4.12c.

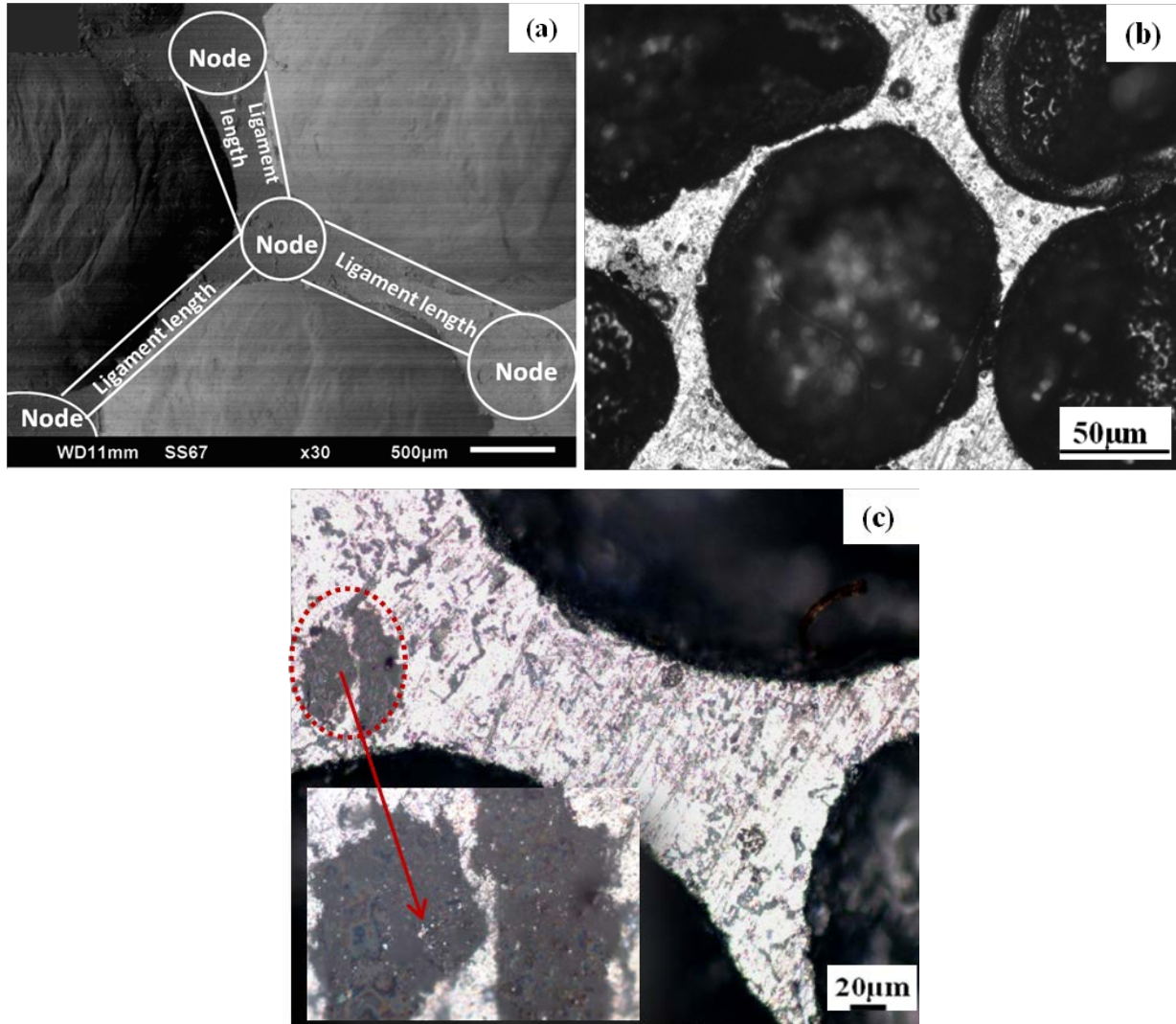


Figure 4.13: (a) SEM image and optical images (b) and (c) of AMF-^{1.5}F_{850 °C}.

Figure 4.13 shows the typical micrographs of AMF-^{1.5}F₈₅₀ °C. SEM image 4.13a shows the resultant morphology of regularly shaped ligaments and node position of cells at 850 °C foaming temperature. The optical microstructure (Fig. 4.13b) shows the distribution pattern of cells. At this temperature, some of the blowing agents having higher particle size do not decompose. This causes non-uniformity in the structure as is shown in Fig. 4.13c.

Table 4.2: The variation in the structural parameters of AMFs with 2wt.% amount of blowing agent at different foaming temperatures

Sample ID	Holding temperature (°C)	Density ₃ (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (μm)	Average Ligament Length (mm)
AMF- ² F ₈₅₀ °C	850	0.62 ± 0.05	2.2 ± 0.10	150 ± 25	0.40 ± 0.05
AMF- ² F ₈₇₅ °C	875	0.69 ± 0.05	2.0 ± 0.10	200 ± 25	0.25 ± 0.05
AMF- ² F ₉₀₀ °C	900	0.90 ± 0.05	1.8 ± 0.10	300 ± 25	0.15 ± 0.05

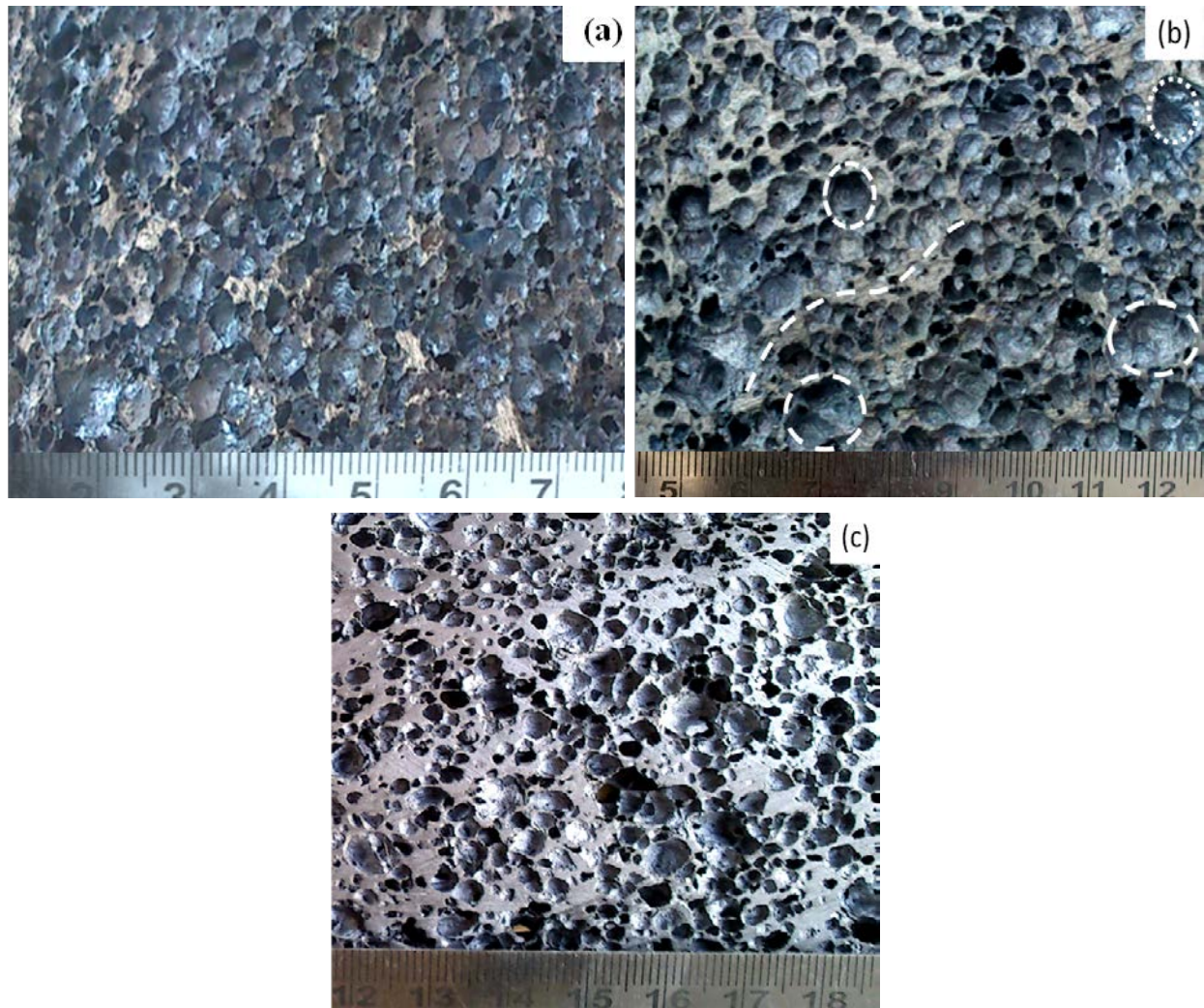


Figure 4.14: Effect of foaming temperature on the AMF with 2wt.% blowing agent (a) 850 °C, (b) 875 °C and (c) 900 °C.

It can be noted from the Fig 4.14 (a-c) that addition of 2wt.% blowing agent CaCO_3 has further refined the microstructure of the AMF at different foaming temperatures. It reveals wide variation in cell structure with different foaming temperature of the LM13 alloy. With the increase in foaming temperature from 850 °C to 900 °C, the density (gm/cm^3) and average cell wall thickness (μm) of foam is increased. However, the average cell size (mm), and average ligament length (mm) of AMF is decreased. At 850 °C foaming temperature, there is no bubble-free zone. It has coarsened cell structure with no unfavorable curvature in alloy foam (Fig 4.14a). At 850 °C, the equilibrium pressure of carbon dioxide gas increases, due to the decrement in viscosity of the alloy [12], which is sufficient enough for the foaming. The released CO_2 gas accelerates and moves upwards on the surface, which leads to rapid coalescence of bubbles.

The uniformity of the shape and size of bubbles to have the required porosity is dependent on the produced carbon dioxide gas inside the melt [13]. Table 4.2 gives the change in the structural parameters of AMF developed with 2wt.% blowing agent at different foaming temperatures. The distribution of cells observed is homogeneous in Fig. 4.14a. There are few cells which collapsed and formed irregular spherical cells in the AMFs at 875°C and 900°C. The changes in the structural parameter generally depend on the formation rate of gas bubbles, size and shape of the bubble (Table 4.2).

Figure 4.14b shows the resultant morphology of regularly shaped cells as well as the overgrowth of some of the cells at 875 °C foaming temperature. Morphology of these cells shows uniform growth as well as overgrowth of gas bubbles due to higher pressure. The cell diameter decreases steeply above 850 °C. The surface tension of the melt decreases with increase in temperature. Moreover, higher volume of gas is released as the temperature increases. For optimum shape and size of cells, a balance between surface tension and volume of gas is required. This means that the decomposition rate of blowing agent has to be controlled to get uniform shape and size of the foam cells. Higher decomposition rate may lead to generation of irregularly shaped cells and overgrowth of some cells in foamed aluminum alloy [16]. These cells even got interconnected as marked by the dotted line arrow in Fig. 4.14b and also overgrowth as encircled.

Further increment in the foaming temperature from 875 °C to 900 °C has led to a rapid generation of carbon dioxide gas that results in increase of the cell size of alloy foam. The liquid film of molten LM13 alloy becomes thin due to the lower viscosity at 900 °C. Consequently, larger size unstable air bubbles are formed. As the gas proceeds upwards the top liquid pressure decreases

and the bubble burst when it reaches to the surface [9, 17]. The cell wall becomes weak and collapses with high pressure. The higher volume of gas makes it escape through the molten mass and results in low foaming efficiency [9, 18]. This results in a total collapse of the foamed structure and ultimately generates the closely packed structure of the aluminum foam having thicker walls as shown in Fig. 4.14c. This analysis indicates that 850 °C foaming temperature is optimum for LM13 alloy.

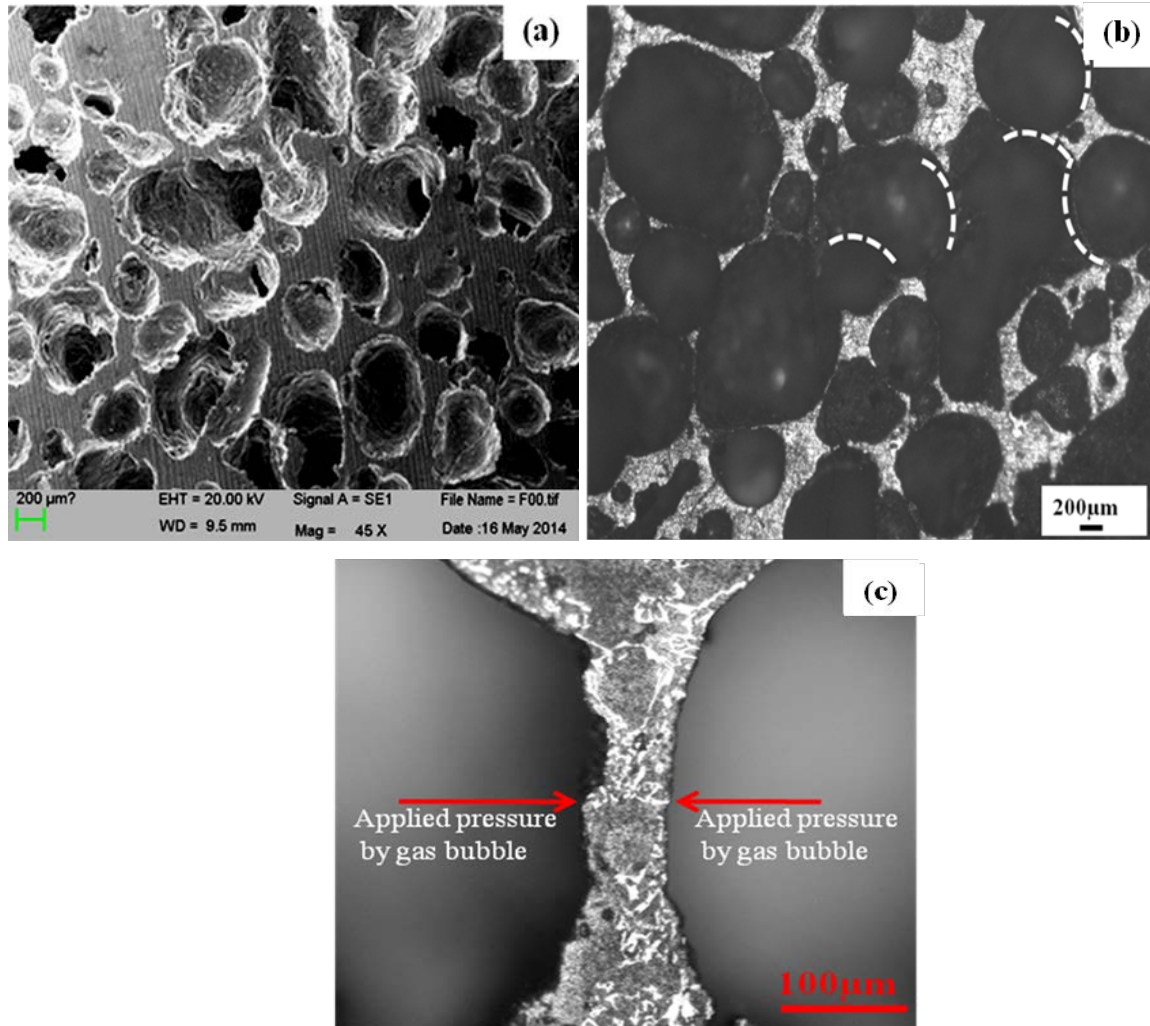


Figure 4.15: (a) SEM image and optical images (b) and (c) of AMF-² F₈₅₀ °C.

Figure 4.15 shows the typical micrographs of AMF-² F₈₅₀ °C. In Fig. 4.15a internal structures are seen on the upper middle portion in the SEM image of the foam. The collapse of some cells occurs during gas diffusion from one cell to other making interconnect path. These interconnects of cell walls are responsible for the drainage, rupture and coarsening of the foam during solidification [10].

The difference in internal gas pressures is responsible for formation of different cells because internal pressure is inversely proportional to the cell radius. The number of interconnections between cells is increased with increased internal pressure, which is seen in the upper portion of the closed cell foam. Cells of non-uniform shape having thinner cell walls are obtained as can be seen in the structure. The helical growth inside the cells demonstrates that the released gas pressurizes the cell walls uniformly.

The microstructure of the closed cell alloy foams was analyzed in the form of the cell size and their distribution which is shown in Fig. 4.15b. The most notable structural feature of alloy foam is the cell size with thinner cell faces at 850 °C which are shown by dotted line in the micrograph. It is obvious that ligament and node have appeared in the cellular structure of the alloy foam. The optical micrograph (Fig. 4.15c) shows the variation in curvature of cell wall due to difference in pressure between two cells. Higher pressure leads to uniform curvature whereas low pressure leads to non-uniform curvature as metal flow occurs from higher pressure to lower pressure.

Table 4.3 gives the change in the structural parameters of AMF developed with 2.5wt.% of blowing agent at different foaming temperatures. At lower foaming temperatures the foaming agent releases less amount of CO₂. The CO₂ gas seems to be adequate for generating uniform cell size foam at 850 °C (Fig 4.16a).

The distribution of cells observed is homogeneous in Fig 4.16b. However, there are few cells which collapsed and formed irregular spherical cells in the AMF at 875 °C. At 900°C, the equilibrium presence of gas increases due to the decrement in viscosity of the alloy. The gas bubble moves upwards, which causes bubble coalescence very rapidly as shown in Fig 4.16c.

Table 4.3: The structural parameters of AMFs.

Sample ID	Holding temperature (°C)	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (mm)	Average Ligament Length (mm)
AMF- ^{2.5} F ₈₅₀ °C	850	0.51± 0.05	2.3± 0.10	0.80± 0.05	0.7± 0.05
AMF- ^{2.5} F ₈₇₅ °C	875	0.45± 0.05	2.5± 0.10	0.30± 0.05	0.8± 0.05
AMF- ^{2.5} F ₉₀₀ °C	900	0.95± 0.05	3.1± 0.10	1.35± 0.05	0.8± 0.05

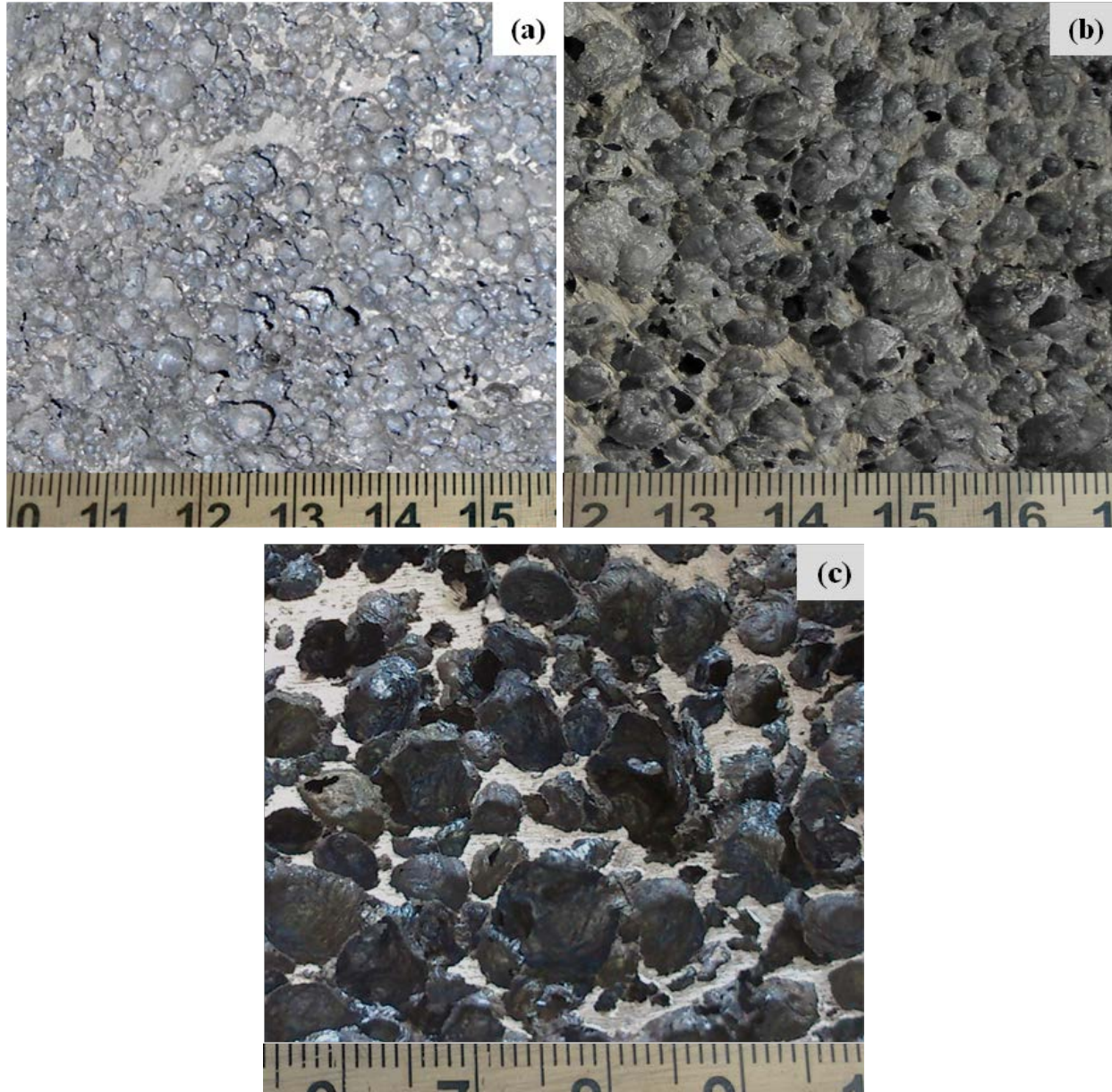


Figure 4.16: The effect of foaming temperature on the AMF with 2.5wt.% blowing agent (a) 850 °C, (b) 875 °C and (c) 900 °C.

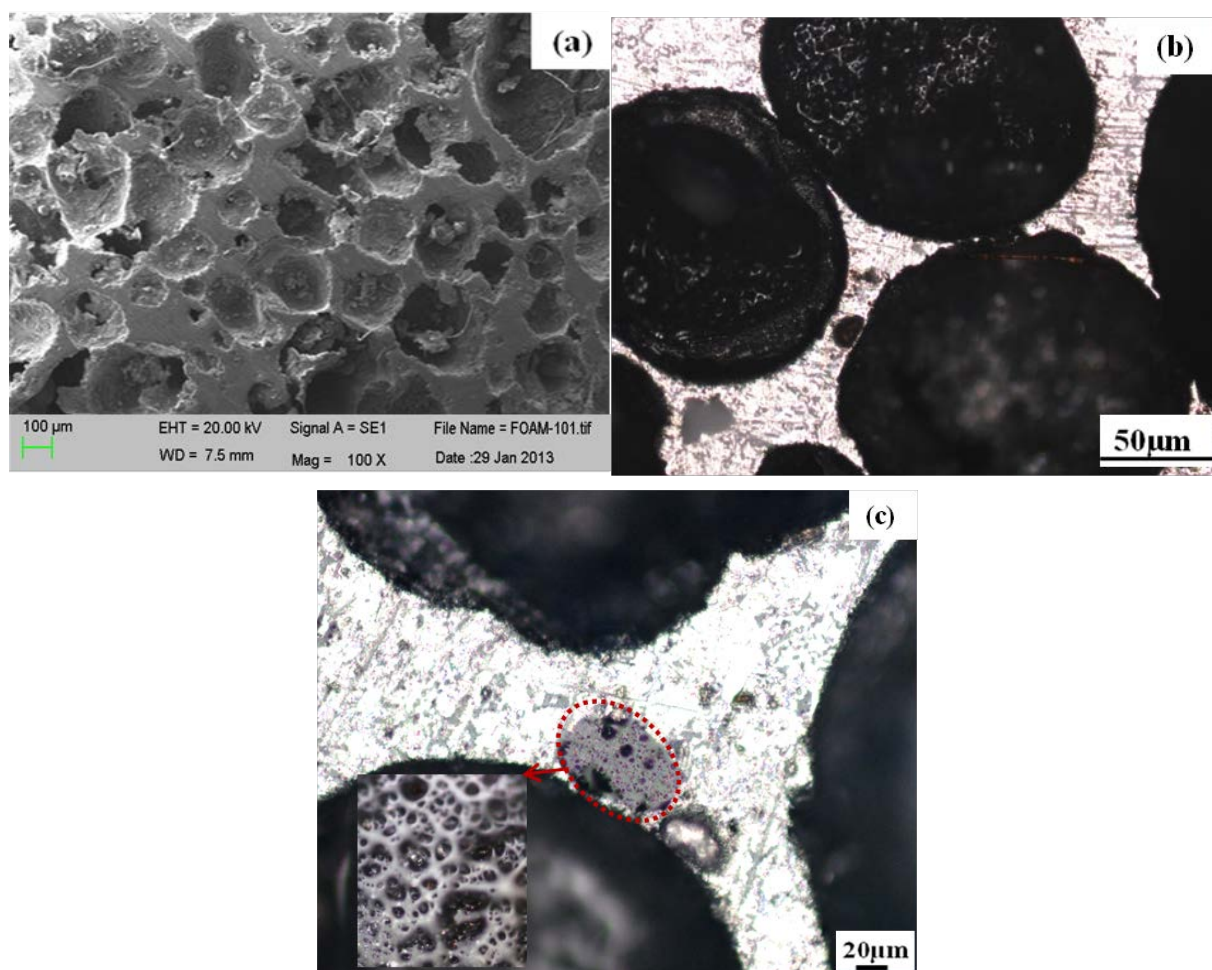


Figure 4.17: (a) SEM image and optical images (b) and (c) of AMF-^{2.5}F_{850 °C}.

