

**STUDIES ON ALUMINUM – IRON ORE IN-SITU
PARTICULATE COMPOSITE**

**A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF**

**Master of Technology
In
Metallurgical & Materials Engineering**

Submitted

By

AKHILESH Kr. SINGH

Roll No.208MM108



**Department of
Metallurgical & Materials Engineering
National Institute of Technology Rourkela
2010**

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Under the guidance of
Prof. S. Sarkar



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CERTIFICATE

This is to certify that the thesis entitled, “**Studies on Aluminum – Iron ore in-situ Particulate Composite**”, submitted by **Akhilesh kr. Singh** in partial fulfillment of the requirements for the award of **Master of Technology** Degree in **Metallurgical and Materials Engineering** at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

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

Place: Rourkela

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Discontinuously reinforced aluminium matrix composites are fast emerging as engineering materials and competing with common metals and alloys. They are gaining significant acceptance because of higher specific strength, specific modulus and good wear resistance as compared to ordinary unreinforced alloys. Reinforcing particles or short fibers normally used are silicon carbide and alumina which are added externally. Recently it has been shown that alumina particles can be produced in-situ by reaction with metallic oxides reduced by aluminium. Alumina particles thus produced are dispersed in the aluminium matrix and the elementary metal gives solid solution strengthening of the matrix.

In-situ particulate composites in comparison with conventional cast particulate composites produced by external addition, promote cleaner interface, eliminates interface incompatibility of the matrices with the reinforcements, help to achieve greater thermodynamic stability of reinforcement particles in the matrix at elevated temperature and also increase the possibility of developing coherency between the matrix and particles formed in-situ. The morphology and the distribution of particles strongly influence the physical and mechanical properties of composites

In the present investigation, iron ore was added to molten aluminium, aluminium-magnesium and aluminium-silicon alloys by vortex method. The iron oxides present in the iron ore are observed to react with aluminium, magnesium resulting in production of Al_2O_3 , MgO and metallic iron which dissolved in liquid aluminium. The composites thus produced were cast into cast iron die.

The mechanical properties of the composites were evaluated. Strength and hardness value of the composites shows considerable improvement. The dry sliding wear behaviour of the composites in the cast condition was studied at different loads and different sliding velocities using Pin-On-Disk configuration wear testing machine. The worn surfaces and the wear debris were also analyzed using optical microscope and scanning electron microscope.

Metals such as aluminium and its alloys are one of the most used material systems in the automotive and aerospace industry due to their high strength to weight ratio as well as because of their high thermal conductivity. It is mostly used in high temperature applications such as in automobile engines and in other rotating and reciprocating parts such as piston, drive shafts, brake rotors and in other structural parts which require light weight but high strength material [1,2]. One of the main drawbacks of this material system is that they exhibit poor tribological properties. Hence the desire in the engineering community to develop a new material with greater wear resistance and better tribological properties, without much compromising on the strength to weight ratio led to the development of metal matrix composites [3,4].

A composite may be defined as a material system comprising of two or more constituent materials that remain separate and distinct while forming a single component. The bulk material forms the continuous phase that is the matrix (e.g. metals, polymers, etc) while the other acts as the discontinuous phase that is the reinforcements (e.g. fibers, whiskers, particulates, etc). While the reinforcing material usually carries the major amount of load, the matrix enable the load transfer by holding them together [5].

The properties of ceramic reinforced metal matrix composites has been reported much better to that of its unreinforced counterpart [6,7]. The addition of reinforcing phase significantly improves the tribological properties of aluminium and its alloy system. The thinking behind the development of metal matrix composites is to combine the desirable properties of metals and ceramics. The combination of refractory particles with metallic matrix produces a material system with properties intermediate to that of matrix alloy and ceramic reinforcements. Aluminium have useful properties such as high strength , ductility, high thermal and electrical conductivity but have low stiffness whereas ceramic reinforcements are stiffer and stronger and have excellent high temperature resistance but they are brittle in nature[8].

There are number of processing techniques which have been developed in recent years for processing metal matrix composites. According to the type of reinforcements, the fabrication

techniques also vary considerably. The different techniques employed for metal matrix composites are powder metallurgy, spray deposition, liquid metal infiltration, squeeze casting, stir casting, etc [9,34]. All of them have their own advantages and disadvantages.

At the early stage of development of metal matrix composites, emphasis was on preparation of fiber reinforced composites. But the high cost of reinforcement fibers, restricted the commercial exploitation of this class except for some high technology applications. The particulate reinforced metal matrix composites are gaining importance nowadays because of their low cost with advantages like isotropic properties and the possibility of secondary processing.

Among the various processing techniques available for particulate or discontinuous reinforced metal matrix composites, stir casting is the technique which is in use for large quantity commercial production. This technique is most suitable due to its simplicity, flexibility and ease of production for large sized components. It is also the most economical among all the available processing techniques.

The present investigation has been focused on in-situ particulate composite formation by utilization of low grade powdered iron ore by its dispersion into aluminum matrix by stir casting method. The objective is to form the reinforcing phase within the metallic matrix by reaction of iron ore with aluminium in the metallic melt. For increasing the wettability, magnesium was also added. The composites were characterized with the help of optical, x-ray diffraction and scanning electron microscopy. Its tensile strength, hardness and wear behavior were also evaluated.

2.1 Composites

Composites are combinations of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and are embedded in the other material called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Typically, reinforcing materials are strong with low densities while the matrix is usually a ductile, or tough, material

Types of composites-

On the basis of Matrix material, composites can be grouped mainly into following types:

a) Polymer-matrix composites (PMC)

The most common matrix materials for composites are polymers. Polyester and vinyl esters are the most widely used and least expensive polymer resins. These matrix materials are basically used for fiber glass reinforced composites. The main disadvantages of PMCs are their low maximum working temperature, high coefficient of thermal expansion and sensitivity to radiation and moisture. The strength and stiffness are low compared with metals and ceramics.

b) Metal-matrix composites (MMC)

The matrix in these composites is ductile metals. These composites can be used at higher service temperature than their base metal counterparts. The reinforcements in these materials may improve specific stiffness, specific strength, abrasion resistance, creep resistance and dimensional stability. The MMCs are light weight and resist wear and thermal distortion, so it mainly used in automobile and aerospace industry. Metal matrix composites are much more expensive than PMCs and, therefore, their use is somewhat restricted.

c) Ceramic-matrix composites (CMC)

One of the main objectives in producing CMCs is to increase the toughness. Ceramic materials are resistant to oxidation and deterioration at elevated temperatures. Some of these materials would be ideal candidates for use in high temperature applications, specifically for components in automobile and air craft gas turbine engines. The developments of CMCs has lagged behind mostly for the reason that most processing routes involve higher temperature and only employed with high temperature reinforcements.

2.2 Metal matrix composites

Due to combination of metallic properties (ductility and toughness) with ceramic properties (high strength and high modulus), metal matrix composites show greater shear and compression strength with capabilities to perform at high temperature. From the last decade, use of metal matrix composites for automobile, aerospace and other high temperature structural and non structural applications has increased a lot. This is due to the availability of relatively inexpensive reinforcements and the development of various processing techniques resulting in reproducible microstructures and properties [10].

The matrix material generally used in metal matrix composites is aluminium and its alloys. Besides aluminium, magnesium, copper, nickel, titanium, etc are also used as matrix material for specific applications. Reinforcement materials include carbides (e.g. SiC, B₄C), nitrides (e.g. Si₃N₄, AlN), oxides (e.g. Al₂O₃, SiO₂) as well as elemental materials (e.g. C, Si). The reinforcements may be in the form of continuous fibers, chopped fibers, whiskers, platelets or particulates. SiC for example, is being used in Aluminium matrix composites in all the above mentioned forms.

2.3 Types of metal matrix composites

MMCs come in several distinct classes, generally defined with reference to the shape of their reinforcement:

Particle-Reinforced metals (PRMs) contain approximately equiaxed reinforcements, with an aspect ratio less than about 5. These are generally ceramic (SiC, Al₂O₃, etc.). PRMs commonly contain below 25vol. % ceramic reinforcement when used for structural applications, but can have as much as 80vol% ceramic when used for electronic packaging[11]. In general, PRMs are at least approximately isotropic. They are produced using both solid state (powder metallurgy) and liquid metal techniques (stir casting, infiltration). Their mechanical properties, while often inferior to those of fiber-reinforced metals, are more or less isotropic and often represent, at moderate cost, significant improvements over those of corresponding unreinforced metals.

Short Fiber- and Whisker-Reinforced metals. These contain reinforcements with an aspect ratio of greater than 5, but are not continuous. These composites are commonly produced by squeeze infiltration. They often form part of a locally reinforced component, generally produced to net or near-net shape. Their use in automotive engines is now well established.

Continuous Fiber-Reinforced Metals contain continuous fibers (of alumina, SiC, carbon, etc.) with a diameter below about 20 μm . The fibers can either be parallel, or pre-woven before production of the composite; this is generally achieved by squeeze infiltration.

Monofilament-Reinforced metals contain fibers that are relatively large in diameter (typically around 100 μm), available as individual elements. Due to their thickness, the bending flexibility of monofilaments is low, which limits the range of shapes that can be produced. Monofilament reinforced metals can be produced by solid state processes requiring diffusion bonding: they are commonly based on titanium alloy matrices, which are well-suited to such techniques.

Interpenetrating phase composites are ones in which the metal is reinforced with a three-dimensionally percolating phase, for example ceramic foam.

Liquid phase sintered metallic materials, include the cemented carbides, in which carbide particles are bonded together by a metal such as cobalt, and the tungsten heavy alloys.

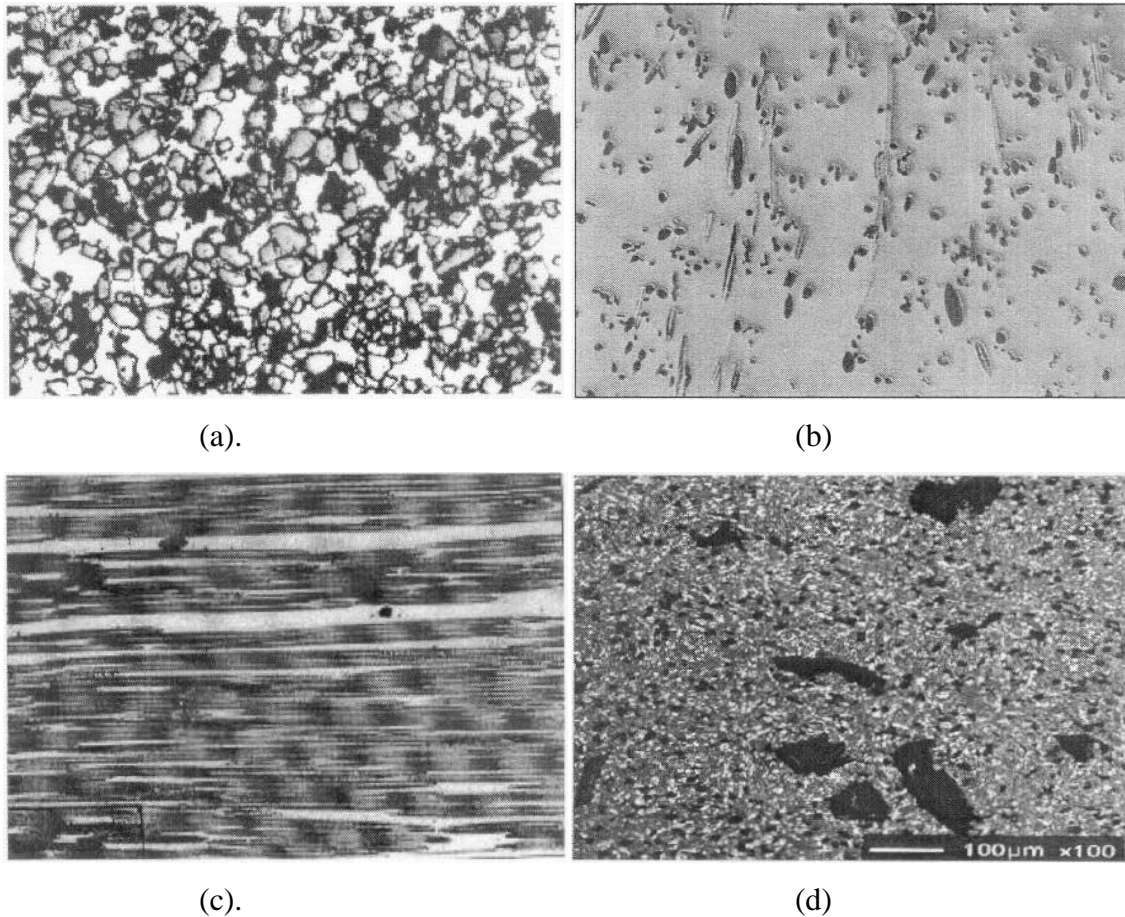


Fig.2.1 SEM Microstructures **(a)** aluminium matrix composite having high volume fraction of SiC particle reinforcement (40 vol%), **(b)** short fiber-reinforced aluminium matrix composite, **(c)** continuous fiber-reinforced aluminium matrix composite, **(d)** hybrid composite containing 10% SiC and 4% graphite particles.[11]

At the early stage of development of metal matrix composites, focus was on continuous fiber reinforced metal matrix composites based on Aluminium and Titanium matrices. But despite of encouraging results, the high manufacturing costs associated with high costs of fibers

restricted the extensive commercial application of these composites, limited, almost exclusively, to some highly specialized applications.

More recently, family of discontinuously reinforced metal matrix composites which include both particulates and short whiskers or fibers has attracted considerable attention because of:

- Availability of various types of inexpensive reinforcements,
- Development of various processing techniques to produce metal matrix composites with reproducible microstructures and properties, and
- Possibility of secondary processing of these metal matrix composites.

Moreover, the problem associated with fabrication of continuously reinforced metal matrix composites such as fiber damage, microstructural non uniformity, fiber to fiber contact and extensive interfacial reactions can be avoided with discontinuous reinforcements. Discontinuously reinforced metal matrix composites with nearly isotropic properties have been successfully utilized in applications such as in automotive components where extreme loading is not required with improvement in strength and stiffness as compared to those available unreinforced materials.

2.4 Fabrication of metal matrix composites

A variety of processing techniques have evolved over the last two decades in an effort to optimize the structure and properties of MMCs. Accordingly, these can be classified into:

- (a) Liquid state fabrication of Metal Matrix Composites,
- (b) Solid state fabrication of Metal Matrix Composites,
- (c) Fabrication of Metal Matrix Composites by co-deposition.

1. Liquid state fabrication of Metal Matrix Composites: This involves incorporation of dispersed phase into a molten matrix metal, followed by its Solidification. In order to provide high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the liquid matrix should be obtained.

Wetting improvement may be achieved by coating the dispersed phase particles (fibers). Proper coating not only reduces interfacial energy, but also prevents chemical interaction between the dispersed phase and the matrix. The methods of liquid state fabrication of Metal Matrix Composites are: **Infiltration** and **Stir Casting**.

Infiltration- This is a liquid state method of composite materials fabrication, in which a preformed dispersed phase (ceramic particles, fibers, woven) is soaked in a molten matrix metal, which fills the space between the dispersed phase inclusions.

The motive force of an infiltration process may be either capillary force of the dispersed phase (**spontaneous infiltration**) or an external pressure (gaseous, mechanical, electromagnetic, centrifugal or ultrasonic) applied to the liquid matrix phase (**forced infiltration**).

Stir Casting- This is a liquid state method of composite materials fabrication, in which a dispersed phase (ceramic particles, short fibers) is mixed with a molten matrix metal by means of mechanical stirring. The liquid composite material is then cast by conventional casting methods and may also be processed by conventional Metal forming technologies. Stir Casting is the simplest and the most cost effective method of liquid state fabrication.