Figure 4.17 shows the typical micrographs of AMF-^{2.5}T_{850 °C}. SEM image (Fig. 4.17a) depicts the distribution of cells which is nearly homogeneous. However, with increase in the amount of blowing agent from 2wt.% to 2.5wt.%, cell walls have become thinner by the easy migration of ligaments because of increased decomposition rate in the melt alloy. The gas pressure inside the bubble is increased and the differences of capillary pressure may cause an over pressure along the walls. This may lead a low level of pressure in the ligament, so movement of the cell wall within the liquid is easily distorted. The over pressure inside of cell leads to thinning of the cell wall and finally it ruptures. As a result, coalescence in cell walls has occurred. However, at certain places bigger size blowing agent does not decompose properly. Some amount of blowing agent particles remain in melt which is shown in Fig 4.17c. At 850 °C, the larger size of blowing agent is not decomposed properly due to higher viscosity. It is also possible that some CaCO₃ particles may agglomerate due to poor wettability between CaCO₃ and molten metal.

4.4 Microstructure Analysis of AMCFs

The mechanical properties of composite foam are affected by the cell geometry and its uniform distribution. One can achieve higher strength if the cells are smaller and have a uniform distribution. In composite foams the cell geometry and their distribution are controlled by reinforcing ceramic powder which leads to stable foams that can prevent foam collapse. The high viscosity and low surface tension is required to make stable composite foam. The viscosity of zircon sand reinforced composite propellant slurry is related to temperature. It also depends on the molten phase ingredients like content, geometry and surface morphology of zircon sand. The cell wall is strengthened by the reinforced ceramic particles [19].

Foams reinforced with different amount of zircon sand particles (2.5, 5, 7.5, 10, 12.5 & 15wt.%), have been developed. The structural features of AMCFs is similar to that of AMFs. As mentioned earlier that higher content of zircon sand as reinforcement leads to higher viscosity of melt causing thick walls. Reinforcement up to 5wt.% zircon sand provide better structural features. To avoid the muddle, we have provided only the macro and micro structures of AMCF reinforced with 5wt.% of zircon sand particles in this section and discussed accordingly. However, products developed were used for subsequent study which has been described in next chapters. Here in this reaction the term “AMCF” used frequently is for AMCF pertaining with 5wt.% of zircon sand in the entire discussion part.

In the following section we have analyzed the theoretical aspect of reinforced particles. In composite foams the cell geometry and their distribution are controlled by reinforcing ceramic powder which leads to stable foams that can prevent foam collapse and coalescence, up to the end of the solidification. The high viscosity and low surface tension is required to make stable composite foam. The viscosity of zircon sand reinforced composite propellant slurry is related to temperature. It also depends on the molten phase ingredients like content, geometry and surface morphology of zircon sand. The slow rate of liquid drainage is responsible for higher viscosity. It affects the critical cell wall thickness. The effect of gravity and low surface tension is responsible to decrease the cell wall thickness. The influence of ceramic particles on the viscosity is significant. The relationship between volume fraction of ceramic particle and relative viscosity is observed by Einstein’s viscosity equation [4.5]:

$$\eta_c = \eta_L [1 + 2.5\phi + 10.25\phi^2 + A \exp(B * \phi)] \quad (4.5)$$

Where, $A = 0.002732$, $B = 16.6$, ϕ is the solid volume fraction, η_c is the viscosity of composite, η_L is an initial viscosity of the LM13 alloy melt. This equation reveals that the volume fraction of ceramic particles is responsible for a higher viscosity of the melt LM13 alloy. However, for uniform distribution, the ceramic particles should not settle to the bottom of the mixer in the mold [20]. The viscosity of composite decreases with increase in foaming temperature (Fig. 4.18) which is affected by the wettability of the phases of solid and liquid. Pelofsky [21] proposed the following relationship between surface tension and viscosity:

$$\ln \sigma = \ln A + \frac{B}{\eta_c} \quad (4.6)$$

Where σ is surface tension, η_c is viscosity while in A and B are substance-dependent constants. We have found the relationship between surface tension and viscosity with the help of Eq. (4.6). However, the use of Eq. (4.6) did not result in an accurate fit of the $(\ln \sigma - \eta_c^{-1})$ data therefore, Eq. (4.6) was modified to find the accurate value of data for its fitting and developed a new relation between surface tension and viscosity as [21]:

$$\ln \sigma = \ln A + B\eta_c^{-1.395} \quad (4.7)$$

Where A and B are substance constants and is dependent on material to materials. In the composite foam, the functional dependence of surface tension with temperature of composite has been established. The corresponding graph has been drawn and shown in Fig. 4.19 with the help of Eq. (4.6). The foaming temperature affects the viscosity and bubbles stability. However, the wettability of the phases of solid and liquid is affected on the thickening and bubbles stability. The distribution and position of the zircon particles on the center of the cell wall and node is dependent on the interface energy balance at the melt-zircon particle interface.

Gergely et al. [22] have investigated the effect of gas reaction on the cell wall surface and amount of ceramic particle which is responsible to increase the foamability. At 850 °C, the viscosity (2.055 mPa-s) and surface tension (832.43 mN/m) of LM13 alloy composite foam are not sufficient to develop the uniform cell size. The increase in foaming temperature from 850 °C to 875 °C and 900 °C, leads to increment in the cell size of composite foam as the viscosity and surface tension of liquid decreased. Due to decrement in the viscosity (1.88 mPa-s) and surface tension (825.12 mN/m) of the melt LM13 alloy composite at 900 °C, the cell formation becomes easier. This decrement in the viscosity and surface tension is responsible to generate uniform cell size and stable foams at this temperature.

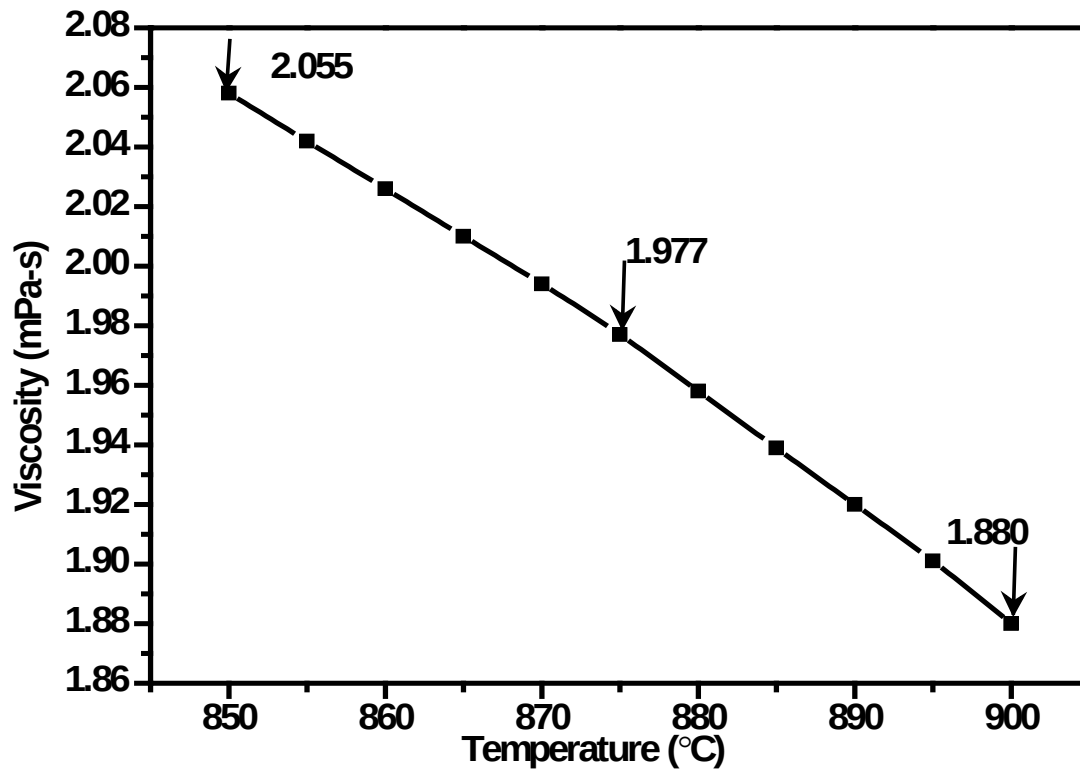


Figure 4.18: The effect of foaming temperature on the viscosity of the LM13 alloy and composite foams.

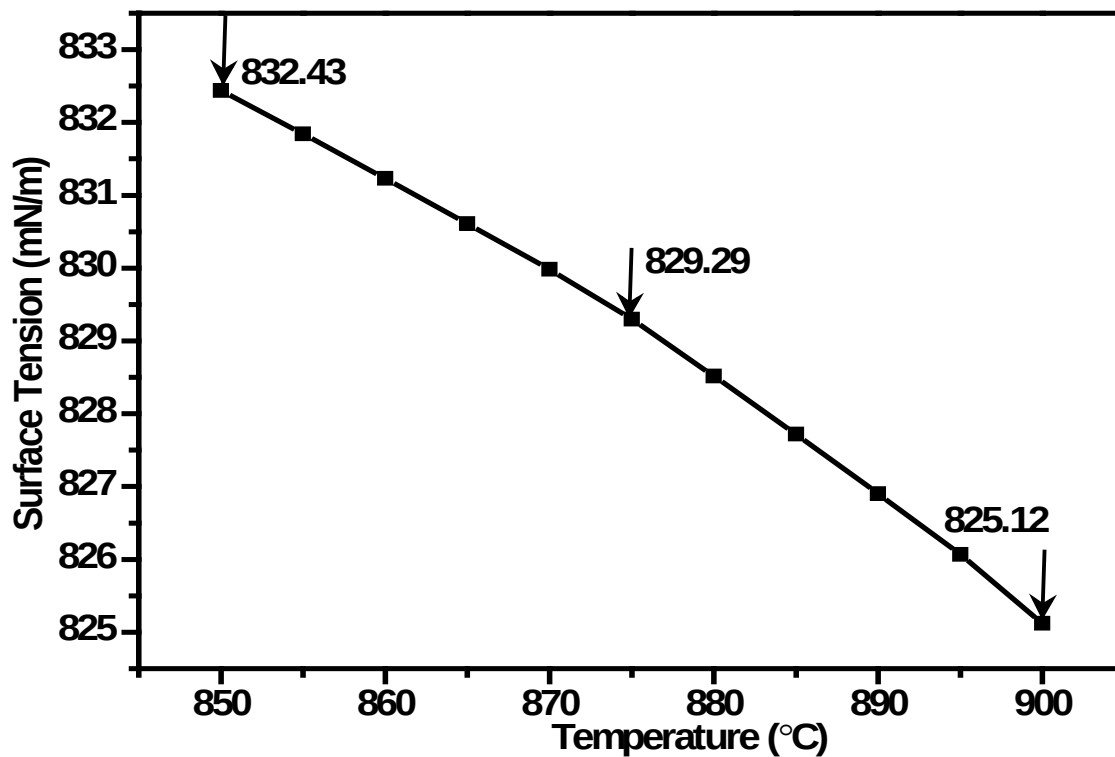


Figure 4.19: The effect of foaming temperature on the surface tension of the LM13 alloy composite foams.

The macrographs of AMCF with 5wt. % ZrSiO₄ and 1.5wt.% blowing agent which was produced at different foaming temperatures are shown in Figure 4.20 (a-c). Table 4.4 gives the change in the structural parameters of AMCF developed with 1.5wt.% of blowing agent at different foaming temperatures. It reveals wide variation in cell structure with variation in foaming temperature.

Table 4.4: The change in the structural parameters of AMCF with 1.5wt.% of blowing agent.

Sample ID	Holding temperature (°C)	Density (gm/cm ³)	Average Cell Size (mm)	Cell wall thickness (mm)	Average ligament length (mm)
AMCF- ^{1.5} C _{850 °C}	850	0.90 ± 0.05	2.2 ± 0.10	1.20 ± 0.10	1.85 ± 0.10
AMCF- ^{1.5} C _{875 °C}	875	0.82 ± 0.05	3.2 ± 0.10	0.70 ± 0.10	0.75 ± 0.10
AMCF- ^{1.5} C _{900 °C}	900	0.85 ± 0.05	3.0 ± 0.10	0.95 ± 0.10	1.20 ± 0.10

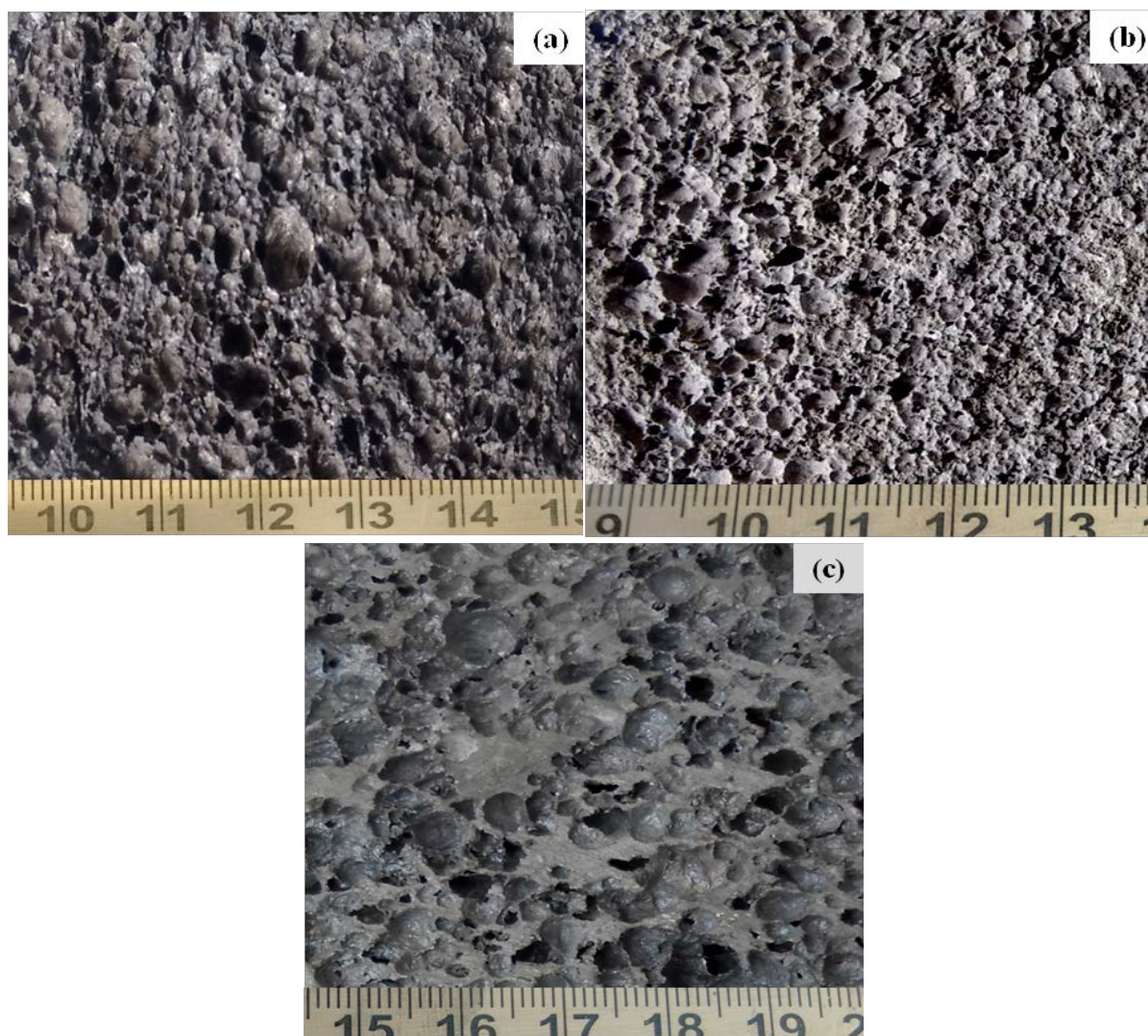


Figure 4.20: Effect of foaming temperature on the AMCF with 5 wt.% ZrSiO₄ and 1.5wt.% blowing agent(a) 850 °C, (b) 875 °C and (c) 900 °C.

As a result, the density, average cell walls thickness and ligament length decreases with increase in the foaming temperature from 850 °C to 900 °C of AMCF. At 850 °C, 1.5wt.% blowing agent with 5wt.% ceramic particles did not produce sufficient gas pressure for making uniform cell size of composite foam (Fig. 4.20a). At this temperature, CaCO_3 was not completely decomposed to generate sufficient gas pressure for making uniform cell size because ZrSiO_4 addition is responsible for increasing the viscosity of molten metal. Figure 4.20b shows that the most of the cells are smaller and some cell sizes are larger due to the collapse of smaller cells. Figure 4.20b shows the closely packed bubbles like structure of the AMCF developed by the addition of 1.5 wt. % CaCO_3 . Foam with a very fine cell structure with some signs of collapse cells is shown in Fig. 4.20c.

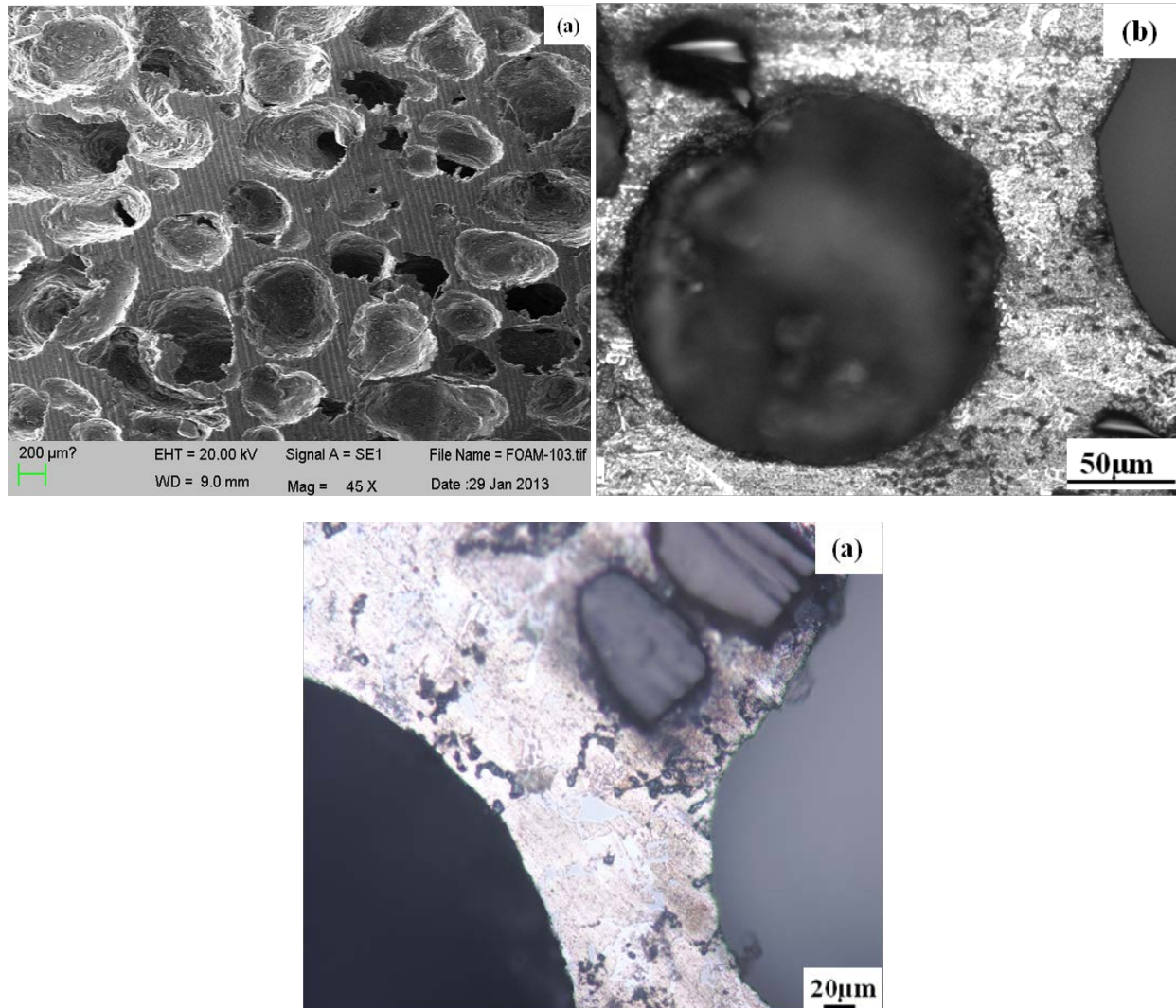


Figure 4.21: (a) SEM image and optical images (b) and (c) of AMCF with 1.5wt.% blowing agent at 900 °C.

This is due to diffusion of gas from one cell to another which is responsible for the drainage and coarsening of the foam during solidification. At 900 °C, the viscosity of molten metal decreases and the decomposition rate of CaCO_3 increases. This leads to the increase in cell size and formation of interconnected cells (Fig.4.20c).

Table 4.5: The change in the structural parameters of AMCF with 2wt.% of blowing agent.