Stir Casting is characterized by the following features:

- Content of dispersed phase is limited (usually not more than 30 vol.%).
- Distribution of dispersed phase throughout the matrix is not perfectly homogeneous:
 - There are local clouds (clusters) of the dispersed particles;
 - There may be gravity segregation of the dispersed phase due to a difference in the densities of the dispersed and matrix phase.
- The technology is relatively simple and low cost.

Distribution of dispersed phase may be improved if the matrix is in semi-solid condition. The method using stirring metal composite materials in semi-solid state is called **Rheocasting**. High viscosity of the semi-solid matrix material enables better mixing of the dispersed phase.

2. Solid state fabrication of Metal Matrix Composites: This is the process, in which Metal Matrix Composites are formed as a result of bonding matrix metal and dispersed phase due to mutual diffusion occurring between them in solid states at elevated temperature and under pressure. Low temperature of solid state fabrication process (as compared to Liquid state fabrication of Metal Matrix Composites) depresses undesirable reactions on the boundary between the matrix and dispersed (reinforcing) phases.

Metal Matrix Composites may be deformed also after sintering operation by rolling, forging, pressing, Drawing or Extrusion. Deformation of sintered composite materials with dispersed phase in form of short fibers results in a preferred orientation of the fibers and anisotropy of the material properties (enhanced strength along the fibers orientation). There are two principal groups of solid state fabrication of Metal Matrix Composites: **Diffusion bonding** and **Sintering**.

Diffusion Bonding- This is a solid state fabrication method, in which a matrix in form of foils and a dispersed phase in form of long fibers are stacked in a particular order and then pressed at elevated temperature.

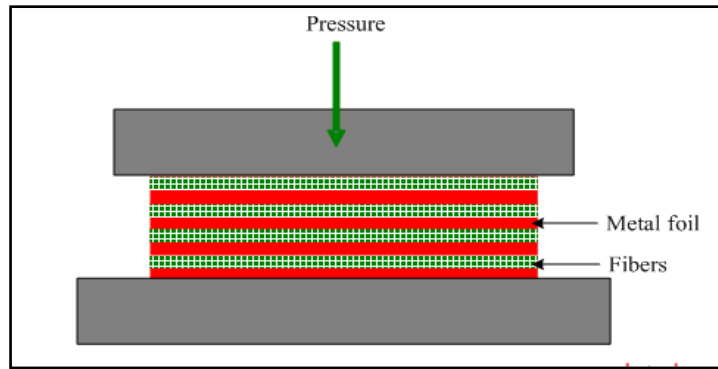


Fig.2.2 diffusion bonding

The finished laminate composite material has a multilayer structure. Diffusion Bonding is used for fabrication of simple shape parts (plates, tubes). Variants of diffusion bonding are roll bonding and wire/fiber winding:

Roll Bonding is a process of combined Rolling (hot or cold) strips of two different metals (e.g. steel and aluminum alloy) resulted in formation of a laminated composite material with a metallurgical bonding between the two layers.

Wire/fiber Winding is a process of combined winding continuous ceramic fibers and metallic wires followed by pressing at elevated temperature.

Sintering- This is a process, in which a powder of a matrix metal is mixed with a powder of dispersed phase in form of particles or short fibers for subsequent compacting and sintering in solid state (sometimes with some presence of liquid).

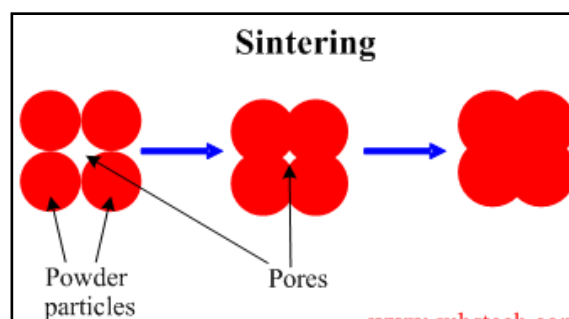


Fig.2.3 sintering

Sintering is the method involving consolidation of powder grains by heating the “green” compact part to a high temperature below the melting point, when the material of the separate particles diffuse to the neighboring powder particles. In contrast to the liquid state fabrication of Metal Matrix Composites, sintering method allows obtaining materials containing up to 50% of dispersed phase.

3. Fabrication of Metal Matrix Composites by co-deposition: It is a process, in which matrix metal is deposited together with the dispersed phase by one of the deposition techniques. The following co-deposition methods are used for manufacturing Metal Matrix Composites:

- **Electrolytic co-deposition**
- **Spray co-deposition**
- **Vapor co-deposition**

Electrolytic co-deposition- This method (Electrolytic co-deposition) involves Electroplating technique, in which electrolyte solution of matrix metal ions contains suspended particles of dispersed phase. When the matrix metal is deposited on a substrate, the dispersed phase particles are entrapped by the coating, reinforcing the matrix material.

Examples of electrolytic co-deposition:

- Nickel matrix composite materials with various dispersed phases are fabricated by electrolytic co-deposition from Nickel Sulfamate and Watts electrolytes:
- Ni-Al₂O₃ - oxidation resistant nickel matrix composite;
- Ni-SiC – wear resistant nickel matrix composite;
- Ni-PTFE, Ni-C, Ni-MoS₂ – antifriction nickel matrix composites.
- Anti-friction coating of Engine bearings consisting of lead-tin-copper alloy and reinforced by alumina (Al₂O₃) is fabricated by electrolytic co-deposition from electrolyte solution of lead, tin and copper with alumina particles.

- Aluminum matrix material reinforced by silica (SiO_2) is prepared from AlCl_3 -dimethylsulfone electrolyte containing fine silica particles.

Spray co-deposition- This method implements thermal spraying technique for atomizing molten matrix metal, droplets of which are delivered to a substrate in a high velocity gas stream together with dispersed phase particles supplied to the stream from a separate container. The method allows fabrication of near-net-shape forming of Metal Matrix Composites. Examples of spray co-deposition are:

Aluminum matrix material reinforced by silicon carbide (SiC) is produced by spray co-deposition followed by Rolling.

High Velocity Oxyfuel Spraying (HVOS) method is used for fabrication tungsten carbide-cobalt (WC-Co) composite material, which is conventionally manufactured by more expensive technology of sintering fabrication of Metal Matrix Composites.

Vapor co-deposition- This is a group of various methods, utilizing materials in vapor state: Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD), and Direct Vapor Deposition (DVD). In these methods coating of solid material is formed as a result of vapor condensation or chemical reaction on a substrate surface. Vapor co-deposition is used for coating fibers, creating multilayer depositions, fabricating nanostructure composite materials.

2.5 In-situ Metal Matrix Composites

In-situ composites are multiphase materials where the reinforcing phase is synthesized within the matrix during composite fabrication. This contrasts with ex-situ composites where the reinforcing phase is synthesized separately and then inserted into the matrix during a secondary process such as infiltration or powder processing [12]. In-situ processes can create a variety of reinforcement morphologies, ranging from discontinuous to continuous, and the reinforcement may be either ductile or ceramic phases.

Different types of Metal Matrix Composites may be prepared by in situ fabrication method:

1. **Particulate in situ MMC** – Particulate composite reinforced by in situ synthesized dispersed phase in form of particles.

Examples: Aluminum matrix reinforced by titanium boride (TiB_2) particles, magnesium matrix reinforced by Mg_2Si particles.

2. **Short-fiber reinforced in situ MMC** – Short-fiber composite reinforced by in situ synthesized dispersed phase in form of short fibers or **whiskers** (single crystals grown in form of short fibers).

Examples: Titanium matrix reinforced by titanium boride (TiB_2) whiskers, Aluminum matrix reinforced by titanium aluminide (TiAl_3) whiskers.

3. **Long-fiber reinforced in situ MMC** – Long-fiber composite reinforced by in situ synthesized dispersed phase in form of continuous fibers.

Example: Nickel-aluminum (NiAl) matrix reinforced by long continuous fibers of Mo (NiAl-9Mo alloy).

Dispersed phases of in situ fabricated Metal Matrix Composites may consist of intermetallic compounds, carbides, borides, oxides and one of eutectic ingredients.

Mechanical Properties of In-Situ particulate Composites

In situ particulate Composite are materials in which dispersed (reinforcing) phase is formed within the matrix as a result of precipitation from the melt during its cooling and Solidification. Generally, the reinforcements in discontinuously reinforced metallic- or intermetallic-matrix in-situ composites are on the order of 0.5-5 μm , and volume fractions range from 0-50 vol%. The potential advantages of in-situ composites as compared to discontinuous metal-ceramic composites produced by ex-situ methods include [12]:

- Smaller reinforcement particle size with higher strength (a contribution from composite-strengthening mechanisms) and improved fatigue resistance and creep
- Small, single-crystal reinforcements (lower propensity for particle fracture)
- Clean, unoxidized particle-matrix interfaces with higher interfacial strength (higher ductility and toughness) and improved wettability
- Thermodynamically stable particles that are weldable and castable, do not dissolve at high temperatures (*vis-à-vis* age-hardened alloys), and do not have a reaction layer (higher interfacial strength, improved corrosion, and long-term stability)
- Better particle-size distribution (improved mechanical properties)
- More conventional processing with the potential for lower cost and production with conventional equipment

These benefits may not be realized for all systems, but tend to be vary system and property specific.

In-situ composites also have disadvantages. The synthesis of in-situ reinforcement is typically by a precipitation process, and, as such, the choice of in-situ reinforcements is limited to particles that are thermodynamically stable within a particular matrix. The size and shape of particles are controlled by nucleation and growth processes, so although significant flexibility may be available during processing and synthesis, the size and shape are ultimately determined by the kinetics of the system instead of the ex-situ synthesis and processing of the reinforcements. Although particle distribution may be more uniform, the potential also exists for the pushing of fine particles during solidification.

2.6 Iron ore as reinforcement

The thermodynamic analysis indicates that there is possibility of reaction between Al melt and the iron ore particles. The iron ore particles contain oxides of iron and alumina, silica and other oxides in traces which during solidification process of Al- iron ore composites or during holding such composites at temperature above 850⁰ C, are likely to undergo chemical reactions. The Ellingham diagram indicates that the stability of common oxides come in order of MgO > Al₂O₃ > SiO₂ > Fe₂O₃. The experiments indicate that there is a progressive reduction of Fe₂O₃ and other oxides by Al and Mg leading to formation of MgO, Al₂O₃, SiO₂ and Fe₂O₃. There is also formation of complex oxides like MgAl₂O₄. All these oxides acts as reinforcement particles and helps in the strengthening of the composites.

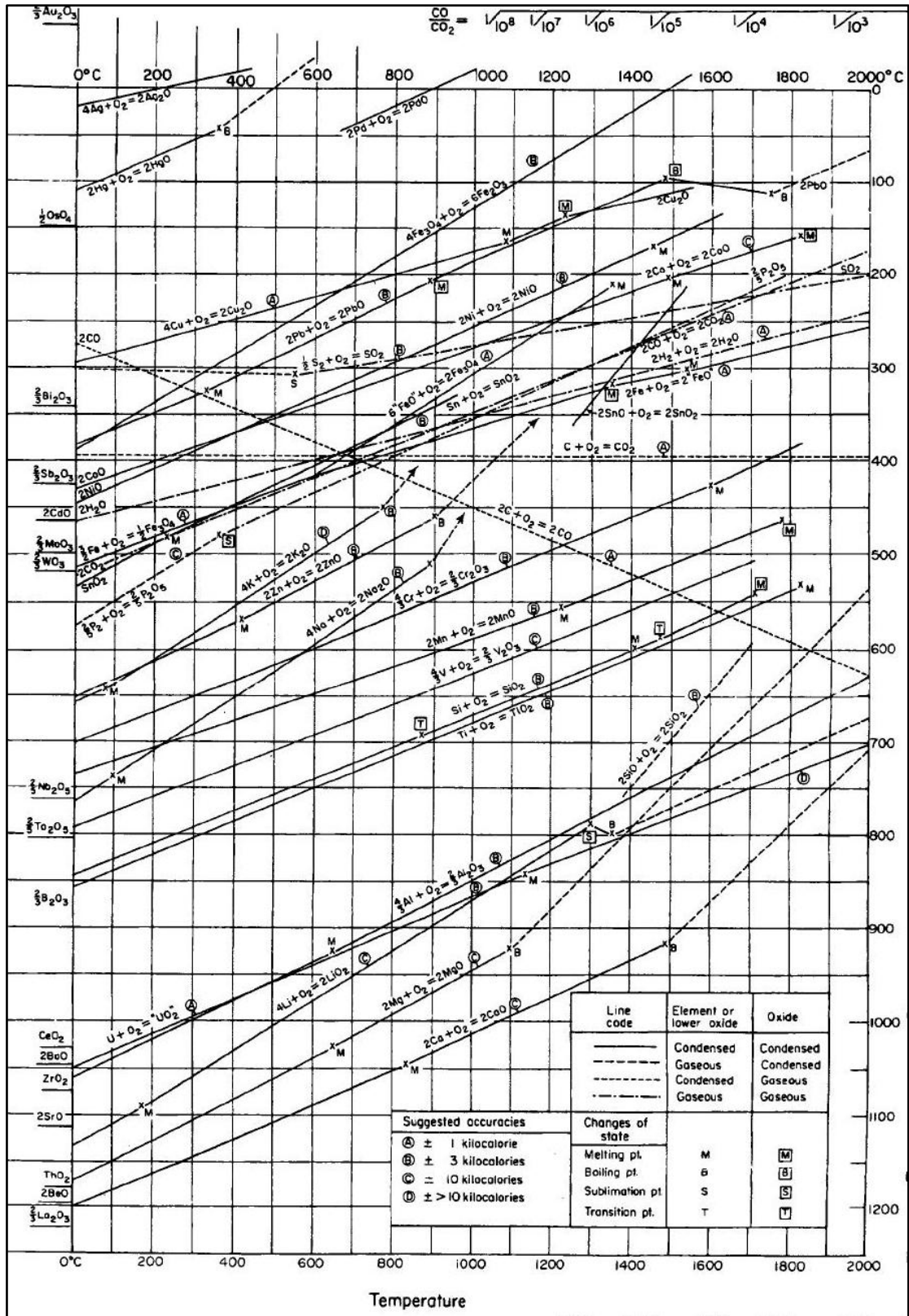


Fig.2.4 Ellingham diagram

2.7 Strengthening Mechanisms in particulate reinforced MMCs

The strengthening mechanisms observed in particulate reinforced MMCs may be divided into two categories, direct and indirect strengthening. Direct strengthening in particulate reinforced metals is an extension of the classical composite strengthening mechanisms used to describe the behavior of continuous fiber reinforced composites [13,14]. Under an applied load, the load is transferred from the weaker matrix, across the matrix/reinforcement interface, to the typically higher stiffness reinforcement. In this manner, strengthening takes place by the reinforcement carrying much of the applied load. Due to the lower aspect ratio of particulate materials, load transfer is not as efficient as in the case of continuous fiber reinforcement, but is still significant in providing strengthening[15,16].

In metal matrix composites, where a high stiffness ceramic reinforcement is embedded in a metallic alloy, the thermal mismatch between the high expansion metallic matrix and the low expansion ceramic is typically quite high. Thus, upon cooling, dislocations form at the reinforcement/matrix interface due to the thermal mismatch. In this manner, thermally induced dislocation punching results in indirect strengthening of the matrix [17].

In age-hardenable matrix materials, the thermally-induced dislocations (formed upon quenching from the solution treatment) serve as heterogeneous nucleation sites for precipitate formation during the aging treatment. Not only there is a preferential distribution of precipitates in the particle/matrix interface region, but the higher density of dislocations also causes acceleration in the time to peak-aging compared to the unreinforced alloy of a similar composition. An increase in reinforcement volume fraction or a decrease in particle size increases the amount of indirect strengthening, since a larger amount of interfacial area exists for dislocation punching to take place [13].