Sample ID	Holding temperature (°C)	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (mm)	Average Ligament Length (mm)
AMCF-2 C ₈₅₀ °C	850	0.54 ± 0.05	3.4 ± 0.10	0.38 ± 0.05	0.95 ± 0.10
AMCF-2 C ₈₇₅ °C	875	0.42 ± 0.05	3.6 ± 0.10	0.35 ± 0.05	1.40 ± 0.10
AMCF-2 C ₉₀₀ °C	900	0.77 ± 0.05	3.1 ± 0.10	0.75 ± 0.05	1.10 ± 0.10

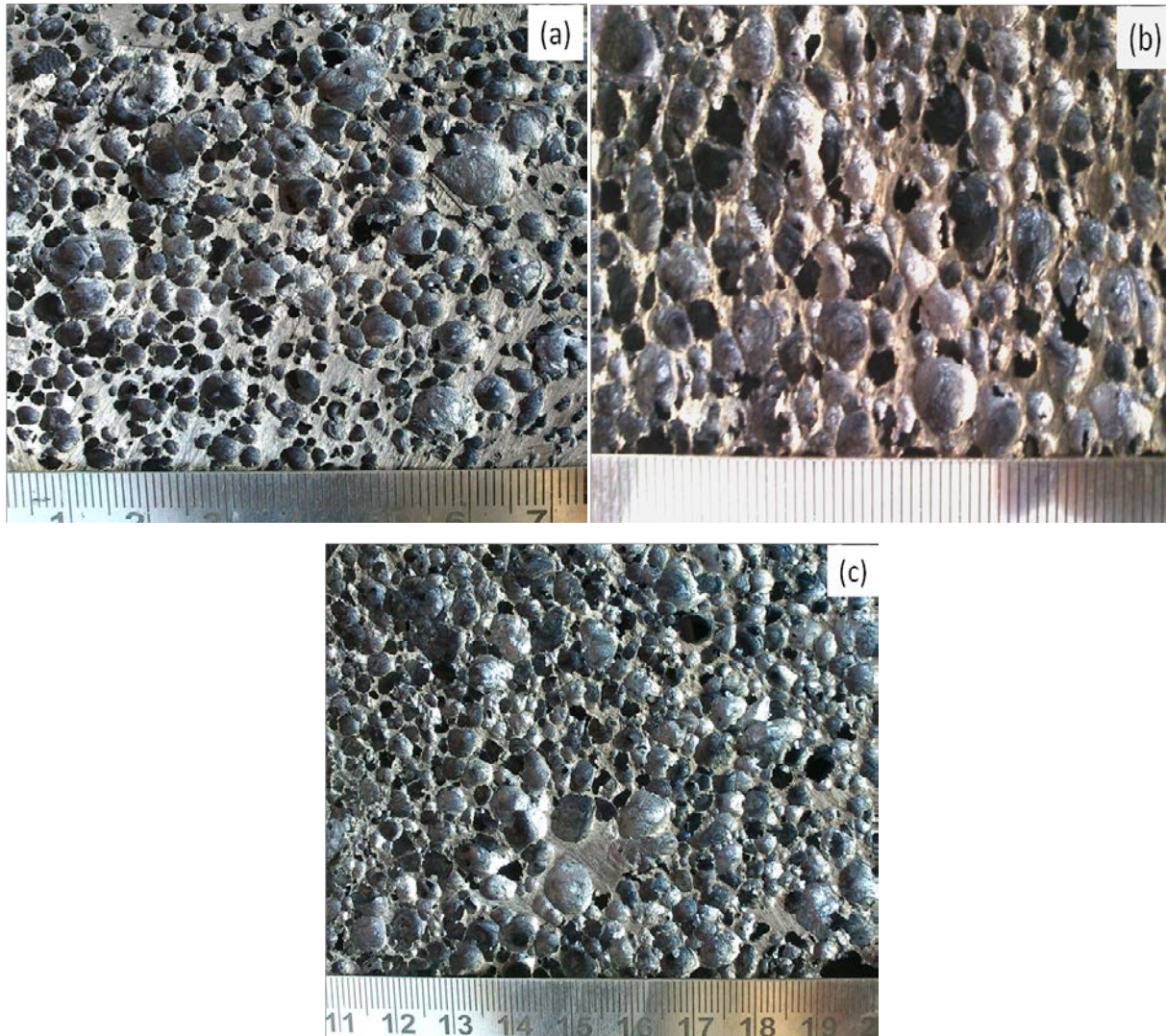


Figure 4.22: Effect of foaming temperature on the AMCF with 5 wt.% ZrSiO_4 and 2wt.% blowing agent(a) 850 °C, (b) 875 °C and (c) 900 °C.

Figure 4.21a shows the SEM image of the closed cell structure. The geometry of the cell structure is nearly of spherical shape. The stability of cell structure (cell size and cell wall thickness) is controlled by the position, size and volume fraction of the ZrSiO_4 particles. The stability of cell structure is increased if the ZrSiO_4 particles occupy the node and ligament of the wall (Fig. 4.21b). At higher magnification, the particle position is shown on the node of cell wall in Fig. 4.21c.

Figure 4.22 (a-c) shows the macrostructure of composite foam prepared at different foaming temperatures (850 °C, 875 °C and 900 °C). Table 4.5 presents the change in the structural parameters of AMCF developed with 2wt.% blowing agent at different foaming temperatures. At 850 °C, addition of 5wt.% ceramic particle increases the viscosity of the liquid LM13 alloy.

At this condition, the pressure of released gas during decomposition of the CaCO_3 is lower as compared to base LM13 alloy. CO_2 gas expands inside the liquid alloy and generates the bubble like structure. At 850 °C, 2wt.% blowing agent with 5wt.% ceramic particles did not produce sufficient pressure for making uniform cell size composite foam (Fig. 4.22a).

The influence of foaming temperature on the cell size and viscosity of AMCF sample is observed to vary. Below 875 °C, there is little variation in porosity as the amount of CO_2 gas produced is just enough. However, foaming temperature directly affects the decomposition rate of the blowing agent and liquid surface tension. At higher temperature viscosity and surface tension decreases. Due to decrement in the viscosity of the liquid alloy and increment in the decomposition rate of CaCO_3 , the cell formation becomes easier [23, 24]. This is responsible to generate uniform cell size and stable foams. Table 4.5 gives the variation in the structural parameters of composite foam developed at different foaming temperatures. The increase in foaming temperature from 850 °C to 875 °C leads to increased cell size of closed cell composite foam as the viscosity of melt decreased which is shown in Fig. 4.22 b. At higher temperature surface tension is decreased [25]. This causes increase in ligament length and node size both, thus an increase in cell size is observed [26]. Furthermore, the increase in the foaming temperature from 875 °C to 900 °C seems to have little effect on the microstructure such as node size and ligament length of the cells which is shown in Fig. 4.22c. The results show that the CO_2 gas flow rate changes the cell size because of the addition of zircon particles. At the higher foaming temperature (900 °C), the rate of decomposition of CaCO_3 becomes higher so the equilibrium carbon dioxide gas pressure also increases. This causes the escaping tendency of gas and thus reduces the stability of bubble because of higher decomposition rate of foaming agent [27]. This leads to non-uniform cell size (Fig. 4.22c).

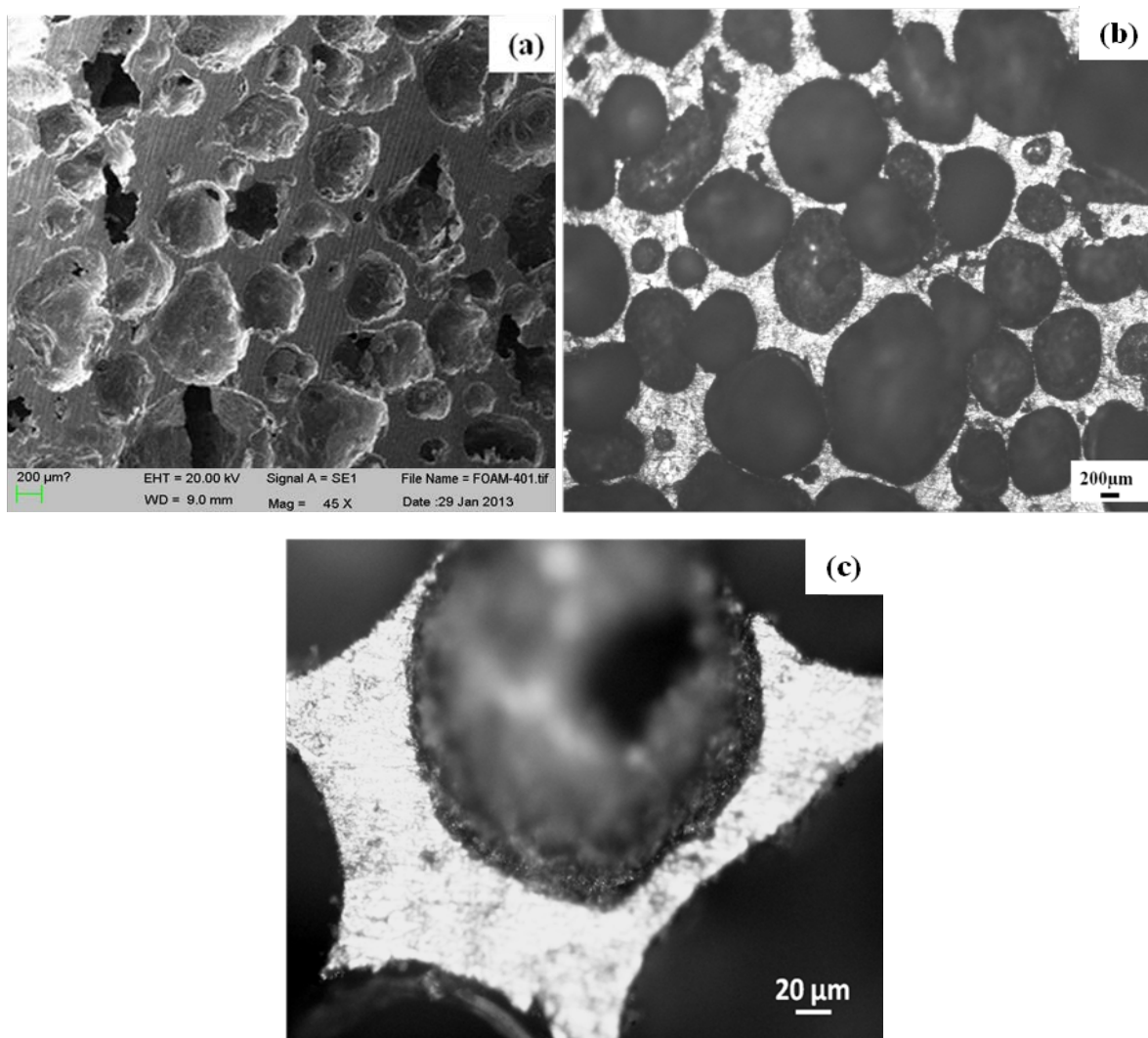


Figure 4.23: (a) SEM image and optical images (b) and (c) of AMCF with 2wt.% blowing agent at 900 °C.

The wettability (**contact angle: 145 °C**) of the zircon particles with the liquid alloy provides the stability to the foam which is greatly dependent on the volume of gas which ruptures the cell wall and drains out. The role of the size, shape and amount of zircon sand on gas drainage causing rupture of cells affects the foam stability. The higher viscosity is responsible to slow down the melt flow, which delays the cell wall drainage before it solidifies during the addition of zircon particles in the liquid alloy [28].

In Figure 4.23a SEM image shows the uniform distribution of cells in the closed cell foam. The cell wall was thinnest in the middle section, became increasingly thicker towards the edges, and eventually formed circular fillets as it intersected with neighboring walls (Fig. 4.23b). At higher magnification, Fig. 4.23c shows that in the middle section of foam, the cell wall was thinnest.

Figure 4.24 (a-c) shows the microstructure of composite foam prepared at different foaming temperatures (850 °C, 875 °C and 900 °C). Table 4.6 gives the change in the structural parameters of AMCF developed with 2.5 wt.% blowing agent at different foaming temperatures. At 850 °C, the uniform cell size is not developed with 2.5wt.% CaCO_3 and 5wt.% ZrSiO_4 . The molten mass at 850°C containing maximum of 2.5wt.% foaming agent releases large amount of CO_2 gas. This gas expands in the liquid metal and creates uniform size bubble structure. These bubbles are interconnected to each other as shown in Fig. 4.24 (a). Moreover, the increase in cell wall thickness also means that more segregation of particles at the particle-liquid interface creates a more tortuous path for the liquid and acts as a liquid flow barrier from the cell wall towards the plateau border. After decomposition, the bubble so formed tries to come out at the surface. Consequently, most of the CO_2 bubbles in the melt meet each other and acquires large area. Under the influence of temperature these unviable bubbles burst on reaching the melt surface. Few stable bubbles ascended to the top surface of the aluminum melt and formed an initial hull of two or three layers of roughly ellipsoidal foam cells [14]. Further increment in the foaming temperature from 850 °C to 875 °C lead to rapid generation of gas bubbles and results in decrement in the cell size of AMCF (Fig. 4.24b). The liquid film of molten metal becomes thin due to lower viscosity at higher foaming temperature (875 °C). Consequently, unstable air bubbles of larger size are formed. As the gas bubbles proceeds upwards the top, liquid pressure decreases and some gas bubble burst when it reaches to the surface [17]. At this temperature, some small gas bubbles closer to burst bubbles are also generated. This is responsible to decrease the cell size of AMCF. The cell wall becomes weak and collapses with high pressure. At high temperature the viscosity of the melt decreases, the gas produced by the decomposition of CaCO_3 is sufficiently high but the lower viscosity at 900 °C allows the gas to escape from the melt. This causes the collapsing of cells and reduces the cell size of AMCF (Fig. 4.24c).

Table 4.6: The change in the structural parameters of AMCFs with 2.5wt.% of blowing agent.

Sample ID	Holding temperature (°C)	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (mm)	Average Ligament Length (mm)
AMCF- ^{2.5} C ₈₅₀ °C	850	0.78 ± 0.05	3.1 ± 0.10	1.2 ± 0.10	1.25 ± 0.10
AMCF- ^{2.5} C ₈₇₅ °C	875	0.63 ± 0.05	3.7 ± 0.10	0.40 ± 0.10	1.60 ± 0.10
AMCF- ^{2.5} C ₉₀₀ °C	900	0.88 ± 0.05	3.3 ± 0.10	0.95 ± 0.10	1.40 ± 0.10

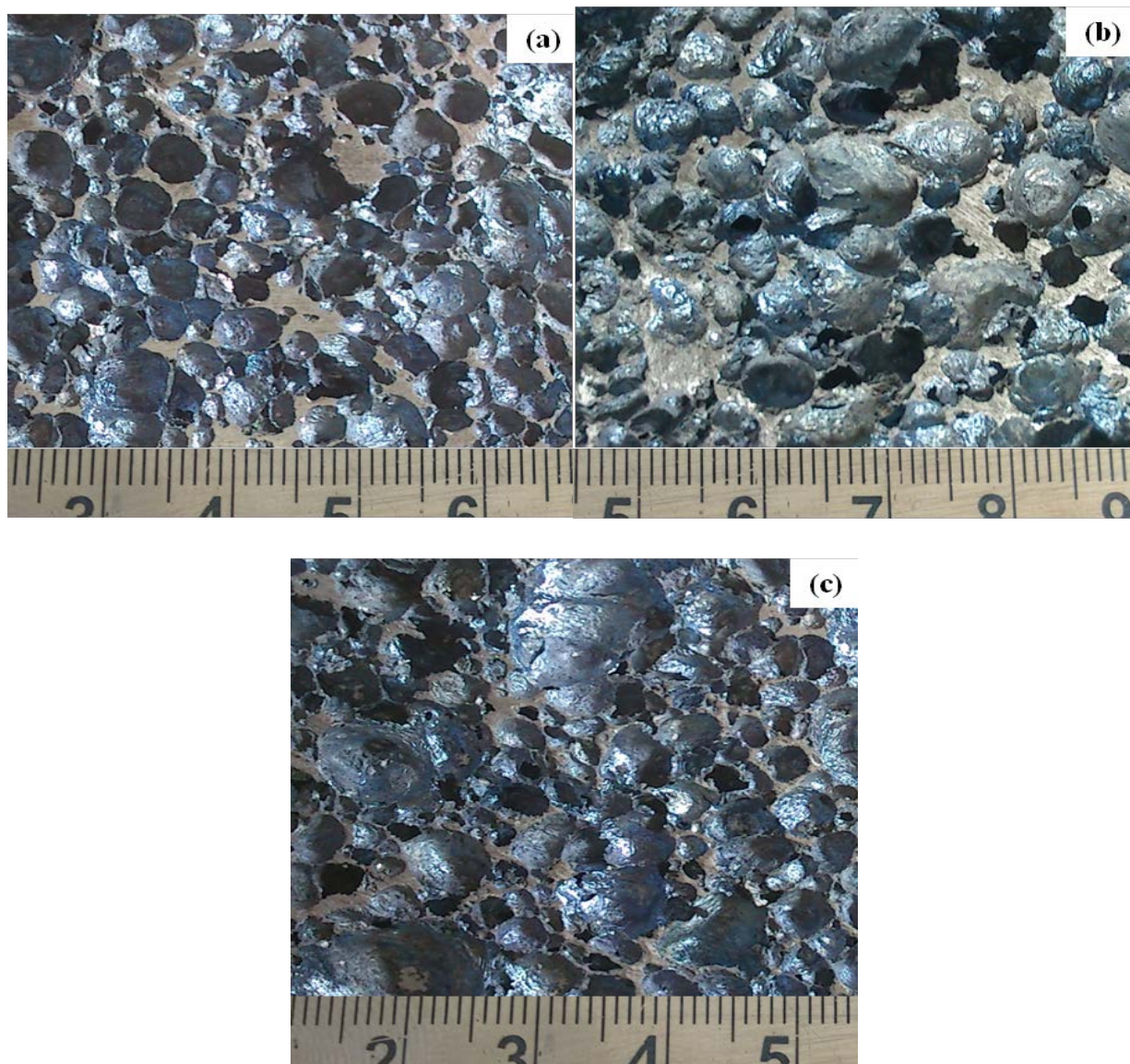


Figure 4.24: Effect of foaming temperature on the AMCFs with 5 wt.% ZrSiO₄ and 2.5wt.% blowing agent(a) 850 °C, (b) 875 °C and (c) 900 °C.

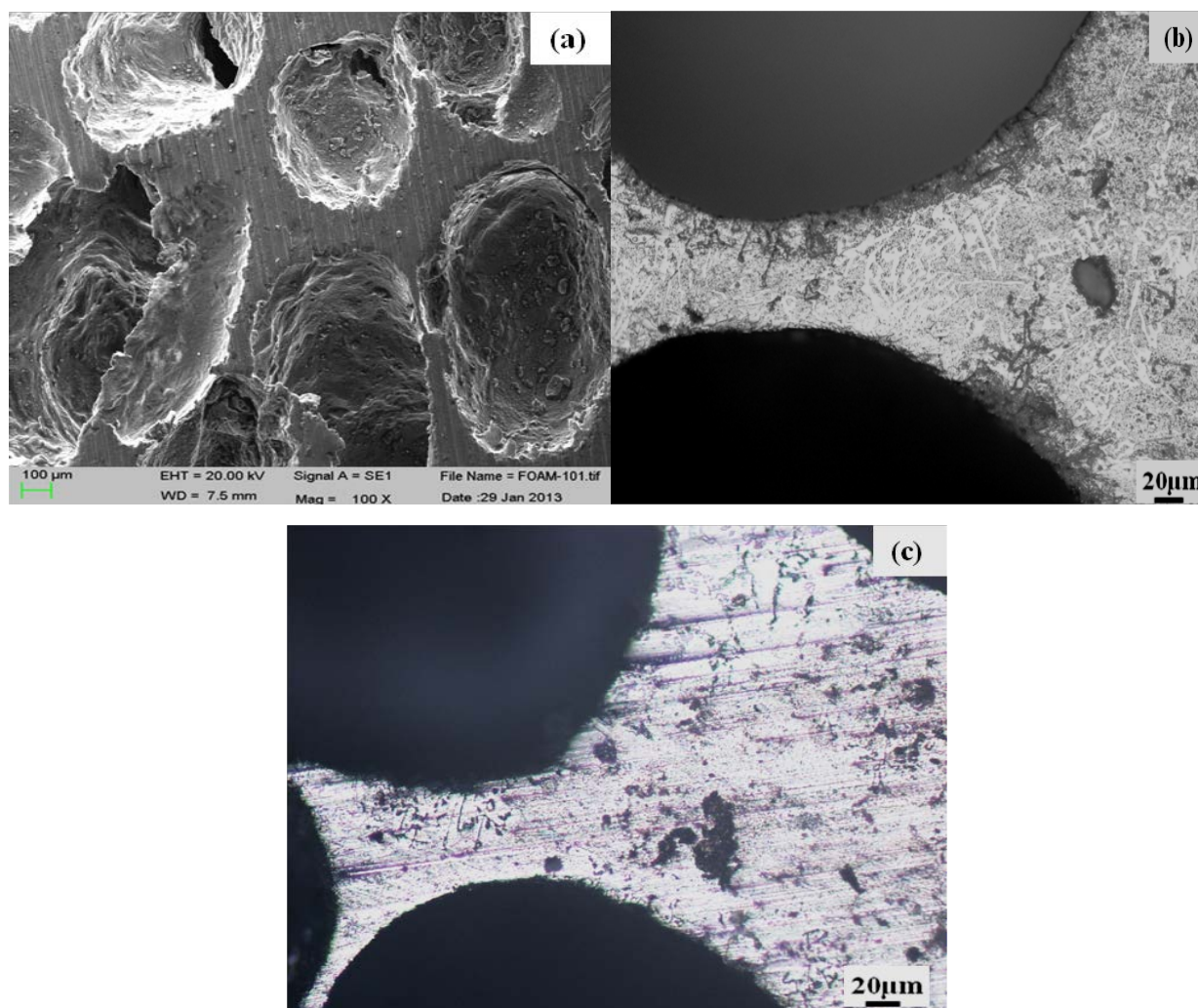


Figure 4.25: (a) SEM image and optical images (b) and (c) of AMCF with 2.5wt.% blowing agent at 900 °C.

SEM image in Fig 4.25 shows the cell geometry of closed cell foam and some ZrSiO_4 particles attached to inside cell wall. This helps to stabilize the cell wall. The stability of the cell walls increases with increase in concentration of ceramic particles, as shown in Figs. 4.25 (b & c). The Si morphology in the form of acicular eutectic is observed in the surrounding area of the particles in Fig. 4.25 b. Acicular eutectic is observed because the brittle phase (ZrSiO_4 particles) offer favored sites for the effective nucleation of eutectic silicon and also the difference of thermal conductivity between the brittle phase (ZrSiO_4 particles) and the ductile phase (matrix) creates a thermal gradient at the interface. The heterogeneous nucleation of silicon in the vicinity of the reinforced particles leads to modify in Si morphology from needle shaped to non-acicular (Figs. 4.25 b & c). This category of globular or blunted type of Si morphology enhances the wear resistance and provides better mechanical properties of AMCFs.

In order to analyse the effect of microstructure of developed materials on the microhardness and wear behavior, this section presents the systematic discussion about microhardness and wear behavior analysis of AMCs, AMFs and AMCF respectively. The results of microhardness and wear tests obtained from cast AMCs, AMF and AMCF is discussed in this section. Reinforcement of fine (1-32 μm) and coarse (106-125 μm) size ZrSiO_4 particles varying from 2.5-15wt.% have been used to develop AMCs and AMCF.

4.5 Microhardness of AMCs

Microhardness measurement at different phases of AMCs has been carried out to study the effect of reinforced particulates on the alloy matrix. Figure 4.26 (a & b) shows the microhardness indentations taken from these regions. The variation in the size of the indentation in different zones was observed. As given in the Table 4.7 the hardness value of AMC increased with the weight fraction of ZrSiO_4 particulates. During the hardness test, the plastic flow of the materials occurs on the surface just below the indenter. This localized plastic flow of the material does not alter the elastic behavior of the material in rest of AMC samples. Hence, plastic flow results in the increase in density as compared to regions away from the depression. At the same time, very large hydrostatic pressure under the indentation leads to the volume contraction of the metal matrix. So during the indentation, the developed pressure increases the particle concentration due to the non-uniform matrix flow in the localized region. Increase in hardness as observed with the increased amount of ZrSiO_4 addition vary with the particle size in AMC.

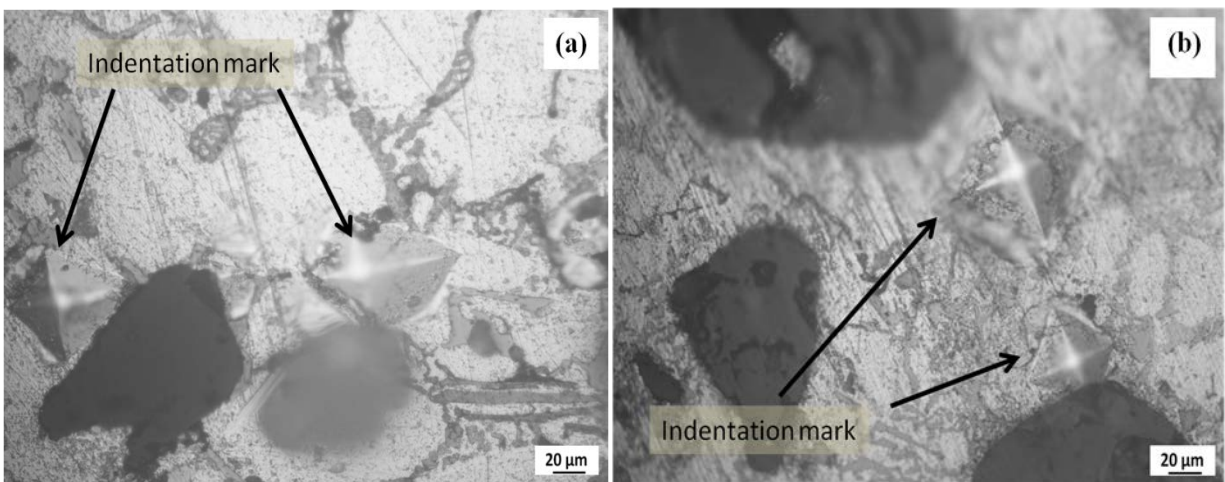


Figure 4.26: Optical micrographs showing different microhardness indentations on different phases of (a) $\text{AMC-}^{15}\text{C}_{\text{fine}}$, (b) $\text{AMC-}^{15}\text{C}_{\text{coarse}}$.