2.8 Material selection for MMCs

The structural efficiency of metal matrix composites is directly related to the density, elastic modulus and tensile strength of the reinforcing phase. The chemical and thermal stability of the reinforcements and compatibility with the matrix phase are important not only for the end application but also during material fabrication. The thermal mismatch strain, ϵ , is also an important factor for composites used in high temperature applications. It is a function of the difference between the coefficient of thermal expansion, $\Delta\alpha$, of the reinforcement and matrix according to the following expression

$$\epsilon = \Delta\alpha \Delta T \quad (1)$$

Where, ΔT is the temperature change. It is important for $\Delta\alpha$ to be a minimum in order to minimize strain accumulation [10].

The use of metal matrix composites for high temperature applications requires the presence of thermodynamically stable dispersoids which can be achieved by using an alloy dispersoid system in which elemental solubility, solid state diffusivity and interfacial energies are minimized, thereby minimizing coarsening and interfacial reactions. For example, titanium addition to aluminium promote the precipitation of Al_3Ti phase which enhances the thermal stability and structural efficiency of the matrix as a result of high m.p.(1330^oC), low density (3.3g/cc), and low diffusivity ($1.69 \times 10^{-14} \text{ cm}^2\text{s}^{-1}$) in aluminium[10].

2.9 Wettability

The nature and characteristics of the interface region formed between the matrix and the reinforcing phase determine load transfer and crack resistance of the metal matrix composites during application. It is well known that by promoting wetting, controlling chemical reactions and minimizing oxide formation, interfacial bond strength can be increased.

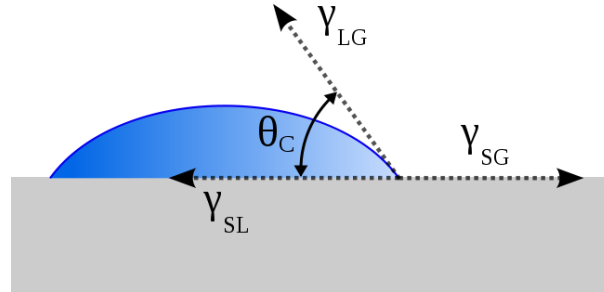


Fig.2.5 Contact angle formed between solid, liquid and gas phases

When the interfacial bond strength between a solid and a liquid exceeds the surface tension of the liquid, wetting is said to be achieved. Wettability is measured in terms of contact angle θ , formed between a solid and a liquid medium, as defined by Young's equation,

$$\gamma_{sg} = \gamma_{sl} + \gamma_{lg} \cos\theta \quad (2)$$

Where, γ_{sl} , γ_{sg} and γ_{lg} are the interfacial energies between solid and liquid, solid and gas, and liquid and gas phases, respectively [10]. In terms of the energetics of wetting, the work of adhesion, W_{ad} , is defined as the energy required to separate a unit area of the solid-liquid interface, according to

$$W_{ad} = \gamma_{lg} (1 + \cos\theta) \quad (3)$$

Hence, wetting is achieved when $\theta < 90^\circ$ (i.e., when $\gamma_{sg} > \gamma_{sl}$ or when the driving force for wetting, D_f , exceeds the liquid interfacial energy (i.e., $D_f > \gamma_{lg}$). The value of D_f depends on the surface tension of the liquid and the strength of the liquid-solid interface, which in turn are influenced by surface characteristics, interfacial reactions, heat of formation, valence electron concentration, temperature and time [10].

Methods to improve wettability

Molten metal's have generally very high surface tension (order of 1000 mJ m^{-2}), hence, wettability is very low in molten metal ceramic systems. Wetting can be increased in these systems by promoting a decrease in the contact angle by:

1. Increasing the surface energy of the solid,
2. Decreasing the solid-liquid interfacial energy, and/or
3. Decreasing the surface tension of the liquid metal.

The surface energy of the particulates can be increased by application of metallic coatings such as Ni and Cu to the ceramic particulates. The metallic coatings alter the nature of the interface from metal-ceramic to metal-metal. Hence, a strong interaction at the interface increases the wettability between the metallic matrix and the ceramic reinforcements. The coating of Ni and Ti-Ni on Al_2O_3 has been now in practice in Al- Al_2O_3 system.

The addition of reactive elements also increases the wettability of metal-ceramic systems by inducing a chemical reaction at the interface and also by decreasing the surface tension and the solid-liquid interfacial energy of the melt. The examples of such elements are Li, Mg, Ca, Ti, Zr and P. In aluminium alloys, for example, addition of elements having high affinity for oxygen increases the wettability of certain ceramic particles. The presence of 3 wt% Mg decreases the surface tension of pure aluminium from 0.760 to 0.620 N/m. Chemical reactions have been observed to occur readily between Al_2O_3 and divalent transition metal oxides resulting in formation of aluminate spinels such as MgAl_2O_4 . These mineral spinels or oxides form strong bonds between metal and ceramics thus promoting interfacial bonding [18].

Wettability in metal matrix composites can also be enhanced through the thermal treatment of ceramic particulates which helps in desorption of adsorbed gases from the ceramic surfaces. In the presence of oxygen, metals with high free energy of oxide formation form stable oxides which acts as effective diffusion barriers that decreases the level of interaction at the interface. As a result, wettability is poor unless a certain temperature threshold is reached at which the oxide can be penetrated by the molten metal. In addition to thermal exposure, ultrasonic irradiation of the melt also induces partial desorption of adsorbed gases from the ceramic particulates, thus improving wettability [10].

2.10 Stir casting technique

The high cost of production of even minimally complex shape components hinders the widespread adoption of particulate metal matrix composites. Casting technology is the solution to this problem. It is one of the cheapest methods for production of particulate matrix composites at large scale. But there are several technical challenges as mentioned below that need considerable attention before going for this technique [19-21,34].

- a) The difficulty of achieving a uniform distribution of particles.
- b) Wettability between the matrix and the particles.
- c) Porosity in the cast metal matrix composites.
- d) Chemical reactions between the reinforcement material and the matrix alloy.

The attainment of uniform distribution of particles within the matrix is essential for achievement of optimum mechanical properties. This is the most common problem which remains with most of the processing techniques including stir casting. Porosity is another common problem associated with casting technique.

In stir casting technique of production of metal matrix composites, a melt of the selected matrix material is produced by heating the material in a furnace. In the molten matrix material, the reinforcing particles are then added followed by stirring of the melt to make the reinforcement particles homogenous to the melt. After stirring, solidification of the melt containing suspended particles takes place to obtain the desired product [19]. For a homogenous distribution of reinforcing particles in the matrix material by stir casting route, the following factors need to be understood:

- 1) Particle density, size, shape and volume fraction plays an important role in the reinforcement settling rate.
- 2) Surface properties of particles determine the ease or difficulty of wetting.
- 3) The reaction of the reinforcing particles with each other and with matrix melt influences the rheological behaviour of the slurry.
- 4) During particle addition or stirring, gas entrapment takes place leading to poor distribution of particles due to attachment of particles to gas bubbles and also increase in porosity in the composite.
- 5) Mixing parameters should be so adjusted that particle distribution in axial and radial directions must be uniform.

- 6) The settling time should be as minimum as possible during solidification of melt.
- 7) The reinforcement particles, in general, occupy interdendritic or between secondary dendrite arm spacing, therefore the matrix grain size or the spacing must be finer for better distribution of particles [19].

In stir casting method, the particle distribution depends on process parameters during the melt and solidification stages. Flow and solidification characteristics are important factors affecting distribution of particles in the matrix. Both factors depends on parameters like viscosity of the melt, heat transfer rate, wettability of materials, stirring conditions, agglomeration of particles before and after mixing , pattern or mould shape and temperature

The addition of reinforcing particles increases the viscosity of the melt and makes it non-Newtonian and thus the casting fluidity of the melt decreases. There are two types of interaction occurs by dispersion of solid particles in the melt, both of which causes an increase in the apparent viscosity of the slurry [19,22]:

- 1) A hydrodynamic interaction between liquid and the particle.
- 2) A non-hydrodynamic interaction between particles themselves.

The chemical interaction between the reinforcing particles and the matrix material which causes a change in shape and volume fraction of the reinforcement is also responsible for the change in apparent viscosity of the slurry [19,23]. The suspended particles in the slurry also interact with each other giving rise to particle agglomeration which are apparently responsible for the observed shear rate dependent behavior of the slurries. The viscosity of the composite slurry decreases with increasing temperature because of the decrease in solid volume fraction as in unreinforced partially solidified matrix alloy melt [24].

An external driving force is required by the ceramic reinforcement particles for better wettability in the metallic melt. The stirring action of the mechanical stirrer in the melt provides this driving force to overcome the surface energy barriers and makes the ceramic particles more wettable. The alloy chemistry, temperature of particle addition and rate of stirring are some other parameters which control the wetting of the reinforcement particles by the melt [25].

The settling rate of reinforcement particles also plays an important role in casting process. According to Gieger and Zaki, the settling rate is also a function of particle density with particle shape and size playing a role. High volume fraction of particles hindered the settling rate by particle interaction with each other.

Richardson and Zaki proposed a model for settling of spherical particles with the particle velocity, V_c , is given by

$$V_c = V_o (1 - f)^n \quad (4)$$

Where, V_o is the stokes velocity, f the volume fraction of particles and n is a factor which depends on the Reynolds number, particle diameter and the diameter of the container. The value of n increases with increasing particle diameter. The studies show that for lower settling rate, the particle should be finer and their volume fraction be higher [19].

Thomas found that the particle shape and size are the most important parameters. According to Ray, when the flow velocity is above a critical value for a given size of particle, the suspension will remain homogenous during flow. If the flow velocity is reduced below critical value, the suspension becomes inhomogenous. Further reduction in flow velocity causes the particles to sediment at the bottom and move by tumbling over each other.

The stirring of slurry causes high shear rate which results in a fairly homogenous particle distribution in radial direction and prevents particle settling. At the same time secondary flow in axial direction causes lifting of particles by transfer of momentum from high to low momentum regions. To correlate particle lifting with flow parameters, a particle dispersion number (PDN) is given which is the ratio of the axial velocity of secondary flow to the terminal settling velocity. If PDN is greater than 1, the settling velocity is smaller than the axial velocity of the secondary flow and the particles will be carried to the upper portion of the melt. On the other hand, for PDN smaller than 1, the particles will remain at the bottom. For homogenous dispersion, PDN should be greater than 4[19,26].

During solidification, particle redistribution is generally affected by three mechanisms namely agglomeration, sedimentation and particle engulfment or rejection (pushing) ahead of the solidification front. Out of these which mechanism will be dominant, depends on the

elements of processing technique as well as the physical and chemical properties of the particle and the matrix. In general, molten metal normally solidify dendritically. When the temperature drops below the liquidus temperature solidification of the molten matrix start. Some very small dendrites appear at this time which grows as the temperature decreases. Since, the ceramic reinforcement particles have generally lower thermal conductivity than the metallic melt; it causes the particles to be surrounded by the last freezing of the molten alloy during solidification. During cooling the melt viscosity increases. There is also slight increase in density. Both of these effects tend to reduce the particle settling rate compared to an isothermal melt bath. The continuously precipitating crystals in the solidifying matrix also hinder the settling rate.

Two mechanisms have been suggested for particle pushing from fluid flow [19,27]. The first mechanism says that the particle is in contact with the solid and moved over the surface by the fluid flow as the solid grows. In the second mechanism, the particle which is located near the solidification front are trapped because of the roughness of the solidification front. The rejection of particles by the growing crystals and its pushing ahead of the advancing interface results in generation of a viscous force which prevents the pushing of the particles. Hence the balance of these counteracting forces decides whether the particles is rejected or engulfed. The various parameters which affect the shape of the solidification front are relative density difference, relative difference in thermal conductivity and heat diffusivity between the particle and the metallic melt, and alloy composition [28,29]. Pushing of particle suggests that the solid metal has no affinity for the reinforcement and that the interfacial bonding is weak.

A low particle-solid interfacial energy is the requirement for both engulfment and nucleation, just like low particle-liquid interfacial energy required for particle incorporation. This lowering of interfacial energy can be achieved if the solid and the particle share the same crystal structure and lattice parameter. Ceramic materials which act as grain refiner such as TiB_2 and TiC are likely to be engulfed within the metal grain rather than pushed to the boundary.

2.11 Impeller parameters

To obtain a homogenous dispersion of reinforcing particles in the melt as well as castings free from porosity are the two major problems which arises during processing of particulate reinforced metal matrix composites. During stir casting process, the impeller parameters like type, diameter and width of impeller as well as angle and number of blades play an important role in the distribution of reinforcement particles in the metallic melt [30]. Thus proper understandings of the impeller parameters are necessary.

Impeller types: Impellers are classified into axial flow and radial flow impellers on the basis of pumping action. In axial flow impellers, the blade makes an angle less than 90° degrees with the plane of impeller rotation. The pumping action is vertically up and down away from the blades parallel to the shaft. In radial flow impellers, the blades make an angle of 90° degree with the shaft. The pumping action of radial flow impellers are horizontally away from the impeller blades. The axial flow impellers will have more discharge than shear while radial flow impellers have more shear than flow for a particular speed [30].

Diameter of the impeller: The effect of impeller diameter (d) can be assessed only with respect to the vessel diameter (D) in which the liquid with secondary particles is being stirred. For solid suspension applications, the practical maximum d/D ratio is 0.6 while the minimum will vary according to process viscosity, increasing as the viscosity increases. Investigators have used d/D values ranging from 0.3 to 0.75. With an increase in d/D ratio of the impeller, the porosity content has been found to drastically increase in the composites.

Width of impeller: The impeller width is usually expressed in terms of impeller diameter. This ratio is significant only when the speed with which the impeller is rotated comes into picture. Table 2 lists the values of w/d ratios used by some researchers.

(w/D) Ratios chosen by investigators for Al MMC synthesis

Investigators	MMC synthesized	w/D ^a
Rana et al. [9]	Al-SiC	0.5
Surappa and Rohatgi [10]	Al-Graphite	0.25
Badia et al. [17]	Al-Graphite	0.3

The increase in blade width causes both the radial and axial flow of the impeller to increase. The suspension is favored by the increase in the axial component while it is hampered by the

increase in the radial component because the radial component counters the axial flow rising along the tank wall. Hence, it is advisable to have a stirrer width falling within the optimum w/d ratio of 0.35. [30]

It is seen that the investigators have used different values of vessel and impeller parameters for preparation of metal matrix composites. The reason should be that the values given by chemical engineering principles are for perfectly wetting systems. In metal matrix composites, the wetting between the particles and the matrix are generally very poor. As per chemical engineering principles, solid suspension applications requires axial flow impellers, investigators have used radial for the synthesis. It is said that a thin air film surrounds each particle. So the increase in the concentration of the particles lowers the interparticle distance, which on reaching critical value leads to the rupture of the liquid film between them. This causes formation of gas bridges between the particles which pulls the particles together leading to spontaneous rejection. Radial impellers generate more shears which hinders the coming of particles together. Thus with axial flow, radial flow is also required in the synthesis of MMCs [30].

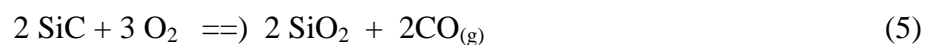
2.12 Porosity

Porosity is a defect which arises during fabrication of metal matrix composites by stir casting method. It is a measure of the void spaces in a material, and is a fraction of the volume of voids over the total volume. The various reasons for porosity formation are [31,32]:

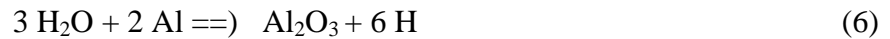
1. Entrapment of gas during stirring of the melt.
2. Air bubbles entering the slurry either independently or as air envelope to the reinforcements.
3. Water vapor on the particle surface.
4. Evolution of hydrogen due to any reaction inside the melt.
5. Shrinkage during solidification [33].