Fine particles offer more surface area of the reinforcement for a given volume fraction so lesser interparticle spacing is available for dendritic growth. This results in smaller size dendrite. Fine size particles with more surface area also exhibit good bonding strength with matrix [29]. Moreover, the surface reactivity of particle at the interface also helps in binding the particles. Finer particle size exhibit improved mechanical property and wear resistance because the particle-matrix interface area is less for small particle at a given particle size, it results in better bonding between matrix and reinforcement than in case of coarse reinforcement. In case of coarse particle interface also provide the sites for crack nucleation more readily as increased interface area can have some area of improper interfacial bonding. Addition of coarse size particles also improves the hardness of the matrix but increase is less as compared to fine size particles. Increased hardness of the interface can also be due to the strain energy at the periphery of the particles dispersed in the material. As is evident from the Table 4.7, the hardness value increased with the increase in weight fraction and decrease in size of ZrSiO₄ particulates in AMC.

Table 4.7: Variation of hardness at different phases in AMCs.

Composite	Microhardness [Hv]		
	At matrix	At interface	At particle
AMC- ^{2.5} C _{fine}	80±0.5	119±0.5	705±0.5
AMC- ^{2.5} C _{coarse}	77±0.5	120±0.5	705±0.5
AMC- ⁵ C _{fine}	81±0.5	121±0.5	706±0.5
AMC- ⁵ C _{coarse}	78±0.5	119±0.5	702±0.5
AMC- ^{7.5} C _{fine}	86±0.5	122±0.5	710±0.5
AMC- ^{7.5} C _{coarse}	81±0.5	119±0.5	705±0.5
AMC- ¹⁰ C _{fine}	88±0.5	127±0.5	711±0.5
AMC- ¹⁰ C _{coarse}	85±0.5	122±0.5	704±0.5
AMC- ^{12.5} C _{fine}	92±0.5	133±0.5	718±0.65
AMC- ^{12.5} C _{coarse}	90±0.5	129±0.5	710±0.65
AMC- ¹⁵ C _{fine}	96±0.5	138±0.5	718±0.65
AMC- ¹⁵ C _{coarse}	94±0.5	131±0.5	709±0.65

4.6. Analysis of Wear Behavior of the LM13 Alloy and AMCs

4.6.1 Analysis of Wear Behavior of the LM13 Alloy

A progressive loss of material from the operating surfaces as a result of relative motion is known as wear. In order to get an idea about the durability of the materials prepared under different conditions were tested for dry sliding wear conditions. The effect of the applied load on the wear

behaviors of LM13 alloy was studied at varying loads in the range of 9.8 N- 49 N. Wear rate of LM13 base alloy as a function of sliding distance at variable loads from 9.8 N to 49 N is shown in Fig. 4.27. At a particular load, two types of wear behavior are displayed by the base alloy during the dry sliding. In the initial stages of run, the abrasive wear between the two surfaces in relative motion is dominant. The abrasive wear is controlled by the asperity to asperity contact of the two surfaces. The pin specimen of the composite and the hard steel counter surface contains large asperities of different height, shape and sharpness [30]. Some of the protruded asperities of the hard surface may penetrate into the softer pin. Subsequent sliding due to the reciprocating motion leads to the removal of material. The abrasive wear is accompanied by the formation of thin and shallow grooves on the specimen. The initial stage of run has shown very heavy wear loss due to the statistical fluctuations in wear. The continuous grinding of these abrasive particles while sliding reduces the sharpness of the asperities.

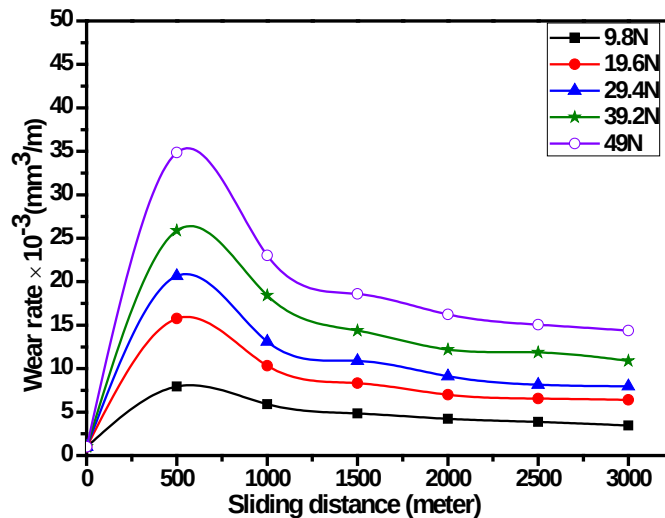


Figure 4.27: Wear rate of LM13 base alloy against sliding distance at different loads.

These blunt shaped smooth abrasives cause fall in wear loss and the steady state is attained. Similar type of wear behavior is also observed by Kumar et al. [31] and Chaudhary et al. [32]. Quinn et al. [33] suggested that worn surface is usually covered with a film of oxide. When two such surfaces slide against each other, oxide film breaks and explore new contact between the both surfaces. When contact pressure is high (49N), the solid layer may fracture and expose the substrate material, particularly if the substrate beneath the solid layer deforms plastically. Small regions of both substrate materials may finally come into contact and weld together thermo-mechanically. After some sliding, there will be a layer of turbulently mixed substances, some of which will fall

out as wear debris, but most of which will remain as a transfer film. The continuous increase in wear rate is observed (Fig. 4.27) with the increase in load from 9.8N to 49N. Application of high load of 49N causes the abrupt change in the wear rate which is evident when the wear rates of 9.8N and 49N shown in Fig 4.27 are compared.

4.6.2 Analysis of Wear Behavior of the AMCs

Figure 4.28 (a-f) shows the wear behavior of the composites containing 2.5, 5, 7.5, 10, 12.5 and 15 wt.% of fine size (1-32 μm) zircon sand particles with variation in the applied loads from 9.8 N to 49 N respectively. An increment in wear rate was observed with increase in applied loads for all the composites (Fig. 4.28(a-f)). The wear rate is affected with increased amount of reinforcement varying from 2.5wt.% to 15wt.%. In AMC-^{2.5}C_{fine}, the particle to particle distance in the matrix is very large as compared to AMC-¹⁵C_{fine}. The load transfer rate from particle to particle is generally dependent upon the particle to particle distance in the matrix. At this condition load transfer rate is low in AMC-^{2.5}C_{fine}. **The large distance between particles to particle in the matrix increased the wear rate due to the higher linear thermal expansion coefficient of matrix phase. Because, low load transfer rate from matrix to particle is responsible to increase the frictional heat between the counter surfaces during wear test and thermal expansion coefficient of matrix [34]. The frictional heat softens the matrix phase which is responsible to increase the wear rate of AMC-^{2.5}C_{fine} at higher applied load. The difference of coefficient of thermal expansion between matrix/reinforcing phase makes it easier to nucleate cracks at the interface with increased load resulting in increased wear rate.** The wear rate of AMC-^{2.5}C_{fine} shows that steady state wear at lower load is approachable after 1500 m sliding distance, while in AMC-⁵C_{fine} as shown in Fig. 4.28b steady state wear is achieved after sliding distance of 1250 m at low loads whereas on high loads the steady state wear is approachable at 2000 m sliding distance. For comparison the wear rate of all the composites at different loads is shown in Fig. 4.28(c-f). Figure 4.28(c-f) shows the wear rate of AMC-^{7.5}C_{fine}, AMC-¹⁰C_{fine}, AMC-^{12.5}C_{fine} and AMC-¹⁵C_{fine} at different loads. All composite samples show similar wear behavior in both states (run-in wear and steady state wear). The wear follows a linear relationship with respect to increasing load which portrays Archard's law of adhesive wear for metals. It clearly shows that all AMC exhibit similar curves which reveal two different types of wear behavior under an applied load. The steep rise in the initial stage gives rise to higher wear corresponding to the run in wear [34].

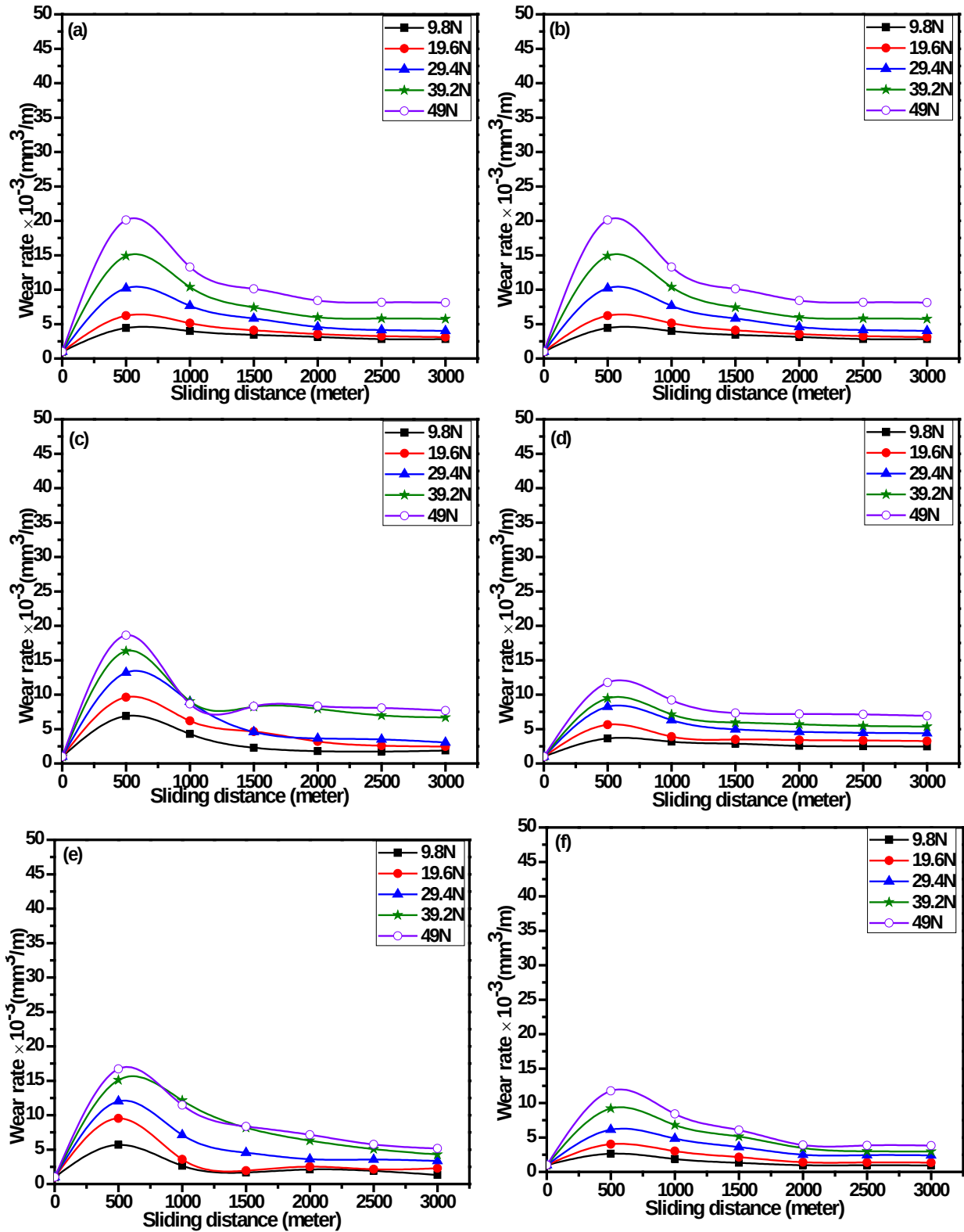


Figure 4.28: Wear rate against the sliding distance at different applied loads for AMC reinforced with fine size (1-32 μm) particles (a) AMC- $^{2.5}\text{C}_{\text{fine}}$, (b) AMC- $^{5}\text{C}_{\text{fine}}$, (c) AMC- $^{7.5}\text{C}_{\text{fine}}$, (d) AMC- $^{10}\text{C}_{\text{fine}}$, (e) AMC- $^{12.5}\text{C}_{\text{fine}}$ and (f) AMC- $^{15}\text{C}_{\text{fine}}$.

However, steady state wear is obtained in the later stage. Moreover, with increase in load the run-in and steady state wear rate are also found to increase. Almost similar trends of variation in wear behavior are observed for different applied loads. The tribological behavior of AMC can be associated with their viscoelastic and temperature-related properties [35]. Sliding contact of two counter surface results in heat generation at the asperities hence increase in temperature at the frictional surfaces of the two materials which influence the viscoelastic property in the response of materials stress, adhesion and load transferring behaviors of AMC [36]. These effects are prominent when the sliding distance is kept in the range of 500-1700 m at all applied loads as shown in Fig. 4.28(a-f). However, with increase in the amount of ZrSiO₄ the particles to particle distance in the matrix decreases. The contact area and load transfer rate increases with decrease in particle to matrix to particles distance in the matrix phase. It results in lower frictional heat generation between counter surfaces. Thus the lower frictional heat leads to improvement in strength of materials at contact and reduces the wear rate of AMC-¹⁵C_{fine}. AMC-¹⁵C_{fine} shows better wear behavior in comparison to the other composites having different amount with fine size particles (Fig. 4.28f).

Figure 4.29(a- f) shows the variation in wear rate with sliding distance at different applied loads for the composites having 2.5, 5, 7.5, 10, 12.5 and 15wt. % of coarse size (106-125 µm) ZrSiO₄ particles, respectively. All the composites have shown similar type of behavior under the loading conditions as observed from the Fig. 4.29(a-f). It is found that the wear rate goes on decreasing with the increase in ZrSiO₄ particle content for all the applied loads. Addition of hard ZrSiO₄ particles into the matrix reduces the wear rate. Hard asperities of the composites protect the matrix from the abrasive action of the asperities of the counter surface [30]. So the initial run in wear was found to decrease with the increase in amount of ZrSiO₄ particles in the AMCs. Steady state wear has also shown a significant improvement in wear rate with respect to increased amount and applied loads. All these curves show similar trend in run-in-wear state and steady-state-wear. In the case of higher applied load, the frictional heat is generated between the counter surfaces of AMC. This frictional heat produces a tribo-oxide layer by reaction with (i) environment (moisture); (ii) oxide of metals; (iii) deposited self-lubricating material readily oxidizes in air, so that, at initial duration of rubbing, the tribo-oxide layer easily separates the two contact material surfaces [37].

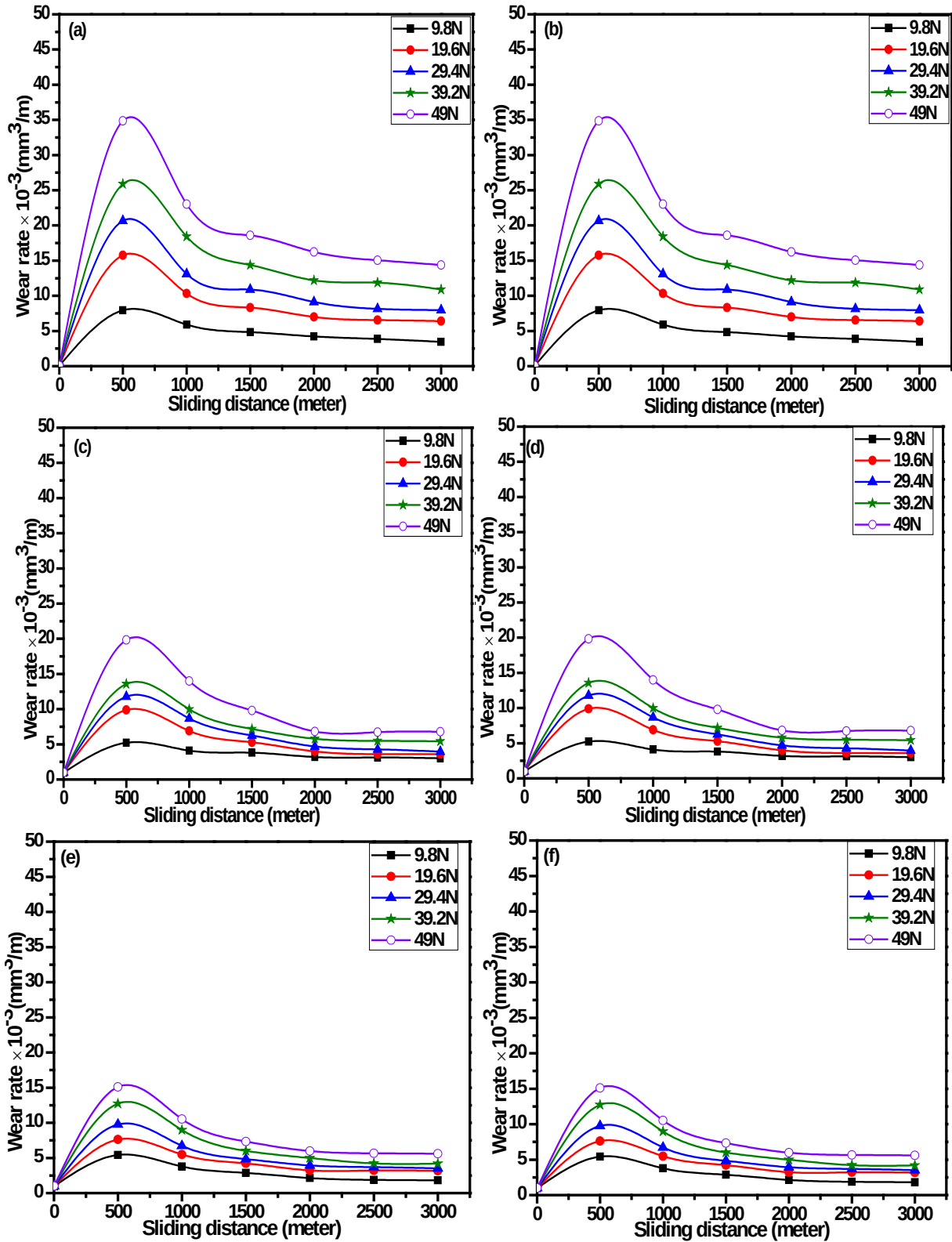


Figure 4.29: Wear rate against the sliding distance at different applied loads for AMC reinforced with coarse size (106-125 μm) particles (a) AMC- $^{2.5}\text{C}_{\text{coarse}}$, (b) AMC- $^5\text{C}_{\text{coarse}}$, (c) AMC- $^{7.5}\text{C}_{\text{coarse}}$, (d) AMC- $^{10}\text{C}_{\text{coarse}}$, (e) AMC- $^{12.5}\text{C}_{\text{coarse}}$ and (f) AMC- $^{15}\text{C}_{\text{coarse}}$.

During initial rubbing, the tribo-oxide layer breaks up and clean surfaces come in contact which increases the bonding force between the contacting surfaces. At the same time inclusion of trapped wear particles and roughening the substrate, the friction force increases due to which the ploughing effect increases. Increase of surface temperature due to frictional heat, viscous damping of the friction surface increases the adhesion due to micro-welding or deformation. Similar trends of results are shown in Fig 4.29(a-f). The tribo-oxide layer production rate is increased with increased amount of $ZrSiO_4$ particles and applied loads.

The wear rate of fine and coarse particle reinforced composites measured at different loads at room temperature is shown in Fig. 4.28 and 4.29, respectively. It depicts better wear resistance of composite reinforced with fine particles at all loads under dry sliding wear condition. It is observed in Fig. 4.28 and 4.29 that wear behavior of the composites is similar at room temperature for all loads. However, the wear rate of both type composites ($AMC-C_{fine}$ and $AMC-C_{coarse}$) increases with the increase in applied load which may be due to the increase in the actual contact area between composite sample and disc. Initially the wear rate is higher which corresponds to the run in wear.

However, the steady-state wear approaches at nearly 2000 m sliding distance. Run-in wear of composite with coarse particle is higher as compared to composite with fine particles at all loads as shown in Fig. 4.28 and 4.29. This indicates that fine particle reinforced composite exhibits better wear resistance is compared to coarse particle reinforced composites. **Fine particle size exhibit improved mechanical property and wear resistance for two major reasons. First, as the particle-matrix interface area is less for small particle at a given particle size, it results in better bonding between matrix and reinforcement than in case of coarse reinforcement. In case of coarse size particle interface provided sites for crack nucleation more readily as increased interface area can have some area of improper interfacial boundary. Secondly, in case of finer particles, dislocations go through them by leaving a dislocation loop around it and this results in improved work hardening.** These results are in good agreement with the earlier reported results [38, 39].

The results of the composites presented in Fig. 4.28 and 4.29 also show the initial stage variation which indicate towards grinding of asperities on the contact sample surface as indicated by the high wear rate with respect to increased loads. Large effective surface area of the fine particles in the matrix is also responsible for the better wear resistance of $AMC-C_{fine}$ in comparison to $AMC-$

C_{coarse} . The shape of the zircon sand particles also plays an important role for the wear behaviour of the composites. The sharp edge particles (fine) may also get inserted easily in the matrix during the applied load as compared to the coarse particles. Since, the coarse particles at these conditions are protruded so they may get fractured and increase the wear rate of the material. Material removal in composites is due to the indentation and ploughing action of the sliding indenters (reinforced particles) [40]. The hardness of the material determines the depth of the indentation of the abrasive particles. The hardness of material increases due to the presence of hard ceramic particles. Incorporation of hard zircon particles in the composites restricts such ploughing action of sliding indenters and improves the wear resistance.

4.6.3 Analysis of Wear Tracks of the LM13 Alloy and AMCs

The effects of the applied load on the wear behaviors of LM13 alloy and the composites were studied at 9.8 N to 49 N loads. Figure 4.30(a-d) shows the SEM images of wear tracks and wear debris of the LM13 alloy collected at low (9.8 N) and high (49 N) loads. As shown in Fig. 4.28 almost similar type of wear behavior of the LM13 alloy with respect to sliding distance at different applied loads is observed. **However, the wear is changing from plasticity dominated wear to mild oxidational wear with increase in applied load.** The SEM images of the wear track and wear debris of base alloy collected at low (9.8 N) and high (49 N) loads are presented. Figure 4.30a shows the wear tracks of the base alloy after the wear test at 9.8 N load. Presence of grooves on the worn surface was observed running parallel to the sliding direction.

Figure 4.30b shows the deeper grooves with larger delaminated area on the worn surface collected at 49N load as compared to the Fig. 4.30a. Figure 4.30c shows the debris collected after the wear test of base alloy with 9.8 N load. Formation of the debris is due to the removal of material during testing at different conditions. Flakes type debris in Fig. 4.30(c & d) clearly indicates that adhesive wear occurs during wear test for the base alloy tested at low (9.8 N) and high (49 N) loads respectively. However, size and amount of debris increases at high load condition, which reveals higher wear rate of the material. Corrugated structure observed on the wear debris is due to the continuous rubbing of materials during the wear test.