These are directly or indirectly affected by the casting route applied, process parameters which consist of holding time, position of impeller and stirring speed, and the volume fraction of reinforcing particle [31,34-36]. The studies show that the increase in particle size, aspect ratio, and volume fraction of particles increases the porosity content [31] whereas increasing the holding time decreases the porosity level. Samples produced using steel mould have been observed to decrease the porosity content compared to graphite mould since faster cooling rate observed in steel mould tends to distribute the reinforcement particles uniformly as well as decreasing the possibility of porosity formation which is most likely to develop during solidification [33].

In stir casting method, due to stirring action, vortex formation takes place which helps in the transferring of particles inside the metallic melt due to pressure difference between the inner and outer surface of the melt. This causes porosity inside the slurry. Water vapor present on the particle surface are also responsible for porosity formation. Along with water vapor, surface of SiC_p are frequently covered with SiO₂ layer which originates during SiC production process. SiC undergoes passive oxidation in presence of atmospheric oxygen leading to stable SiO₂ film formation,



During aluminium casting, hydrogen evolution also results in porosity formation by the reaction of aluminium with water vapor [33],



Since, materials are generally denser in the solid state than in the liquid state, contraction or shrinkage occurs during solidification. When one surface solidifies more slowly than the other or if solidification begins at all surfaces of casting the bulk of shrinkage occurs as cavities. Interdendritic shrinkage, also known as micro shrinkage or shrinkage porosity, exists in aluminium alloys. These are small, normally isolated pores between the dendrite arms formed by the shrinkage that accompanies solidification. Faster cooling rates may reduce this problem, where the dendrites will be shorter, permitting liquid to flow through the dendritic network to the solidifying solid interface [33].

2.13 Physical properties of MMCs

Low density alloys such as that of aluminium and magnesium are in use for making low density metal matrix composites. Most metallic matrices generally have very high thermal conductivities (TC), but their coefficient of thermal expansions (CTEs) is substantially higher as compared to most of the available reinforcements. Most of the carbide and oxide reinforcements exhibit CTEs of nearly zero. Thus combination of these can produce MMCs with extremely low CTEs [10].

There are various mathematical models, formulated for predicting the properties of MMCs from the properties of individual components. Rule of Mixture (ROM), is one such model by which any desired property like CTE, density, strength, modulus, etc can be approximately calculated from the weighted average of the individual components.

$$\alpha_c = \alpha_m V_M + \alpha_r V_r \quad (7)$$

where, α is the desired property, V the volume fraction and subscripts c , m and r refer to composite, matrix and reinforcement respectively. Turner proposed a new model for calculation of CTE by considering the effects of isostatic stress,

$$CTE_c = (CTE_m V_m K_m + CTE_r V_r K_r) / (V_m K_m + V_r K_r) \quad (8)$$

Where, K is the bulk modulus of the phase. A more complicated model has been proposed by Kener which has taken into account, the effects of shear stresses between matrix and isotropic approximately spherical reinforcements.

$$CTE_c = CTE_m - V_r (CTE_m - CTE_r) A/B \quad (9)$$

Where

$$A = K_m (3K_r + 4\mu_m)^2 + (K_r - K_m) \times (16\mu_m^2 + 12\mu_m K_r)$$

$$B = (3K_r + 4\mu_m) [4V_r \mu_m (K_r - K_m) + 3K_m K_r + 4\mu_m K_r]$$

Where, μ is the shear modulus. The value predicted by this model comes between those predicted from ROM and turner model. For calculation of thermal conductivity, Rayleigh has proposed the following model,

$$k_c = k_m [(1 + 2V_r) (1 - k_m/k_r) / (2k_m/k_r + 1)] / [(1 - V_r)(1 - k_m/k_r) / (k_m/k_r + 1)] \quad (10)$$

where k is the thermal conductivity. This equation has however, not taken into consideration, the effects of thermal barriers due to matrix reinforcement interface. For calculation of elastic modulus, Hashim and Shtrikman have proposed the following model,

$$E_c = E_m [E_m V_r + E_r (V_r + 1)] / [E_r V_m + E_m (V_r + 1)] \quad (11)$$

where, E is Young's modulus, the remaining terms have been earlier defined[10].

2.14 Tensile strength

The reinforcing phase in the metal matrix composites bears a significant fraction of stress as it is generally much stiffer than the matrix. Microplasticity in MMCs which takes place at fairly low stress has been attributed to stress concentrations in the matrix at the poles of the reinforcement and/or at sharp corners of the reinforcing particles [37]. The increase in volume fraction of reinforcing particles initially decreases the microyielding stress due to increase in number of stress concentration points [38].

The particle incorporation results in an increase in work hardening of the material. The higher work hardening rate observed in the composites is due to geometric constraints imposed by the presence of the reinforcement. The work hardened matrix is under great constraint. The inability of the matrix for strain relaxation causes the onset of void nucleation and propagation, which takes place at a much lower applied strain than that observed in the unreinforced material.

The increasing volume fraction (decreasing matrix volume) increases the work hardening rate. The earlier onset of void nucleation with increasing volume of reinforcement results in lower ductility of the composite. This lowering in ductility is further enhanced by the high stress concentrations at the tips of the cracked particles.

The effect of particle size on tensile behavior indicates that with a decrease in particle size, ductility increases [39]. This is due to increase in particle strength with decrease in particle size, since the probability of a strength-limiting flaw existing in the volume of the material decreases. The decreasing particle size also results in higher work hardening rate due to formation of dislocation tangles around the particles. This is due to plastic incompatibility between the reinforcement and matrix, and the formation of a dislocation cell structure with a cell size inversely proportional to the interparticle spacing [40].

2.15 Wear behavior

Wear is a process of surface damage or removal of material from one or both of two solid surfaces occurring during sliding, rolling, or impact motions relative to one another. It is usually a progressive loss of weight generally slow but steady and continuous. The loss of material during wear is expressed in terms of volume. The volume loss gives a truer picture than weight loss, particularly when comparing the wear resistance properties of materials with large differences in density.

Wear has been categorized in various ways. The phenomenological approach is based on a macroscopic description of appearance of the worn surface. For that is suffering, rubbing fitting. But this system has its limitations as it does not focus on the mechanism of wear and therefore must rely almost entirely on imperial solutions to wear problems. Another way to categorize wear is on the fundamental mechanism that operating but this approach is complicated by the fact that more than one mechanism may be operating at a time and by the lack of sufficient information .A third type of classification describes wear based on the shape and size of the wear debris particles generated. Depending on the nature of movement of the media, wear has been classified as:

Abrasive wear	Erosive wear
Adhesive wear	Fatigue wear
Corrosion	wear Fretting

Under dry sliding condition, the dominant wear mechanism is abrasive wear under mild load and at room temperature, and the dominant wear mechanism changes to adhesive wear with the increase in load or temperature. The dominant wear mechanism of the composites under lubricated condition is microploughing, but microcracking also occurs to a certain extent

Wear loss is determined by weight loss technique. The weight loss is converted to volume loss and then wear data is plotted as cumulative volume loss as a function of sliding distance. The wear rate is calculated by using the following formula:

$$W (\text{mm}^3/\text{m}) = [M(\text{g}) / D (\text{g}/\text{mm}^3)] / \text{sliding distance (m)} \quad (12)$$

where M is the mass loss during abrasive wear, and D is the density of the respective composite

3.1 Choice of materials

The matrix material used in the present investigation was commercially pure aluminium. The iron ore was collected from Khanda Bandha OMC Ltd. Orissa, India. Commercially pure Si and Mg were added to improve the wettability.

3.2 Melting and Casting

The aluminum- iron ore metal matrix composite was prepared by stir casting route. For this we took 500gm of commercially pure aluminum and desired amount of iron ore in powder form. The iron ore particles were preheated to 300⁰C for three hours to remove moisture. Commercially pure aluminum was melted in a resistance furnace. The melt temperature was raised up to 720⁰C and by purging hexachloroethane tablets degassed it. Then the melt was stirred with the help of a mild steel turbine stirrer.