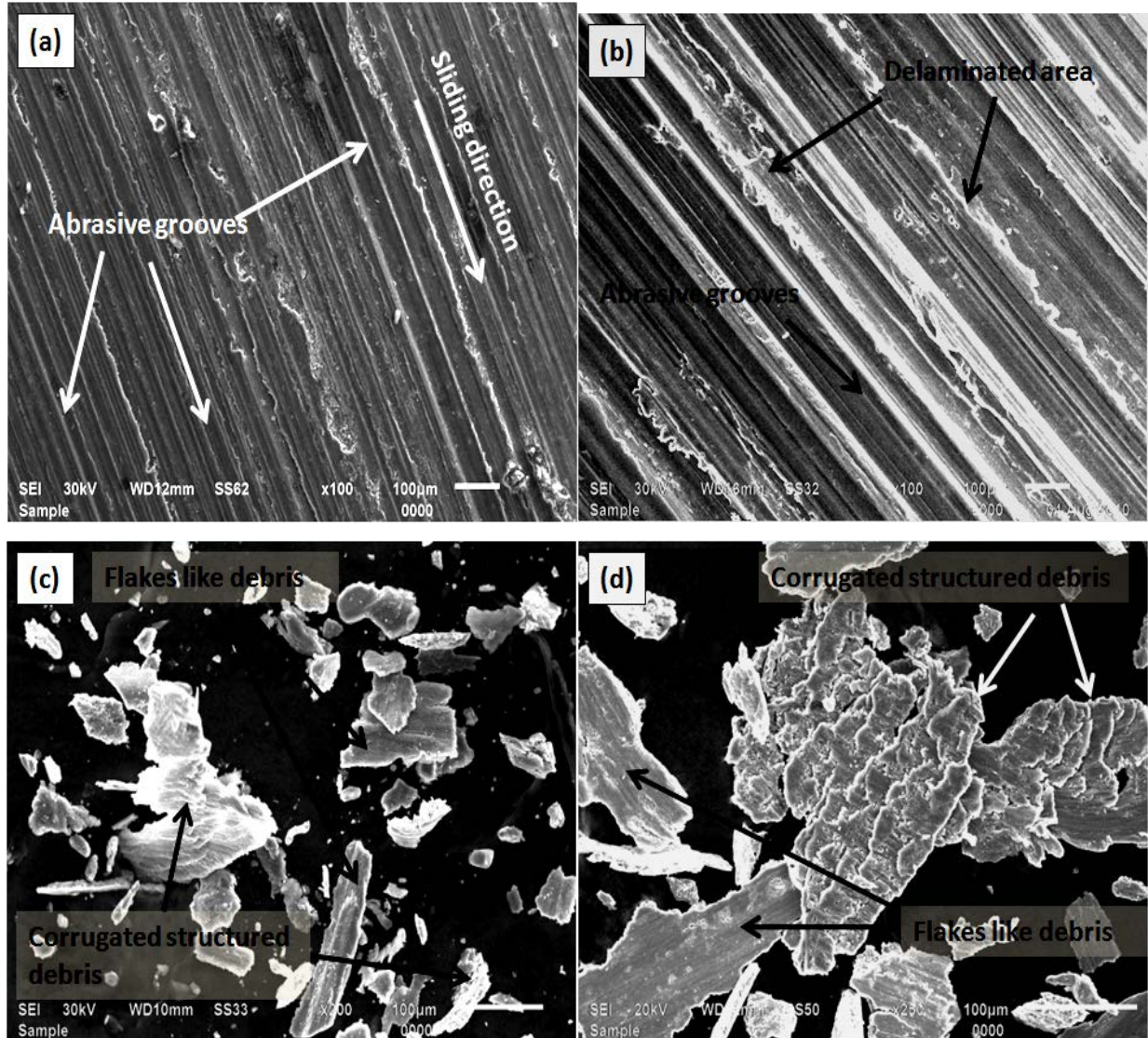


Figure 4.30: SEM images of LM13 alloy for (a) wear track at 9.8 N load, (b) at 49 N load (c) wear debris at 9.8 N load and (d) at 49 N load.

The SEM micrographs of wear track of AMC-¹⁵C_{fine} tested at different loads of 9.8- 49 N are presented in Fig. 4.31. Figure 4.31a shows the SEM micrograph of worn pin of AMC-¹⁵C_{fine} at 9.8 N load. The worn surfaces are smooth and ploughing strips are very shallow on the surface. At 49 N load material removal rate is significantly higher. Adhesive wear is dominating at this stage.

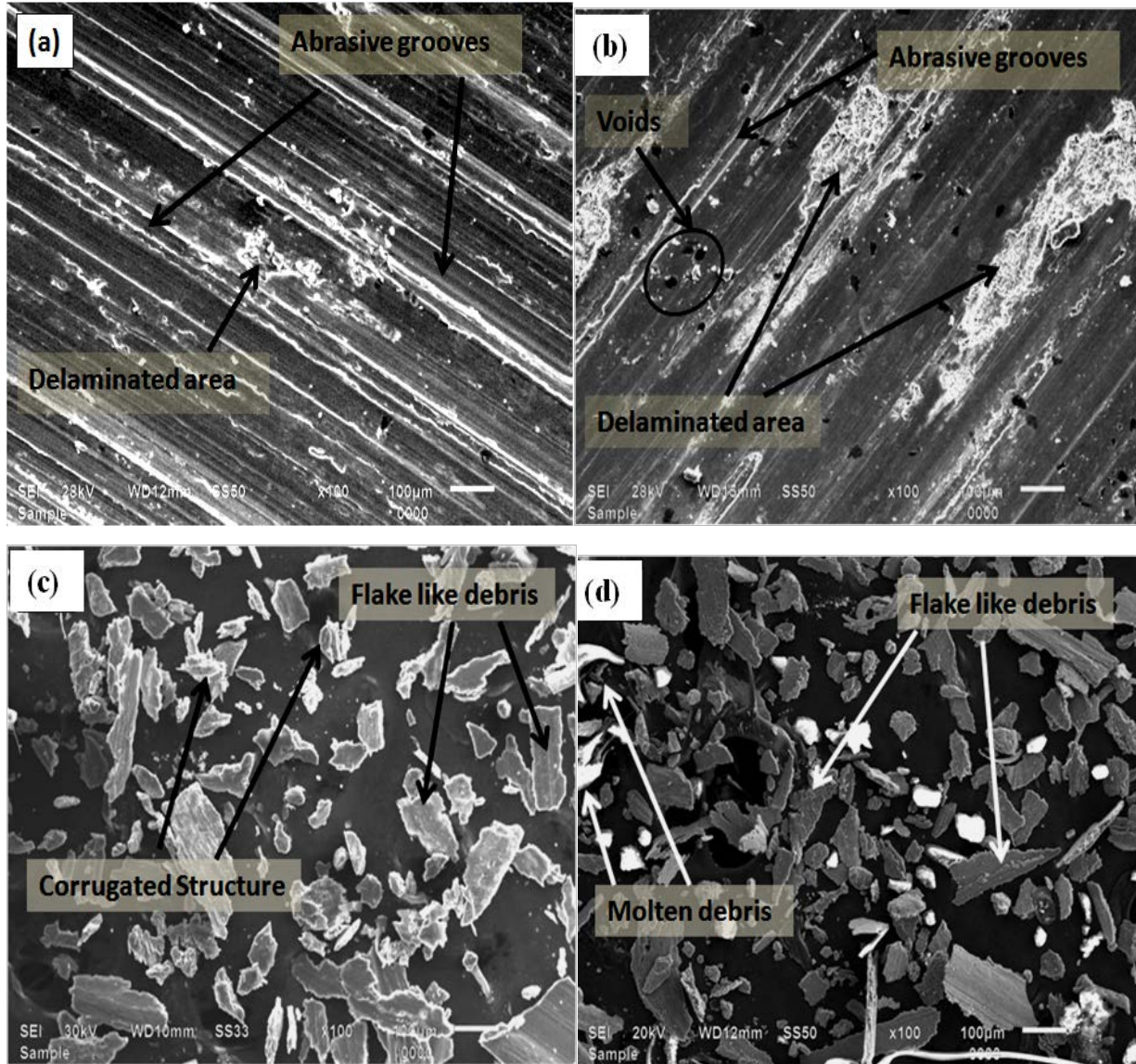


Figure 4.31: SEM images of AMC-¹⁵C_{fine} for (a) wear track at 9.8 N load, (b) wear track at 49 N load (c) wear debris at 9.8 N load and (d) wear debris at 49 N load.

Microcracks result delamination, which in turn damage the parent material by excessive material loss. At low (9.8N) load condition flake type delaminated metallic debris along with some debonded ZrSiO₄ are generated which indicates that adhesive wear dominates in this condition as shown in Fig. 4.31c. Wear debris at 49 N load as shown in Fig. 4.31d. It shows the plate or flakes of alloy matrix and debonded zircon sand particles which get spheroidized as they are trapped in the track during sliding action. The debris particles are likely to act as the third-body abrasive particles and could be responsible for the higher wear rate.

4.7 Microhardness of AMFs

Measurement of microhardness of AMF has been carried out to study the effect of foaming temperature and blowing agent on the alloy matrix. Figure 4.32 shows the microhardness indentations taken from nodes and ligament regions.

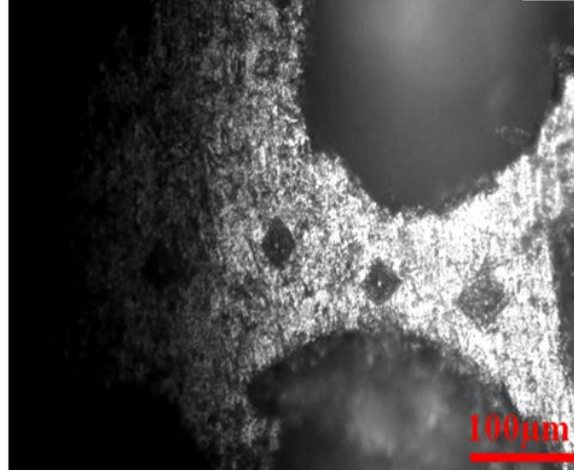


Figure 4.32: Optical micrographs showing different microhardness indentations on different zones of AMF-²F₉₀₀.

The variation in the size of the indentation in different zones (node and ligament) was observed. The effect of amount of blowing agent and foaming temperatures on the microhardness of AMF is not linearly uniform which is given in Table-4.8. AMF-²F₉₀₀ shows high microhardness value at matrix and nodes.

Table 4.8: Variation of microhardness of AMF samples (superscript shows amount of blowing agent and subscript for foaming temperature °C).

LM13 Alloy Foam	At Matrix	At Node
AMF- ^{1.5} F ₈₅₀ °C	69.49± 0.30	69.60± 0.25
AMF- ^{1.5} F ₈₇₅ °C	69.88± 0.25	70.10± 0.25
AMF- ^{1.5} F ₉₀₀ °C	70.12± 0.25	70.25± 0.25
AMF- ² F ₈₅₀ °C	69.67± 0.25	69.37± 0.25
AMF- ² F ₈₇₅ °C	68.34± 0.25	68.56± 0.25
AMF- ² F ₉₀₀ °C	70.58± 0.25	70.83± 0.25
AMF- ^{2.5} F ₈₅₀ °C	70.11± 0.25	70.19± 0.25
AMF- ^{2.5} F ₈₇₅ °C	69.44± 0.25	69.60± 0.25
AMF- ^{2.5} F ₉₀₀ °C	70.18± 0.25	70.35± 0.25

4.8 Analysis of Wear Rate of AMFs

4.8.1 Effect of applied load on wear behavior

The wear rate of AMF-^{1.5}F_{875 °C} at room temperature with different loads is presented in Fig. 4.33. It is apparent from figures that the wear rate nature of AMF-^{1.5}F_{875 °C} follows a similar trend upto 1250 m at all applied loads. For all the applied loads, the wear rate increases with increase in sliding distance in run in wear state (500 m) however, in steady-state conditions wear rate decreases. AMF-^{1.5}F_{850 °C} and AMF-^{1.5}F_{900 °C} shows the higher wear rate as compared to AMF-^{1.5}F_{875 °C} at different sliding distance (1000, 2000 and 3000m) which is shown in Table 4.9. The wear rate increases with increased cell wall thickness of the AMF and sliding distance which is shown in Table 4.9. At the higher load (49 N), AMF-^{1.5}F_{875 °C} shows the increased wear rate with sliding distance 1250 m to 2000m due to the plastically deformation of the cell wall. After 2000m sliding distance, wear rate of AMF-^{1.5}F_{875 °C} is nearly constant because at this condition the formation of the tribo-oxide layer which resist wear rate. The tribo-oxide layer production rate is increased with increased sliding distance and applied loads.

Table 4.9: Variation of wear of AMF with 1.5wt.% CaCO₃ samples for the load of 49N.

Sample ID	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (μm)	Wear Rate		
				1000m	2000m	3000m
AMF- ^{1.5} F _{850 °C}	0.95 ± 0.05	2.5 ± 0.10	1500 ± 100	47 x 10 ⁻³	38 x 10 ⁻³	33 x 10 ⁻³
AMF- ^{1.5} F _{875 °C}	0.65 ± 0.05	2.8 ± 0.10	1100 ± 100	33 x 10 ⁻³	36 x 10 ⁻³	35 x 10 ⁻³
AMF- ^{1.5} F _{900 °C}	0.80 ± 0.05	2.3 ± 0.10	1400 ± 100	43 x 10 ⁻³	36 x 10 ⁻³	29 x 10 ⁻³

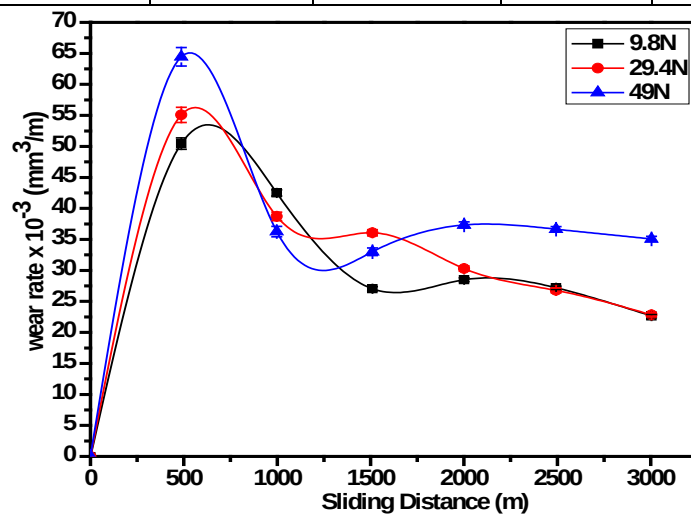


Fig. 4.33: Wear rate of AMF-^{1.5}F_{875 °C} against sliding distance at different loads.

Figure 4.34 represents the variation in the wear rate as a function of sliding distance for AMF-²F₈₇₅ °C under dry conditions. Increase in the sliding distance above 1500 m leads to the decrease in the wear rate of AMF-²F₉₀₀ °C. For the same dry sliding wear conditions, AMF-²F₉₀₀ °C shows marginally higher wear rate in comparison to that of AMF-^{1.5}F₉₀₀ °C. AMF-²F₉₀₀ °C has better wear resistance as compared to other AMF samples viz. AMF-²F₈₅₀ °C and AMF-²F₈₇₅ °C (Table 4.10).

Table 4.10: Variation of wear rate of AMF with 2wt.% CaCO₃ samples for the load of 49N.

Sample ID	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (μm)	Wear Rate		
				1000m	2000m	3000m
AMF- ² F ₈₅₀ °C	0.62 ± 0.05	2.2 ± 0.10	150 ± 25	57 x 10 ⁻³	42 x 10 ⁻³	39 x 10 ⁻³
AMF- ² F ₈₇₅ °C	0.90 ± 0.05	1.8 ± 0.10	300 ± 25	49 x 10 ⁻³	39 x 10 ⁻³	33 x 10 ⁻³
AMF- ² F ₉₀₀ °C	0.90 ± 0.05	1.8 ± 0.10	300 ± 25	33 x 10 ⁻³	39 x 10 ⁻³	31 x 10 ⁻³

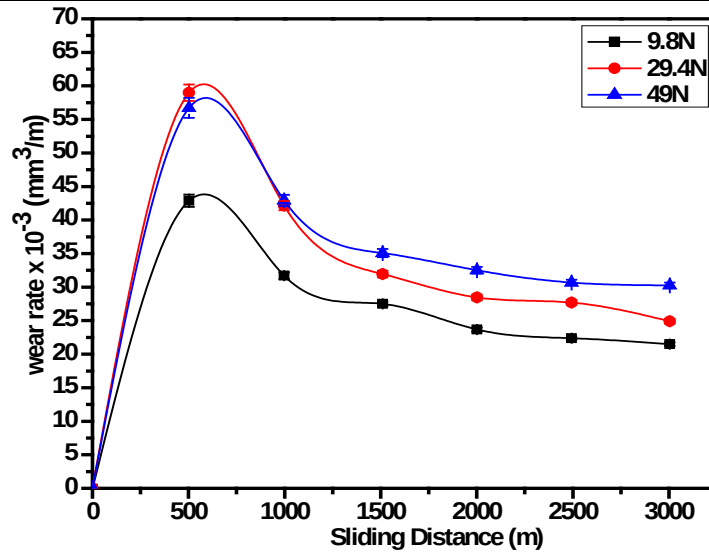


Figure 4.34: Wear rate of AMF-²F₉₀₀ °C against sliding distance at different loads.

It is observed from Fig. 4.35 that wear rate remains almost unchanged up to applied load of 9.8N and then increases sharply when load increased from 29.4N to 49N. This indicates transition in wear mechanism when the applied load is increased [41]. This could be due to several reasons. However, the domination factor is cell structure where cell acts as a crater which reduces the nominal contact [36]. Once initial transition period (run in wear) comes to an end the wear rate of the AMF-^{2.5}F₉₀₀ °C remains nearly constant. However, with the increase in applied load from 9.8 N to 29.4 N, wear rate of AMF-^{2.5}F₉₀₀ °C is increased. There is a sharp change in wear rate at high load

(49 N) with respect to low load (9.8 N and 29.4 N). During wear when two contact surfaces slide against each other, there will be some squeezing out phenomenon of the adsorbed films. When the applied load is increased from 9.8 N to 49 N, contact pressure on the ligament and node becomes higher, the cell may fracture and some plateau material slide over the cell. After some sliding, there will be a layer of turbulently mixed substances, some of which will fall out as wear debris, but most of which will remain as a transfer film. The contact area between counter surface and foam sample surface depends on the cell wall thickness of the AMF-^{2.5}F₉₀₀°C. The contact area is increased due to increase in the cell wall thickness of the AMF-^{2.5}F₉₀₀°C and wear rate is decreased. Therefore, the load transfer phenomenon depends on the contact area and the deformation rate of the cell wall of the foam sample, which is responsible to decrease the wear rate of the AMF-^{2.5}F₉₀₀°C.

Table 4.11: Variation of wear of AMF with 2.5 wt.% CaCO₃ samples for the load of 49N.

Sample ID	Density (gm/cm ³)	Average Cell Size (mm)	Cell Wall Thickness (mm)	Wear Rate		
				1000m	2000m	3000m
AMF- ^{2.5} F ₈₅₀ °C	0.51 ± 0.05	2.3 ± 0.10	0.80 ± 0.05	57 x 10 ⁻³	42 x 10 ⁻³	39 x 10 ⁻³
AMF- ^{2.5} F ₈₇₅ °C	0.45 ± 0.05	2.5 ± 0.10	0.30 ± 0.05	48 x 10 ⁻³	41 x 10 ⁻³	34 x 10 ⁻³
AMF- ^{2.5} F ₉₀₀ °C	0.95 ± 0.05	3.1 ± 0.10	1.35 ± 0.05	35 x 10 ⁻³	42 x 10 ⁻³	38 x 10 ⁻³

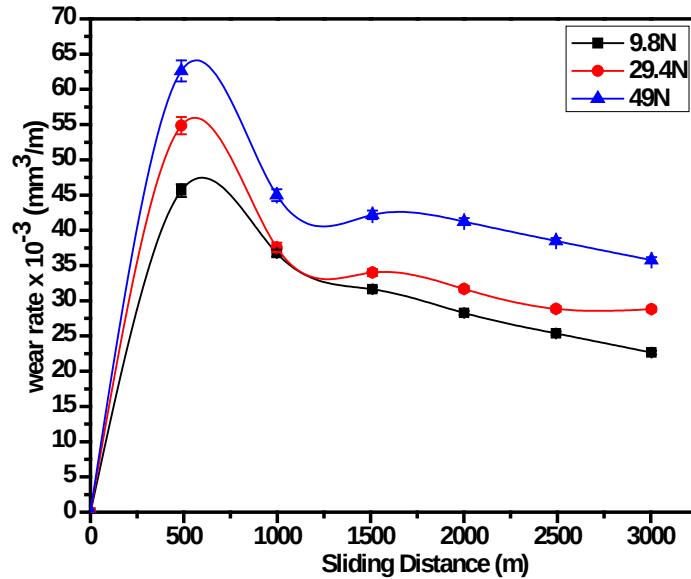


Figure 4.35: Wear rate of AMF-^{2.5}F₉₀₀°C against sliding distance at different loads.

The wear behavior of AMF (2.5% blowing agent) at different applied load condition is presented in Fig. 4.35. It is evident from the Fig. 4.35 that there is a rise in wear rate at the initial stage. This may be due to the adhesive nature of wear at the initial stage [34]. Once the initial transition period

(run in wear) comes to end, the wear rate of the material decreases, and a steady-state value of wear rate is attained. However, with the increase in the applied load from 9.8 N to 49 N, the wear rate of AMF increases. There is a sharp change in wear rate at high load (49 N) with respect to low load (9.8 N and 29.4 N). More material removal during the wear test at higher load is observed.

4.8.2 Analysis of Wear Tracks and debris of the AMFs

Figure 4.36(a-c) shows the SEM images of the worn pin surface of AMF- $^{2.5}F_{900}^{\circ C}$ with different loads. Figure 4.36a shows the worn surface of AMF- $^{2.5}F_{900}^{\circ C}$ tested at 9.8N load, where formation of continuous wear grooves along with some damaged regions on the ligament and node can be seen.

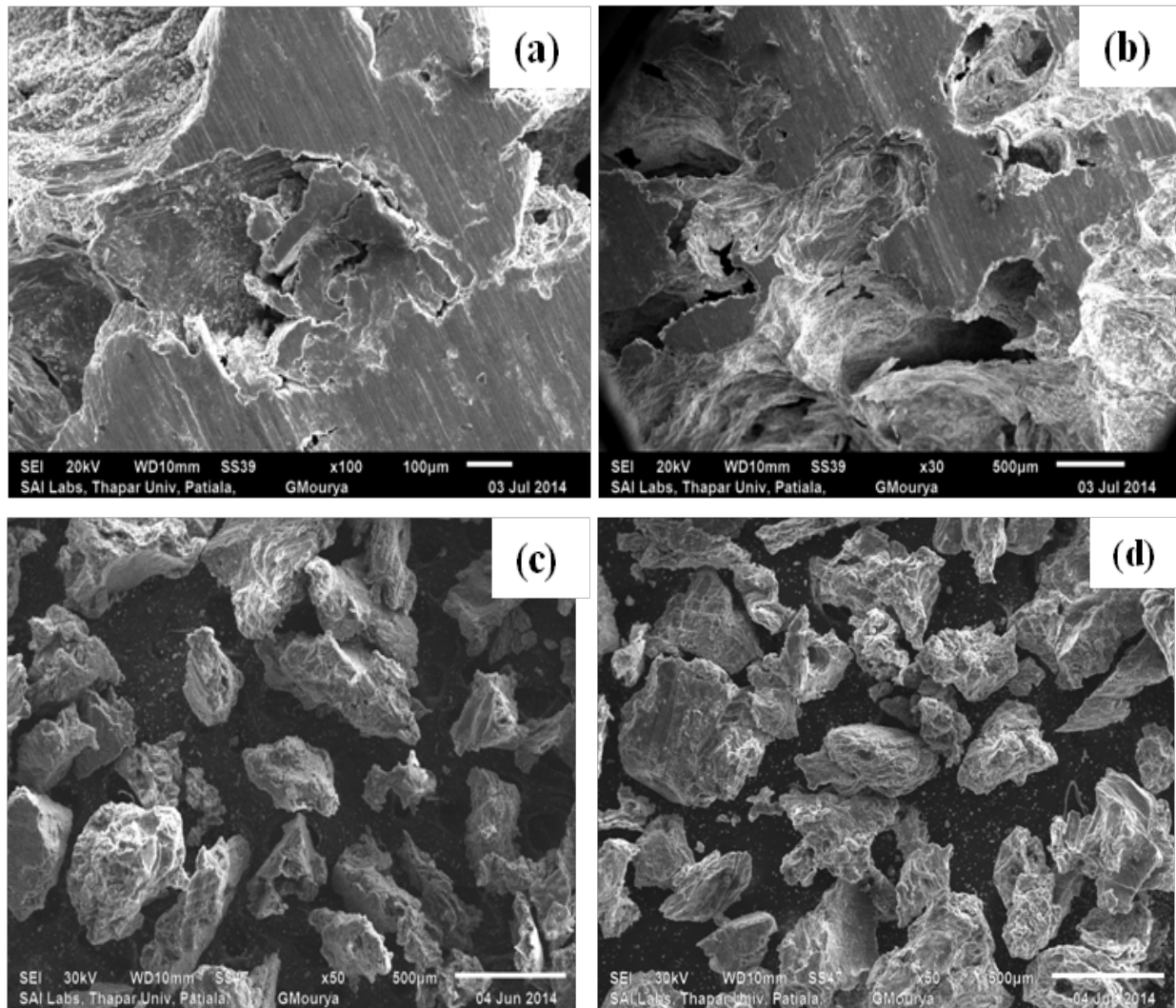


Figure 4.36: SEM images of wear track of AMF- $^{2.5}F_{900}$ at (a) 9.8 N (b) 49 and wear debris at (c) 9.8N (d) 49 N load.

Flow of materials along the sliding direction, generation of cavities due to delamination of surface material and tearing of surface materials can be seen as shown in Fig. 4.36a. Figure 4.36b shows the wear track of matrix tested at 49 N load with greater depth of grooves and more delaminated material moving inside cell because edges of cells are easily deformed. At 49 N load, thinner cell and node is easily deformed during sliding motion which is responsible to increase the wear rate of AMF-^{2.5}F₉₀₀. Majority of rough spherical debris have number of cracks due to repetitive stress at 9.8 N load. At this load, the dominant wear mechanism is severe plastic deformation as shown in Fig. 4.36c. At higher load, the increment in size and amount of wear debris can be easily seen in Fig. 4.36 (c & d). It supports the observation of higher wear rate at higher load.

4.9 Microhardness of AMCFs

The measurement of microhardness of AMCFs has been carried out to study the effect of reinforced particulates with different size on the alloy matrix. Figure 4.37 shows the microhardness indentations at matrix phase, particle and interface on the ligament and nodes. The variation in the size of the indentation in different phases was observed. As given in the Table 4.12 the hardness value of AMCF increased with the weight fraction of ZrSiO₄ particulates. Increase in hardness as observed with the increased amount of ZrSiO₄ addition vary with the particle size in AMCs.

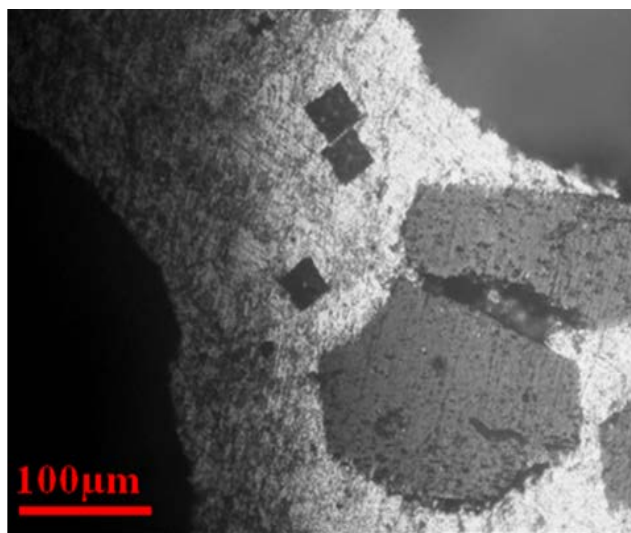


Figure 4.37: Optical micrographs showing different microhardness indentations on different phases of AMCF-¹⁵C_{fine} (2wt.% CaCO₃/900 °C).

Reinforced particles show higher hardness which decreases as we move away from particle (Fig. 4.37a). The high hardness at particle/matrix interface indicates good interfacial bonding between particle and alloy matrix. **Interaction of the ZrSiO₄ particles with the Al alloy matrix results**

to form harder phase Al_2SiO_2 leading better hardness at the interface. The formation of Al_2SiO_2 phase is observed in the XRD analysis (Fig. 4.1b). This phase at particle/matrix interface exhibits higher hardness in comparison to the matrix phase and also binds the particles with matrix [42].

In the AMCF, higher hardness at particle/matrix interface observed in the ligament and node exhibits higher hardness in comparison to the matrix phase. It also indicates good binding of particles with matrix. $\text{AMCF-}^{15}\text{C}_{\text{fine}}$ shows high microhardness value at particle, interface, and matrix.

Table 4.12: Variation of microhardness of different phases of AMCF samples (superscript shows amount of reinforcement and subscript for coarse and fine particles).

Microhardness (Hv)				
AMCF (with 2wt. % CaCO_3 /900 °C)	At Particle	At Matrix	At Interface	At Node
$\text{AMCF-}^{2.5}\text{C}_{\text{fine}}$	652.45± 0.20	78.54± 0.25	118.72± 0.30	78.91± 0.25
$\text{AMCF-}^{2.5}\text{C}_{\text{coarse}}$	650.55± 0.20	68.28± 0.25	117.34± 0.30	70.54± 0.25
$\text{AMCF-}^5\text{C}_{\text{fine}}$	667.22± 0.20	85.60± 0.25	130.92± 0.30	86.40± 0.25
$\text{AMCF-}^5\text{C}_{\text{coarse}}$	655.93± 0.20	80.88± 0.25	126.19± 0.30	78.10± 0.25
$\text{AMCF-}^{7.5}\text{C}_{\text{fine}}$	670.51± 0.20	86.56± 0.25	132.12± 0.30	88.79± 0.25
$\text{AMCF-}^{7.5}\text{C}_{\text{coarse}}$	666.45± 0.20	85.89± 0.25	130.78± 0.30	86.82± 0.25
$\text{AMCF-}^{10}\text{C}_{\text{fine}}$	666.74± 0.20	88.12± 0.25	133.67± 0.30	90.78± 0.25
$\text{AMCF-}^{10}\text{C}_{\text{coarse}}$	665.55± 0.20	87.78± 0.25	132.33± 0.30	88.46± 0.25
$\text{AMCF-}^{12.5}\text{C}_{\text{fine}}$	671.34± 0.20	89.34± 0.25	136.67± 0.30	91.55± 0.25
$\text{AMCF-}^{12.5}\text{C}_{\text{coarse}}$	664.98± 0.20	88.55± 0.25	134.54± 0.30	90.45± 0.25
$\text{AMCF-}^{15}\text{C}_{\text{fine}}$	672.22± 0.20	90.56± 0.25	139.12± 0.30	93.54± 0.25
$\text{AMCF-}^{15}\text{C}_{\text{coarse}}$	669.56± 0.20	88.34± 0.25	137.34± 0.30	92.12± 0.25

4.10 Analysis of Wear Rate of AMCFs

Surface roughness is affected by the amount and size of zircon sand which shows variation in wear rate of AMCF. The abrasive wear rate relates to the number of asperity contacts on the surface as the number of contact points is directly related to the contact area and applied load. Wear rate is linearly related to contact area and plastic deformation rate of asperities.

In all the study the steady state is attained after nearly 1300 m sliding distance. As the normal load is increased from 9.8 N to 49 N, the number of asperities engaged in ploughing is changed, and wear rate is increased which is shown in Fig. 4.38(a-f). The temperature rises between counter surfaces due to frictional heating especially in dry condition that leads to thermal expansion of matrix phase hence contact area between matrix and counter surface increases. In Fig. 4.38(a & f), it is observed that wear rate is higher for AMCF- $^{2.5}C_{fine}$ as compare to AMCF- $^{15}C_{fine}$ at different applied loads as the thermal expansion of AMCF- $^{2.5}C_{fine}$ is larger in comparison to AMCF- $^{15}C_{fine}$. During sliding, asperities get deformed plastically due to frictional heating leading to increase in contact area. Large effective surface area of the fine particles in the matrix is also responsible to increase the load transfer rate from soften phase (matrix) to harder phase (ceramic particle). This higher load transfer rate has provided better wear resistance of fine size particle reinforced composite foam in comparison to coarse size particle reinforced composite foam.

Generally, with applied increased load on the composite sample, wear rate increases in the similar pattern as has been observed by Das et al. [43]. During dry sliding, the oxide film which acts as a cover envelope to the metal surface finally breaks, thus bringing the two solid surfaces in contact [44, 45]. High pressure applied on specimen during sliding fractures the oxide film and lead to exposure of the substrate material causing plastic deformation beneath the surfaces. This close contact also leads to the welding of the materials at the edges of cells. However, some will be trapped within the cells also. The abrasive wear is controlled by the asperity to asperity contact of the two surfaces. The pin specimen of the composite foam and the hard steel counter surface contains large asperities of different height, shape and sharpness [33]. The height, shape and size of asperity and inter-asperity distance on the ligament and node of composite foam depends on the amount, the nature of distribution of reinforced particles, and their sizes in matrix with loads. The real area of contact is only a fraction of the apparent area of contact.

Kapoor and Johnson [46] proposed a theoretical analysis for the steady-state wear of two sliding asperities. They showed that the heights and curvatures during wear will change in such a way that the corresponding contact pressure drops to the shakedown limit and further sliding will involve no more plastic flow of asperities on the node of composite foam.

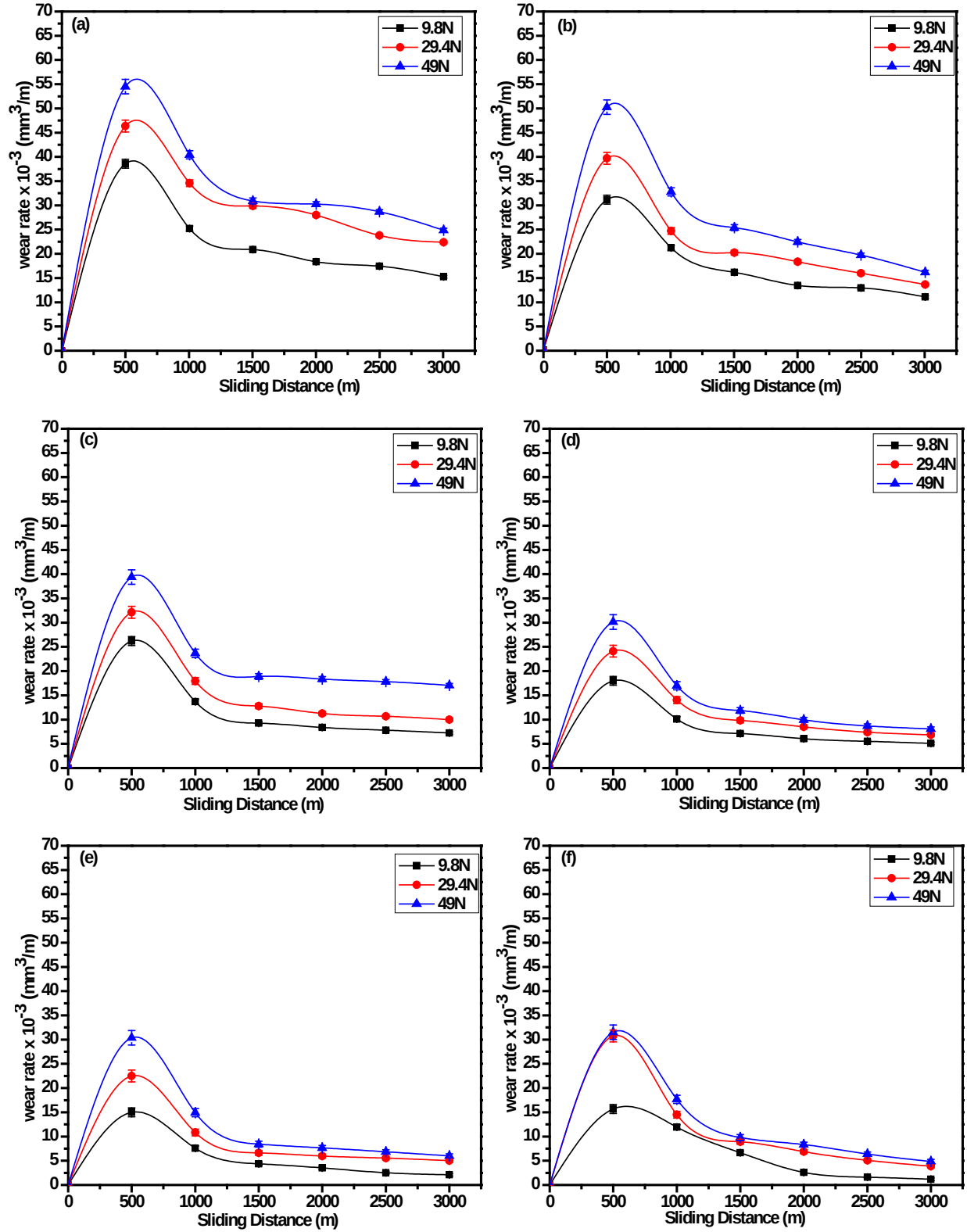


Figure 4.38: Wear rate against the sliding distance at different applied loads for AMCF reinforced with fine size (1-32 μm) particles (a) AMCF- $^{2.5}\text{C}_{\text{fine}}$, (b) AMCF- $^5\text{C}_{\text{fine}}$, (c) AMCF- $^{7.5}\text{C}_{\text{fine}}$, (d) AMCF- $^{10}\text{C}_{\text{fine}}$, (e) AMCF- $^{12.5}\text{C}_{\text{fine}}$ and (f) AMCF- $^{15}\text{C}_{\text{fine}}$.

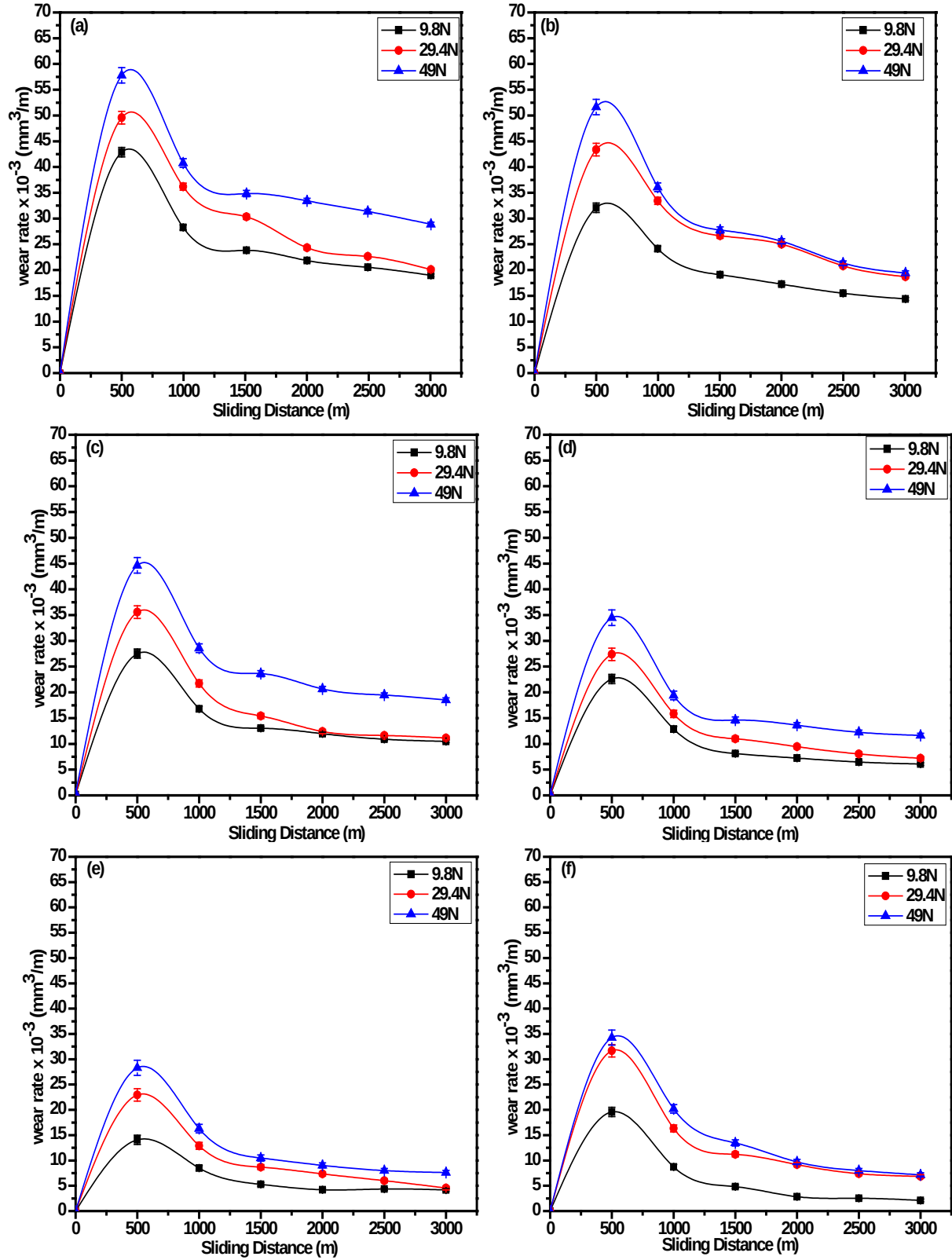


Figure 4.39: Wear rate against the sliding distance at different applied loads for AMCF reinforced with coarse size (106-125 μm) particles (a) AMCF-^{2.5}C_{coarse}, (b) AMCF-⁵C_{coarse}, (c) AMCF-^{7.5}C_{coarse}, (d) AMCF-¹⁰C_{coarse}, (e) AMCF-^{12.5}C_{coarse} and (f) AMCF-¹⁵C_{coarse}.

As the applied load is increased, the separation between surfaces decreases and more of the softer asperities undergo changes in height and shape by the passage of the highest hard asperities on the ligament and node of AMCF. The hard asperities (particles) will develop higher contact pressures as compared to lower neighbors. The number of asperities of fine size reinforced composite foams is higher than that of coarse size reinforced composite foams. But the size of asperity and inter-asperity distance on the ligament and node of composite foams with fine size reinforcement depends on the particle to particle distance. The fine size reinforced composite foams have minimum particle to particle distance as compared to coarse size reinforced composite foams. Though, the particle to particle distance decreases with increased amount of reinforcement in the developed composite foams, but the applied load is increased up to 49 N, so the separation between surfaces decreases and more of the softer asperities (due to frictional heat) undergo changes in height and shape by the passage of the highest hard asperities.

4.10.1. Analysis of Wear Tracks and Debris of AMCF

It is observed that the wear rate remains almost unchanged up to applied load of 9.8 N and increases sharply when load is increased from 29.4 to 49 N. This indicates transition in wear mechanism when the applied load changes from 9.8 N to 49 N which could be understood from examination of wear surface as shown in Fig. 4.40(a & b). Because of cell structure, the cell acts as a crater which leads to reduce the nominal contact. These craters help in accumulating the wear debris due to higher contact load and gets compacted within the craters [34]. Thus, the fraction of material removed from the surface gets reduced. The weak interface bonding between zircon particles and matrix in outer wall which are fragile leads to generation of very fine particles. These fine particles get mixed over the wear surface because of combined effect of frictional heating and surface plastic deformation followed by material flow. Finally, it makes a stronger mechanically mixed layer (MML) especially at higher applied load over the surface. At the higher load, during dry sliding the oxide film ($\text{Al}_2\text{O}_3/\text{Fe}_2\text{O}_3$) which acts as a cover envelope to the metal surface breaks, thus bringing the two solid surfaces in contact (Fig. 4.40b). During dry sliding condition, both the surfaces come to each other and contact area increased at higher pressure. At this condition, materials surface may be transferred and some of the which will fallout as wear debris in layer or multilayer form [33]. Developed oxide film can only protect the composite foam sample in the region of intermediate frictional temperature and load. During wear rapid removal and regeneration of the tribo-layer at the interface takes place.

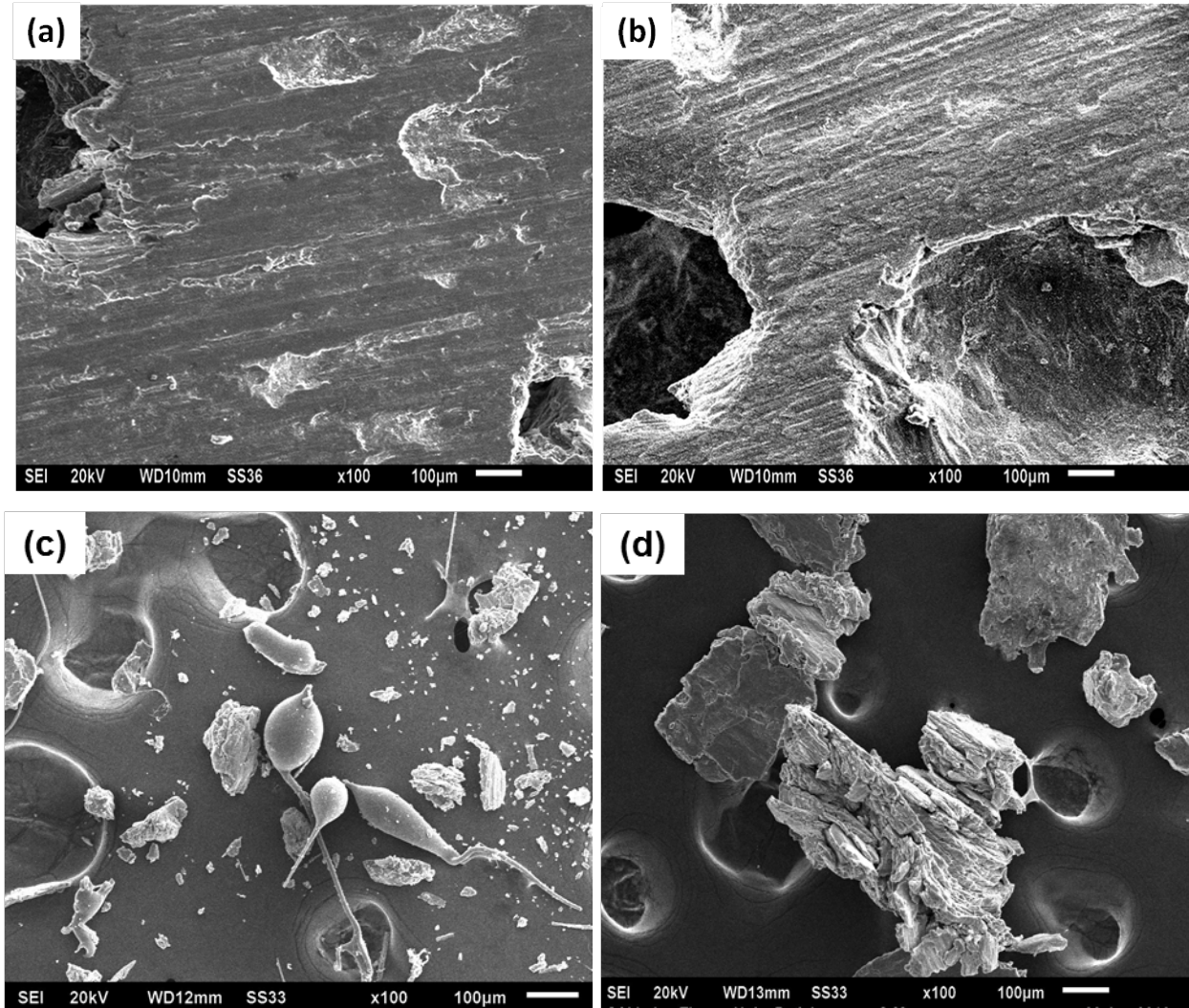


Figure 4.40: SEM images of wear track (a) AMCF-¹⁵F_{fine}, (b) AMCF-¹⁵F_{coarse}, and wear debris (c) AMCF-¹⁵F_{fine}, (d) AMCF-¹⁵F_{coarse} at 49N load.

The interface temperature also increases during the wear test and this causes oxidation of the sample. The increase of interface temperature during the wear test significantly enhances the loss of mass by oxidation, matrix softening, cracking and delamination wear. The temperature at the interface depends on the thermal boundary resistance of matrix phase and reinforced phase. Now assuming a constant heat flux, due to this interfacial thermal resistance, there exists a temperature difference between matrix phase and reinforced phase at their interface. This thermal boundary resistance is due to carrier scattering at an interface and it is known as interfacial thermal resistance. The interfacial thermal resistance depends on the difference in the coefficient of thermal expansion (CTE) between the matrix and the ceramic particles. The wear rate increased due to

loose interface bonding between particle and matrix. This carrier scattering is responsible to decrease the wear rate of AMCF. Molten metal balls are formed by the continuous rubbing of the thread like debris between the two surfaces which is shown in Fig 4.40c. Multilayered welded and twisted debris reveal the repetitive nature of stress that occurred at high load sliding conditions. Some debris with grooves is observed in Fig 4.40d which indicates that coarse particles have chipped out from matrix and this particle moves between contact surfaces in the sliding direction and generate grooves on the debris. Wear is governed by delamination which gives plate-like morphology of debris with microcracks [33].

4.10.2. EDS Analysis of Wear Tracks

During the sliding wear, some the ceramic particles have been pulled out or crushed as the pin moved over the disc, and acted as an abrasive medium. The crashed ceramic particles along with ferrous oxides from the counterface and aluminium oxides from the pin were transferred and embedded into surfaces.

The continuous grinding of these abrasive particles while sliding reduces the sharpness of the asperities. These blunt shaped smooth abrasives cause fall in wear loss and the steady state is attained. AMF having thinner cell wall thickness (lower real contact area) is main cause of high wear rate as compared to AMCF. This is due to fact that load transfer per unit area for the foam sample containing lower contact area is high, which leads to higher wear rate of the material. Composite foam samples having higher cell wall thickness introduce higher real contact area, which leads to lower load transfer per unit area and hence lower wear rate. The composite foams with fine particles have more number of particles distributed in the matrix which decreases the inter-particle distance; as a result, hardness of the composite foam increases as compared to the coarse particle composite foam which further reduces the wear rate. With the increase of zircon sand content with coarse grade from 2.5 to 15wt.%, almost similar type of variation is observed as shown in Fig. 4.39. At higher load (49 N), less change in wear gradient is observed in AMCF- $^{15}\text{F}_{\text{coarse}}$ compared to AMCF- $^{12.5}\text{F}_{\text{coarse}}$ which shows the enhanced stability provided by the higher concentration of zircon sand to the matrix. The higher contents of zircon sand in composite foams have shown the delayed transition in the wear. Even during plastic deformation, the variation in slope for fine zircon sand composite foam is less as compared to coarse zircon sand.

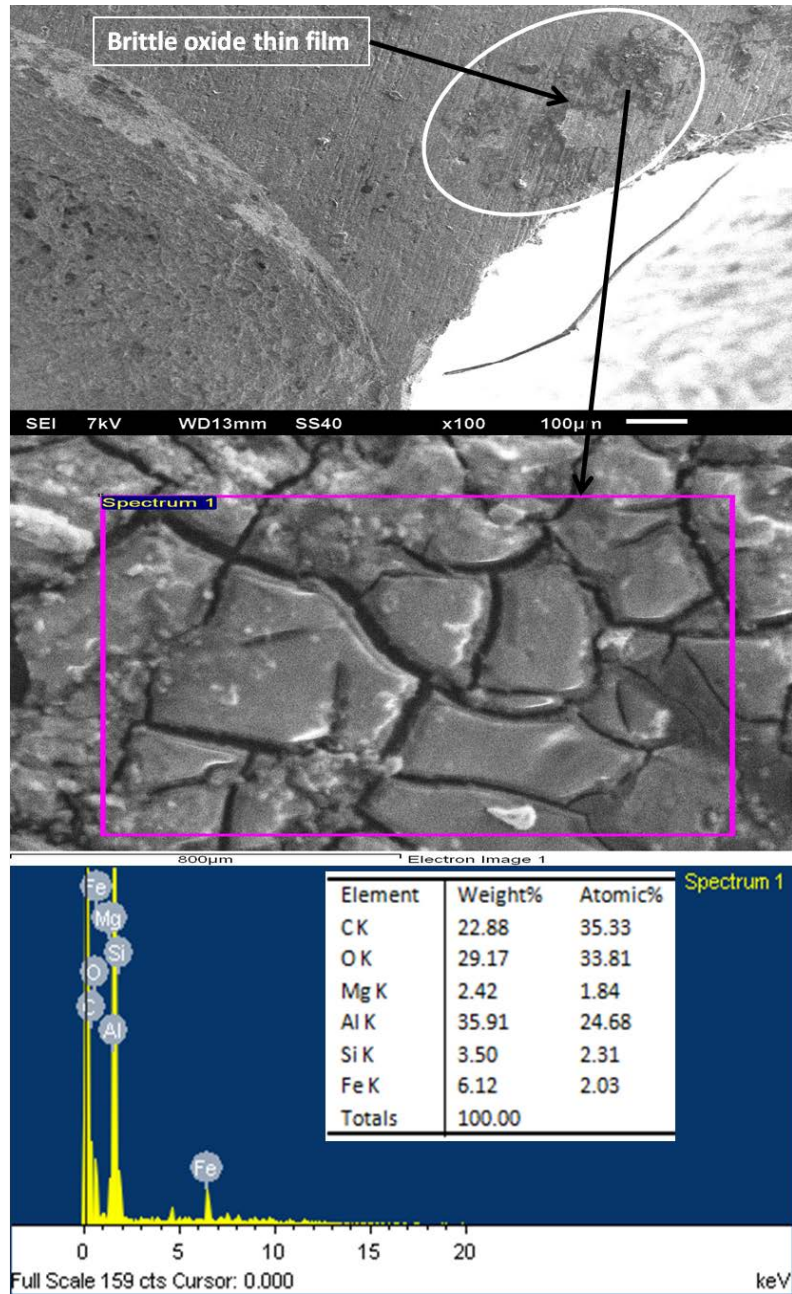


Figure 4.41: SEM-EDS images of brittle oxide thin film on the wear track of AMCF- $^{15}\text{F}_{\text{fine}}$ at 49N load.

This clearly indicates that composite foam with 15wt.% with fine zircon sand particles comes out to be a better wear-resistant foam material as compared to others AMCF materials. The mutual transfer of materials between the two counter surfaces resulted in development of mechanically mixed layer (MML) on test specimen [33]. MML improved the wear property by providing a hard covering between the mating surfaces [44]. Therefore, the zircon sand particles provided improved

wear resistance to the LM13 alloy composite foams than the unreinforced LM13 alloy foam by the formation of MML at the interface, which is verified using EDS and can be seen in Fig. 4.41. The increase in wear rate of LM13 alloy composite foam at higher applied loads may be due to increased pressure on the MML resulting in continuous deformation and work hardening of MML, which was accompanied with subsurface microcrack propagation [42]. The intersection of these cracks resulted in the detachment of sheet-like wear particles, leaving behind shallow craters. Thus, an increase in load weakened the existing MML by delamination wear mechanism of foam materials.

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CHAPTER 5

Blast Mitigation

Overview:

This chapter describes the details of the blast mitigation test of AMF and AMCF. The effective range of blast pressure depends on the density and cell geometry of the AMF and AMCF. Blast mitigation tests were performed on AMF and AMCF samples prepared by using different wt.% of blowing agent (for AMF) and ZrSiO_4 reinforcement (for AMCF). Blast mitigation trials were conducted using CE pellet having 35g of tetryl charge. The results of the blast tests done on developed foams in Terminal Ballistics Research Laboratory, Defence Research and Development Organization, Ramgarh, Chandigarh, India have been compiled and discussed.

5.1 Blast Mitigation Analysis:

5.1.1 Blast Mitigation Analysis of AMFs

The design of blast resistant structures require knowledge of the blast loading and the behavior of structures under these loadings. The explosion protection system consists of three components: (1) donor system (amount, type and location of explosive), (2) the acceptor system (personnel, equipment, and acceptor explosives), and (3) the protection system(protective structure, structural components or distance). The most important feature of blast resistant structure is the ability to absorb blast energy without causing catastrophic failure in the structure or injury to personnel or damage to equipment. Ductile material with longer plastic deformation is adequate for blast protection such as metal foams. The AMFs structure showed excellent pressure mitigation and reduced the transmitted pressure under certain level of its compressive strength [4]. However, the effective range of blast pressure depends on the relative density and cell geomatery of the AMFs and AMCFs. The relative density and cell geomatery of AMFs and AMCFs vary with manufacturing process parametes such as amount of blowing agent, type of reinorcement, their distribution during foaming and holding temperature. The results of blast pressure mitigation of AMFs with different density of the foams is given in Table-5.1.

Table-5.1: Blast Testing for AMCs.

Sample ID	Sample weight(gm)	Density (gm/cm ³)	Sample height (cm)	Transmitted pressure (Reflected) in bar	Percentage attenuation
Soil	-	-	-	1800	0.0
AMF- ^{1.5} F _{900 °C}	547.95	0.80 ± 0.05	7.3 ± 0.1	38 ±2	97.9
AMF- ² F _{900 °C}	530.44	0.59 ± 0.05	7.2 ± 0.1	60 ±2	96.7
AMF- ^{2.5} F _{900 °C}	505.15	0.52 ± 0.05	7.0 ± 0.1	72 ±2	96.0

As is evident from the data given in table, the initial pressure exerted on the bottom of foam for all samples is 1800 bar (soil). The pressure on other side of foam observed is 38, 60 and 72 bar for AMF-^{1.5}F_{900 °C}, AMF-²F_{900 °C}, and AMF-^{2.5}F_{900 °C}, respectively. Here the impact of foaming agent CaCO₃ which varies from 1.5 to 2.5wt.% has been studied.

Using different material models, extensive parametric analyses have been performed for various explosive conditions [2]. The open air-blast test was only considered as suitable one for present analysis for all conditions of AMFs and AMCFs. The typical results of AMF and AMCF is analyzed here.

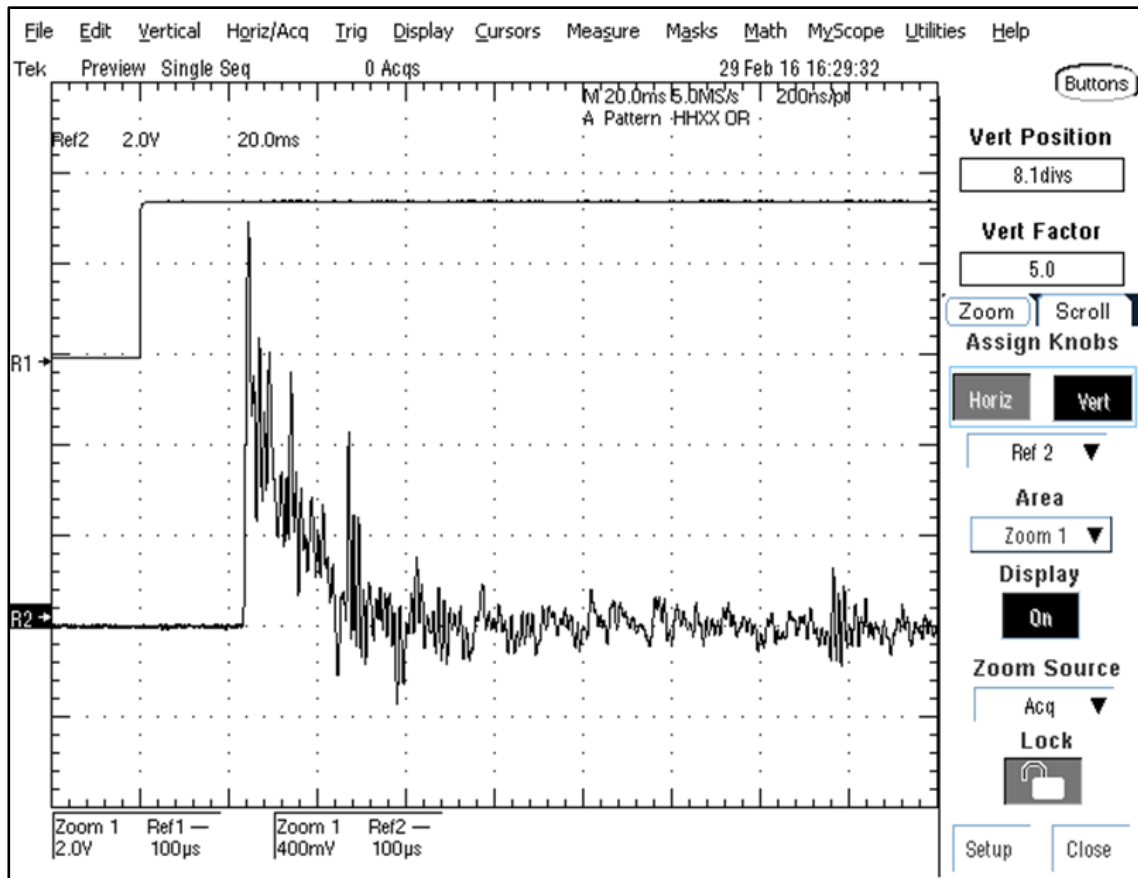


Figure 5.1: Pressure versus time graph of AMF-^{1.5}F_{900 °C}.

There are two factors which are important in this analysis. First is the pressure and second one is the percentage of transmitted pressure. Since the foaming temperature are kept constant so the influence of the amount of the foaming agent CaCO₃ is studied. Figure 5.1 shows a typical graph of test done for AMC-^{1.5}F_{900 °C} sample. It shows a considerable drop in pressure from 1800 to 38 bar whereas for AMF-²F_{900 °C} and AMF-^{2.5}F_{900 °C} it is 60 and 72 respectively.

The analysis of above results indicate that amount of blowing agent play an important role. It ultimately controls the density of foam and cell geometry. Closed cell foams having higher relative density interact effectively with the blast waves. Hanssen et al. [2] did blast tests to investigate the blast pressure mitigation by aluminum foam panels considering different scaled distances and relative density. Aluminum foams are more effective to reduce high overpressure by explosion as it shows large plastic deformation capacity [3-5].

5.1.2 Blast Mitigation Analysis of AMCFs

AMCF samples have different densities and cell geometry. The densities and cell geometry of AMCFs is affected by addition of different amount of reinforcement. The idea to add reinforcement was to strengthen the walls which can help in resisting the blast waves. The results of different samples tested are presented in Table-5.2. Some results of typical test are shown in Fig. 5.2 to 5.5.

Table-5.2: Blast Testing for AMCFs.

Sample ID	Wt. % ZrSiO ₄	Sample weight (gm)	Density (gm/cm ³)	Sample height (cm)	Transmitted pressure (Reflected) in bar	Percentage attenuation
Soil	-	-	-	-	1800	0.0
AMCF- ^{2.5} C _{fine-0.63}	2.5	528.92	0.65 ± 0.05	7.5 ± 0.1	210	88.3
AMCF- ^{2.5} C _{fine-0.63}	2.5	637.75	0.63 ± 0.05	7.1 ± 0.1	Not Recorded	-
AMCF- ⁵ C _{fine-0.51}	5.0	699.26	0.51 ± 0.05	7.3 ± 0.1	Not Recorded	-
AMCF- ⁵ C _{fine-0.51}	5.0	532.80	0.51 ± 0.05	7.3 ± 0.1	1300	27.2
AMCF- ^{7.5} C _{fine-0.86}	7.5	497.39	0.86 ± 0.05	7.5 ± 0.1	200	88.9
AMCF- ^{7.5} C _{fine-0.80}	7.5	774.56	0.80 ± 0.05	7.2 ± 0.1	105	94.2
AMCF- ¹⁰ C _{fine-0.70}	10.0	532.48	0.70 ± 0.05	7.3 ± 0.1	60	96.7
AMCF- ¹⁰ C _{fine-0.77}	10.0	564.23	0.77 ± 0.05	7.2 ± 0.1	50	97.2
AMCF- ^{12.5} C _{fine-0.54}	12.5	592.59	0.54 ± 0.05	7.5 ± 0.1	30*	98.3
AMCF- ^{12.5} C _{fine-0.44}	12.5	494.52	0.44 ± 0.05	7.0 ± 0.1	160	91.1
AMCF- ¹⁵ C _{fine-0.73}	15.0	755.36	0.73 ± 0.05	7.2 ± 0.1	130	92.8
AMCF- ¹⁵ C _{fine-0.70}	15.0	700.77	0.70 ± 0.05	7.2 ± 0.1	145	91.9
AMCF- ¹⁵ C _{fine-0.69}	15.0	710.22	0.69 ± 0.05	7.5 ± 0.1	110	93.9
AMCF- ^{2.5} C _{fine} (With marble powder)	12.5	719.14	0.84 ± 0.05	7.7 ± 0.1	75	95.8

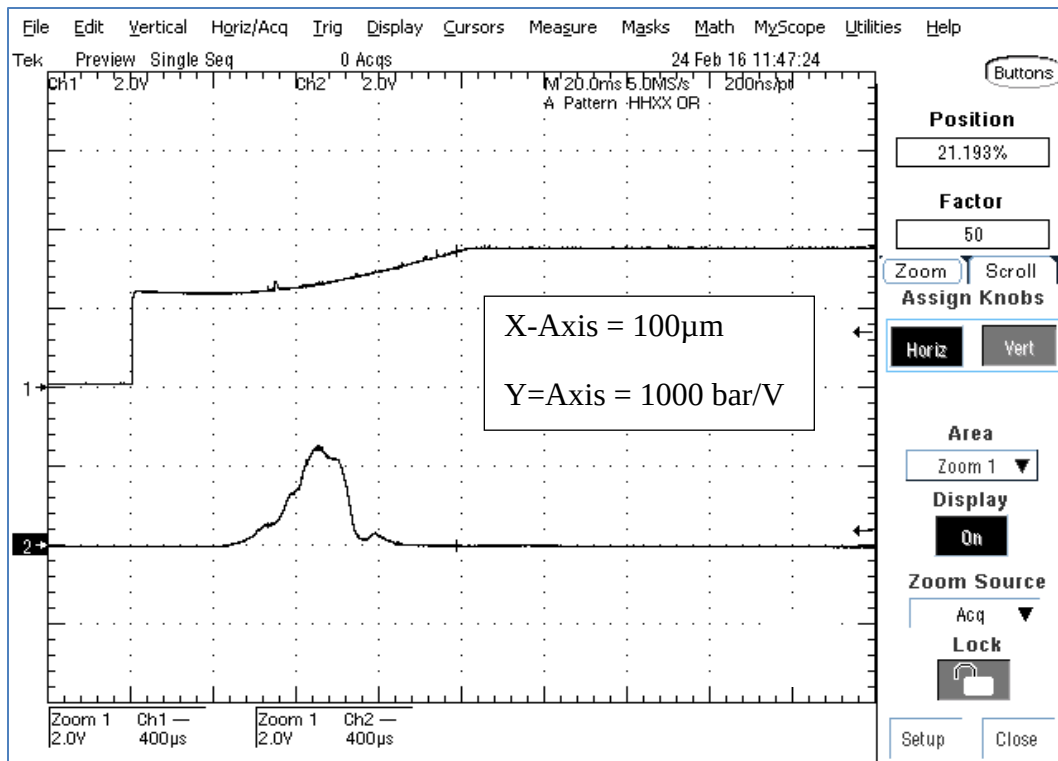


Figure 5.2: Pressure versus time graph of AMCF-⁵F_{fine-0-57}.

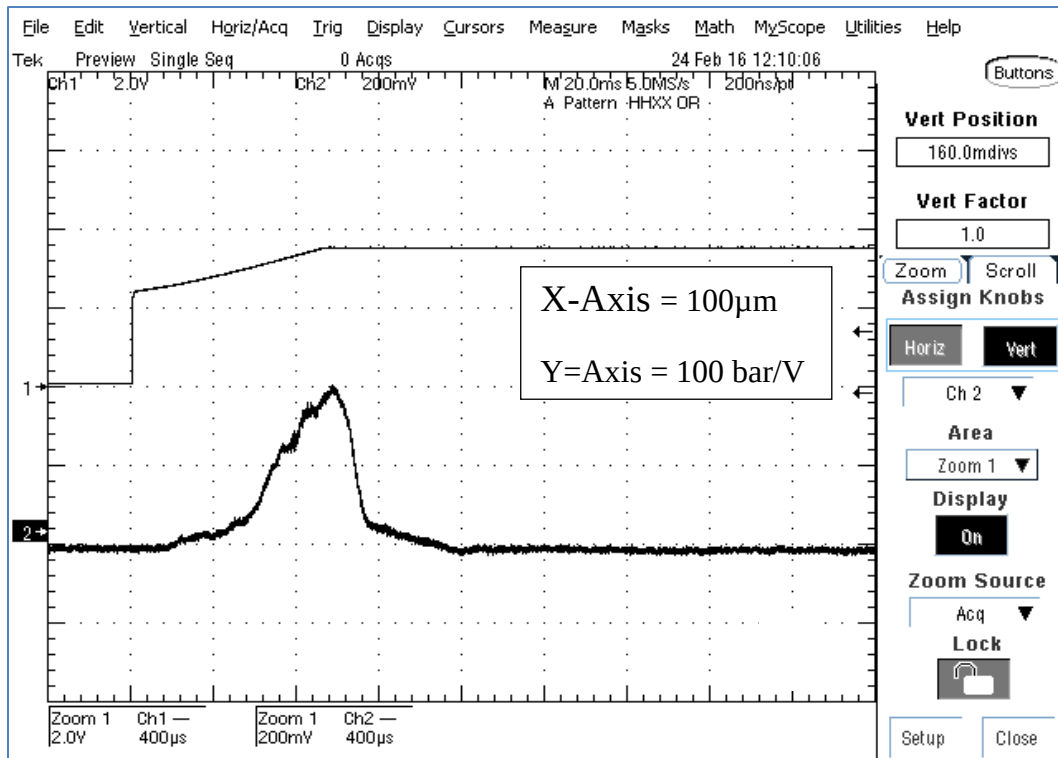


Figure 5.3: Pressure versus time graph of AMCF-^{7.5}F_{fine-0-86}

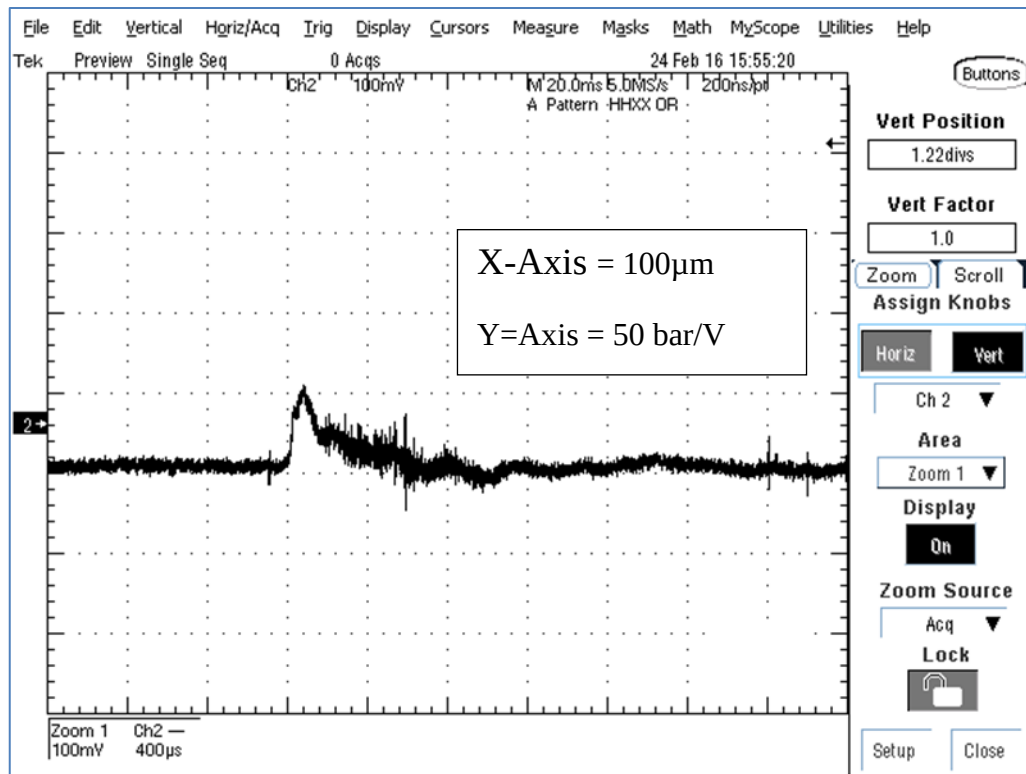


Figure 5.4: Pressure versus time graph of AMCF- $^{10}F_{\text{fine-0.77}}$

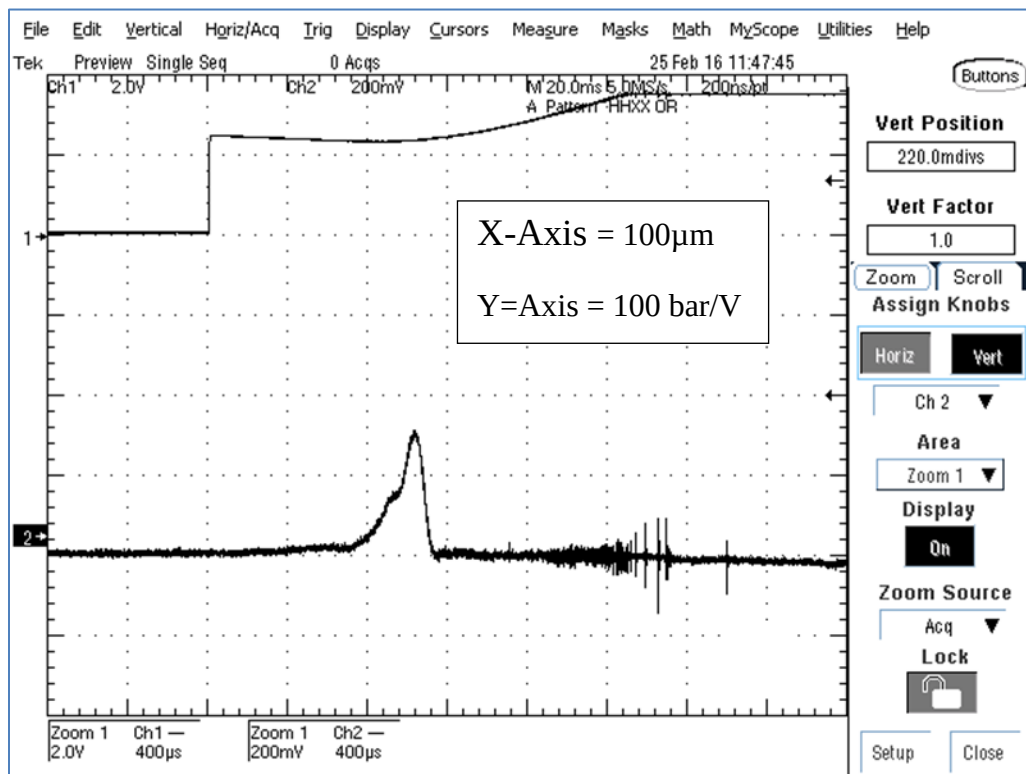


Figure 5.5: Pressure versus time graph of AMCF- $^{15}F_{\text{fine-0.44}}$

The analysis of the blast mitigation test done on zircon sand reinforced composites foam reveals that percentage attenuation is increasing with increase in amount of reinforcement. Moreover, it also depends upon the density of foam. Since the tests were done on two similar samples of same amount reinforcement where variation in attenuation is little, so reliability of data of the tests are more authentic. The variation in percentage attenuation within the two samples of same category tested containing 7.5, 10, 12.5 and 15wt.% reinforcement is 2.5, 0.5, 7.0, 1.0%. It is observed that reinforcement above 7.5% have given good results where percentage attenuation obtained is above 89%. This indicates that amount of reinforcement has greater influence in the strengthening the cell walls. However, reinforced in the range of 10 to 12.5wt.% have given better results in comparison to other samples.

When compared to AMF where no reinforcement in composite foam has been done, the data of AMCF where different amount zircon sand is reinforced, better results are observed. In case of AMF, the best results are obtained in AMF-^{1.5}F_{900 °C} sample having density 0.8 gm/cm³. On similar lines aluminum matrix containing reinforcement from 10-15wt.% have density in the range of 0.6-0.8 gm/cm³ but percentage attenuation in composite foam is higher. This clearly indicates that reinforcement of zircon sand strengthen the cell wall.

The above analysis also indicate that foams that exhibit higher resistance to wear, also show better blast resistance.

5.1.3 Failure Analysis

The results for the blast mitigation tests have shown variation even for the higher zircon sand reinforced foams. The basic reason for such variation is due to variation in geometry of cells and their size. The process of foaming involves mixing of reinforcement by rotating blades in the molten metal. During rotation process vortex formation occurs. It is possible that each blade during rotation may push up the reinforcement which may lead to development of composites containing zircon sand particles which get distribution throughout the melt. However, the particles may also get segregated at some places because of counter created inside the melt. Through the process parameters have been optimized but such tendency of aggregation cannot be avoided. Similarly, when foaming agent is added, it will also follow the similar trend. During foaming process such segregation may lead to formation of interconnected pores as has been described in earlier section. The particle size of foaming agent also varies. This may also lead to variation in structure. Since time of decomposition is same for each CaCO₃ particles (foaming agent) so some of the particles

will not undergo complete decomposition but will undergo partial decomposition. Because of this phenomenon there will be lot of stresses even within the foam structure. Analysis of fractured samples reveals that most of the foams got broken into many pieces. This is because of stress gradient existing from center to periphery. Moreover, the detonator is also placed at center so it will create radial stress in the foams. Some of the failed samples are shown in Fig. 5.6 to 5.9 with variation in reinforcement. Figure 5.6 (a & b) shows similar trend of failure where the failed sample shows compression followed by rarest zone. This is because of fact that initially cells got compressed in the middle. When it propagates with lower intensity it starts pushing the cell structure further apart.

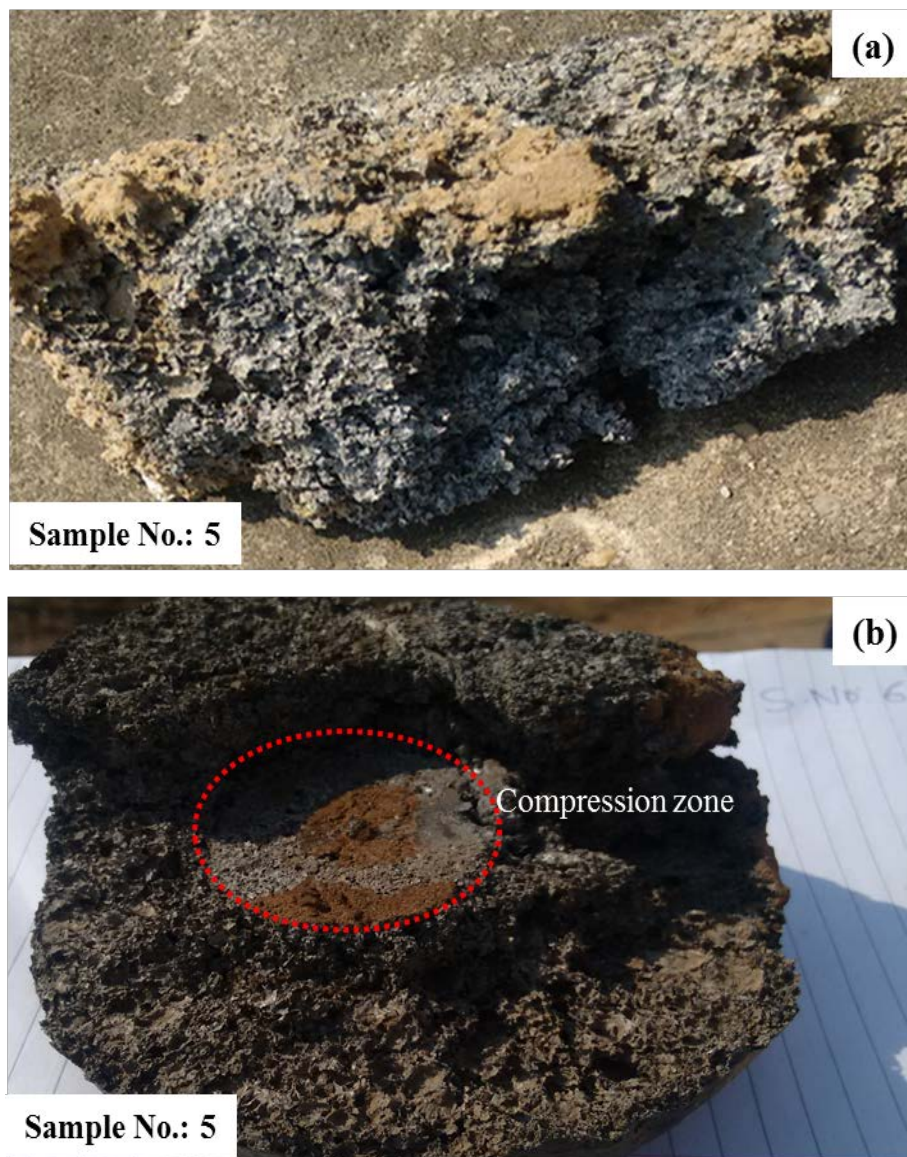


Figure 5.6: Photographs of sample (a) AMCF- $^5F_{\text{fine-0.51}}$ and (b) AMCF- $^{7.5}F_{\text{fine-0.86}}$ collected after blast test.

Figure 5.7 shows the partially molten form with flow pattern. It clearly detects the tearing and wave propagation direction.



Figure 5.7: Photograph of sample AMCF- $^{10}F_{\text{fine-0.70}}$ collected after blast test.

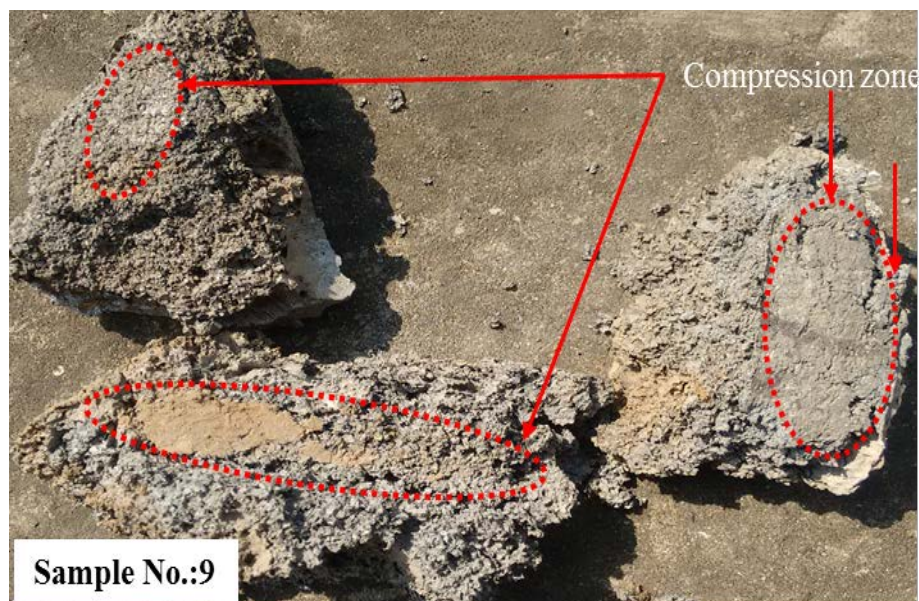


Figure 5.8: Photograph of sample AMCF- $^{12.5}F_{\text{fine-0.54}}$ collected after blast test.

As shown in Figure 5.8 (sample no 9) there exist two different features. One depicts the flow patterns and other compression zone. The flow pattern indicated the aligned structure because of movement of waves in that direction. The compressed zone helps in absorbing the waves by the collapse of cell walls as has been marked.

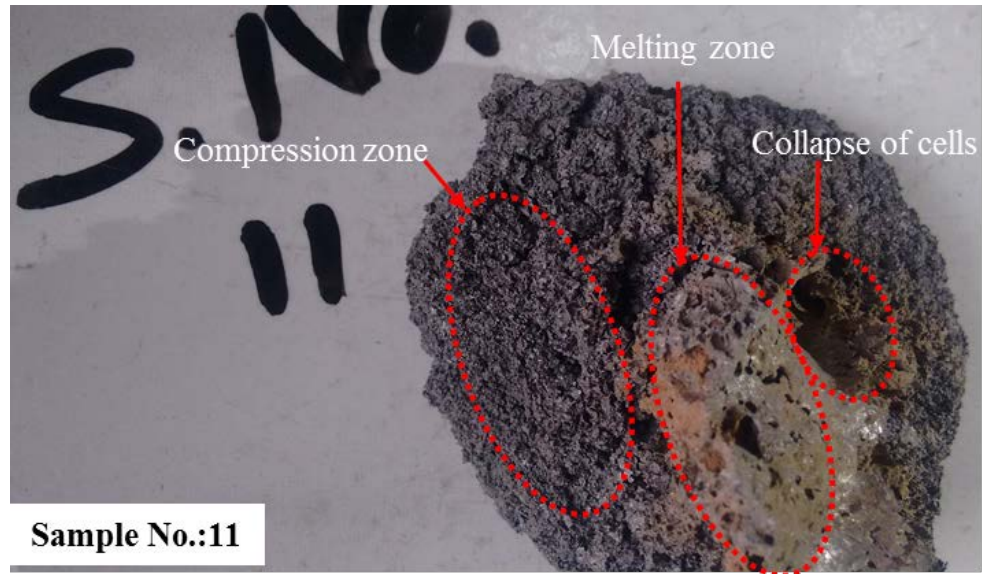


Figure 5.9: Photograph of sample AMCF- $^{15}\text{F}_{\text{fine-0.73}}$ collected after blast test.

In Figure 5.9 also shows two different zones that have been marked. Due to partial melting the layered structure is not clearly visible but alignment has been observed.

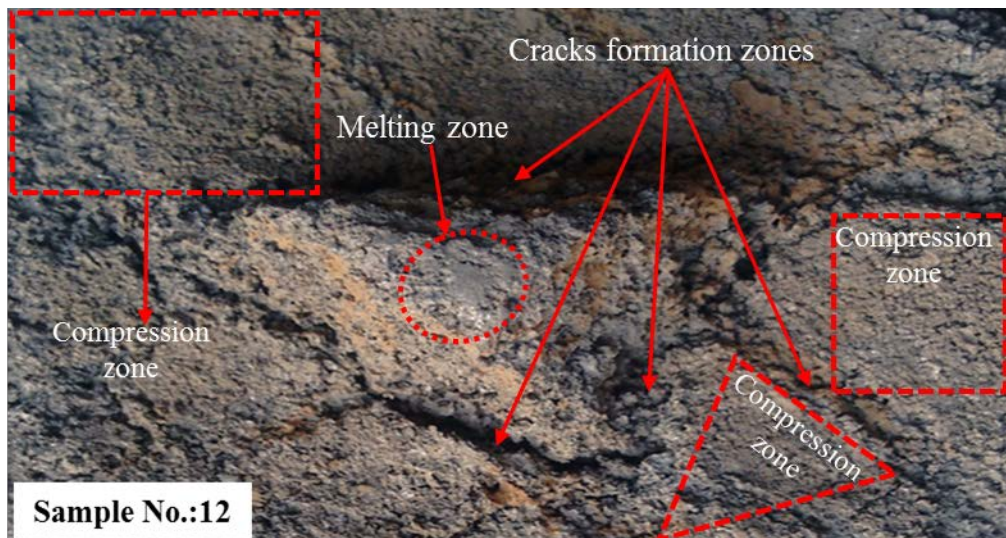


Figure 5.10: Photograph of sample AMCF- $^{15}\text{F}_{\text{fine-0.70}}$ collected after blast test.

Structural features shown in Figure 5.10 is for composite foam reinforced with 15wt.% fine zircon sand particles. This has shown better attenuation compared with other samples. The sample failed but is intact to greater distance. Random orientation of cracks indicates that higher resistance to wave propagation in all directions is offered by the sample. Near the cracks formation zones flow pattern is seen. The star like structure clearly shows the radial flow pattern. Because of this type of failure, the resistance offered is higher.

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CHAPTER 6

CONCLUSIONS AND FUTURE SCOPE

Overview:

The present chapter concludes the results obtained from various experiments discussed in previous chapters. Effect of reinforcement on the morphology and hardness as well as wear behavior of AMCs at different applied loads has been summarized here in this chapter. Effect of foaming temperature on the cell structure and density of the AMFs and AMCFs is given in brief. Effect of applied load on the wear behavior of AMFs and AMCFs has been summarized. Blast mitigation test also summarized.

6.1 Conclusions

In current scenario of materials development shows that composites are one of the most adaptable engineering materials known to men. Progresses in the field of materials science and technology have given birth to these fascinating and wonderful materials. Durability of the metals can be improved with addition of hard particles (ceramics). Development of metal foam has conveyed the new path for the automobile, defence and other engineering areas. Light weight to higher strength ratio make aluminum foam for industrial application. During this work, we have chosen LM13 alloy (Al-Si alloy) as matrix and zircon sand particles (mineral) as reinforcement to study the change in wear resistance.

X- ray diffraction (XRD), Optical microscopy, Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), Vickers hardness tests and wear test with pin-on-disc machine were done to study the effect of zircon sand particles on the microstructural, mechanical and tribological properties of LM13 alloy matrix.

A good interfacial bonding of the zircon sand particles with matrix is observed in the optical micrographs. However, rate of agglomeration increases with increasing the amount of the fine particles. At particle/ matrix interface, different structural features were observed. A modification from acicular to globular morphology was observed for Si.

Micro hardness of composites was measured using Vicker's hardness tester. Improvement in the hardness was examined with increasing amount of both fine and coarse size of zircon sand particles in the matrix. However, hardness of the composites increases with decreasing the size of zircon sand particles. **XRD study shows the presence of new harder phase (Al_2SiO_5) at the particle/matrix interface. This phase is responsible for the improvement in hardness of the composites.** Higher hardness of composite containing fine size particles is observed. Wear test of LM13 alloy and composites containing different amount of zircon sand was done. Composites containing higher amount of zircon sand (both fine and coarse) exhibited higher wear resistance at all studied loads. However, for same fraction of reinforcement, fine size zircon sand reinforced composite offer higher wear resistance as compared to composites containing coarse particles.

It was observed that foaming temperature has greater impact on the cell morphology of the AMF. When the temperature increases from 850 °C to 875 °C and 900 °C, the cell size of LM13 alloy foam is not uniform due to the decrease in surface tension and viscosity. The concentration of gas bubbles in the melt liquid is increased and the pressure inside the bubble is enhanced at this

condition and the probability of non-uniform cell size is increased. 2wt.% CaCO_3 blowing agent and 875 °C foaming temperature is optimum combination for the uniform cell size of closed cell LM13 alloy foam.

The cell geometry of the AMCFs is affected with size and volume fraction of ZrSiO_4 reinforced particles and variation in foaming temperature. The increase in foaming temperature and gas flow rate enlarge the cell size and decreases the cell wall thickness due to the difference pressure between cell wall and node.

Wear behaviour of the developed AMFs was studied by testing the samples with pin- on- disc machine at different operating parameters. The specimens of 25mm in length and 12mm diameter having flat ends were used for wear testing. Three different types of specimens were prepared; one foamed with 1.5wt.%; second 2.wt.% and third with 2.5wt.% CaCO_3 powder. Each specimen was tested at constant sliding velocity of 1.6 m/s. Wear behaviour of the AMFs was tested at different loads (9.8N, 29.4N, and 49N). Change in wear rate of the material was assessed by measuring the change in height of material during wear test at different testing conditions. Change in wear behaviour of the AMFs was monitored with sliding distance at different loading conditions.

It was observed that wear rate of the all AMFs increases with increasing the applied load from 9.8N to 49N. While at higher load of 49N, the increased depth and width of the grooves indicates the transition from mild wear to severe wear. This is due to fact that the pores in the foam itself absorb the wear debris during the rubbing process causing high friction due to incomplete contact. At higher load of 49N, the wear loss is due to the combination of adhesive and delamination. Delamination wear is prominent at higher load because of the fragmentation of oxide layer which covers the surface due to the accelerated oxidation of the metal surface layers in contact. SEM examination of the wear surfaces reveals distinct pattern of grooves and ridges running parallel to one another in the sliding direction.

Micro-hardness of the AMCFs material increases with an increase in the amount of reinforced zircon sand particles. However, AMCF- $^{15}\text{F}_{\text{fine}}$ shows maximum micro-hardness in comparison to other configuration of the composite foam at different phases.

The most favourable condition to achieve stability of the bubbles were: holding temperature close or below the liquidus temperature of the AMCFs with different amount of zircon sand from 2.5wt.% to 15wt.%. At the foaming temperature (900 °C), the stability of the bubbles is affected with increase in amount of zircon sand from 2.5wt.% to 15wt.% because the increased viscosity

of molten metal stabilizes the gas bubbles.

Wear rate of the AMCFs was found to decrease with increase in the amount of ZrSiO_4 particles from 2.5wt.% to 15wt.% in the matrix at all loads. Increased amount of ZrSiO_4 particles in the matrix enhances the hardness as well as the load bearing capacity of the matrix which ultimately leads to decrease in wear rate. The studies revealed that the all AMCF- F_{fine} exhibited better wear behaviour as compared to all AMCF- C_{coarse} at all applied loads. Fine size ZrSiO_4 particles have less particle to particle distance in the matrix which increases the capacity to transfer the load from matrix to particle and hence reduces the wear rate of the composite and composite foams. The transition in wear mechanism depending on the porous structure, especially foam sample because the pores act as craters which lead to reduced nominal contact. After the dry sliding wear test, worn surfaces and wear debris were analysed under SEM to understand the wear mechanism. SEM micrographs indicate that both abrasive and adhesive wear mechanism are involved during the wear test.

AMF- $^{1.5}F_{900\text{ }^{\circ}\text{C}}$ has the most important feature for blast resistant structure with maximum percentage attenuation transmitted pressure because the effective range of blast pressure depends on the density and cell geometry of the AMF. The percentage attenuation transmitted pressure of blast pressure depends on densities and cell geometry of AMCFs. The densities and cell geometry of AMCF is affected by addition of different amount of reinforcement. The percentage attenuation transmitted pressure of AMCFs is increased with increases amount of ZrSiO_4 from 2.5wt.% to 10 wt.%. All the data presented in this thesis can be further summarized as follows:

- AMCs, AMFs and AMCFs were developed by stir casting process.
- From microstructural analysis, it has been found that there is good bonding between the LM13 alloy and ZrSiO_4 particles.
- Hardness of the composite material increased with an increase in reinforced amount. The increase in size of the ZrSiO_4 particles causes the hardness to decrease.
- Wear resistance of the composite materials increases with an increase in reinforcement of ZrSiO_4 from 2.5 wt.% to 15wt.% with the same size.
- Wear rate of the both AMFs and AMCFs increased with an increase in applied load.
- From microstructural analysis, it has been found that uniform cell structure is obtained for the AMF prepared by 2wt.% CaCO_3 at 875 $^{\circ}\text{C}$.
- The influence of size and volume fraction of reinforcement has similar effect on mechanical

properties of AMC and AMFC. Influence of size and volume fraction on wear resistance of AMC and AMFC is similar and independent of wear mechanism.

- The percentage attenuation transmitted pressure of blast is maximum of AMF and AMCF which is prepared by 1.5wt.%CaCO₃ at 900 °C (AMF-^{1.5}F_{900 °C}) and fine size 10wt.%ZrSiO₄ at 900 °C with 0.77gm/cc density (AMCF-¹⁰C_{fine-0.77}), respectively.

6.2 Future Scope

The work done in present investigation has led to some conclusions which have been described in this work. However, the possibilities of further investigations of these developed AMCs, AMFs and AMCFs based on above work can be explored, some of which are as follows:

- The developed AMCs, AMFs and AMCFs can be further investigated for their compressive strength, corrosion resistance along with some electrical and thermal measurement. It will help to widen their area of application in different engineering designs and applications.
- The wear behaviour of the AMCs, AMFs and AMCFs can be further investigated at different environmental conditions like under lubricating condition and corrosive environment where temperature variability may be taken into account.
- Apart from this, a new AMCs, AMFs and AMCFs of sandwich nature can be fabricated for different military applications. This particular type of structure will be useful in defence for following purposes;
 - Porous aluminium foam material can be used as sound absorber in shooting range complex.
 - In different parts of defence vehicles.
 - Beneath the vehicles to absorb shock waves.
 - To make safety box to carry different defence materials during warfare.