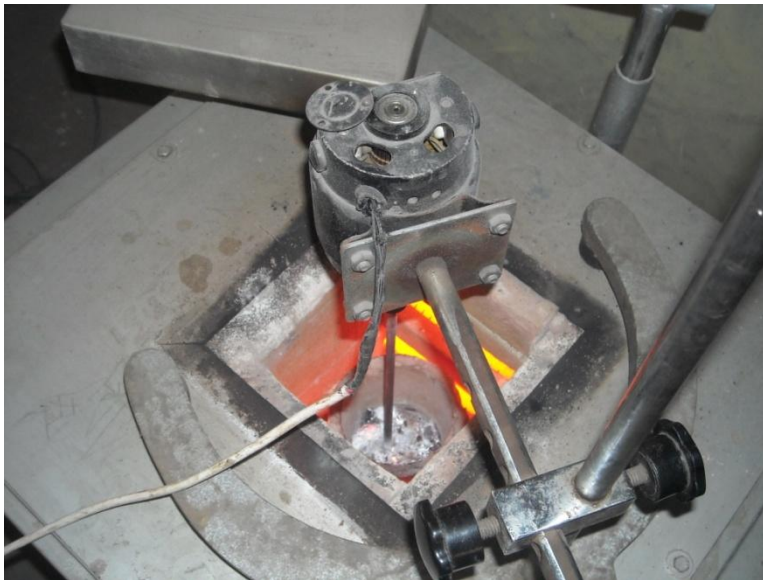


Fig.3.1 Laboratory stir casting set up

The stirring was maintained between 5 to 7 min at an impeller speed of 200 rpm .The melt temperature was maintained 700⁰C during addition of Si or Mg and iron ore particles. The dispersion of iron ore particles were achieved by the vortex method. The melt with reinforced particulates were poured into the preheated permanent metallic mold. The pouring temperature was maintained at 680⁰C. The melt was then allowed to solidify in the mould.

3.3 Particle size analysis

Particle size of the iron ore powder was measured by Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron). First, the liquid dispersant containing 500 ml of distilled water was kept in the sample holder. Then the instrument was run keeping ultrasonic displacement at 10.00 micron and pump speed 1800 rpm. The dispersant used was Sodium hexametaphosphate (15ml).



Fig.3.2 Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron).

3.4 X-Ray diffraction analysis

The composites prepared were analyzed with the help of x-ray diffraction technique to check the presence of different compounds in the composites.

3.5 Microstructural Characterization

Scanning electron microscopy: Microstructural characterization studies were conducted on unreinforced and reinforced samples. This is accomplished by using scanning electron microscope. The composite samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent. The SEM micrographs of composites were obtained using the scanning electron microscope. The images were taken in both secondary electron (SE) and back scattered electron (BSE) mode according to requirement. Microscopic studies to examine the morphology, particle size and micro structure were done by a JEOL 6480 LV scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector of Oxford data reference system. Micrographs are taken at suitable accelerating voltages for the best possible resolution using the secondary electron imaging.



Fig.3.3 JEOL JSM-6480LV scanning electron microscope

Optical microscopy: The composites produced were examined by optical microscope to analyze the microstructure. A section was cut from the castings which is first belt grinded followed by polishing with different grade of emery papers. After that they were washed and again cloth polishing of the sample was done. After etching with Keller's reagent they were examined for microstructure under optical microscope at different magnifications.

3.6 Mechanical Properties Observation

Hardness

Bulk hardness measurements were carried out on the base metal and composite samples by using standard Vickers hardness test machine. Vickers hardness measurements were carried out in order to investigate the influence of particulate weight fraction on the matrix hardness. Load applied was 10 kg and indenter used was square based diamond pyramid.

Tensile behavior

The tensile testing of composites was done on series IX automated materials testing system 1.26 manufactured by Instron Corporation. The sample rate was 9.103 pts/sec and cross-head speed 5.0 mm/min. Standard specimens with 28mm gauge length were used to evaluate ultimate tensile strength, yield strength and percent elongation.

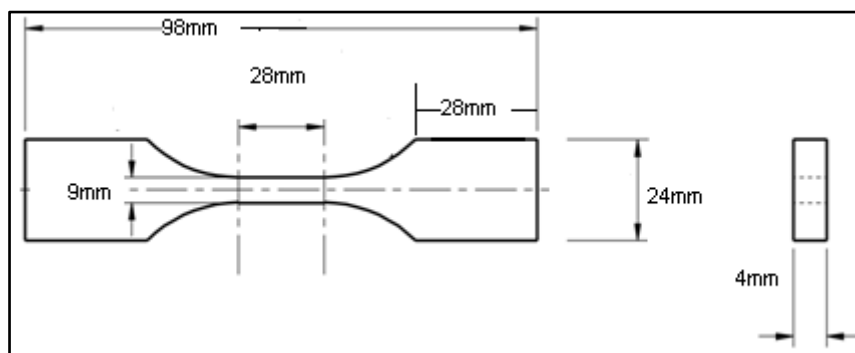


Fig.3.4 Tensile specimen

Sliding Wear Behavior

Wear has been defined as the displacement of material caused by hard particles or hard protuberances where these hard particles are forced against and moving along a solid surface. Two body sliding wear tests were carried out on prepared composite specimens. A Ducom, Bangalore made computerized pin on- disc wear test machine was used for these tests. The wear testing was carried out at different sliding velocities with normal loads of 10N, 15N, 20 N. A cylindrical pin of size 1.1cm diameter and 2.1cm length, prepared from composite casting, was loaded through a vertical specimen holder against horizontal rotating disc. Before testing, the flat surface of the specimens was abraded by using 2000 grit paper. The rotating disc was made of carbon steel of diameter 50 mm and hardness of 64 HRC. Wear tests were carried out at room temperature without lubrication for 30 min. The principal objective of investigation was to study the coefficient of friction and wear. The weights were measured before and after each test segment to determine the wear loss of each sample. Scanning electron microscopy was used to analyze the morphology of the worn surfaces of sample.

Worn surface and debris analysis

The wear debris and worn out surfaces from wear tests was analyzed with the help of optical and scanning electron microscopy to study different wear mechanisms operating on the surface.

4.1. Iron ore analysis

Composition of iron ore: Besides iron oxides, it also contains compounds of Si and Al with other elements in traces. The SEM micro-graph of the iron ore particles is shown in fig.4.1.

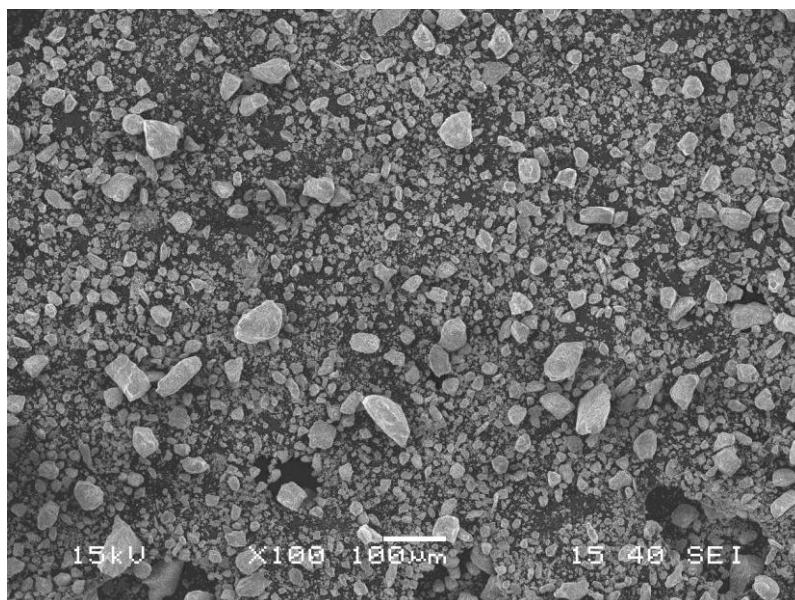


Fig.4.1 SEM Micrograph of iron ore powder used in the present investigation.

The chemical compositions of iron ore was determined by X-ray fluorescence technique at the Rourkela steel plant. The result obtained is shown in Table 1.

Table 1 Composition of iron ore used

Compounds	Percentage (%)
Fe (Total)	57.80
Fe ₂ O ₃	82.65
Al ₂ O ₃	5.10
SiO ₂	4.70

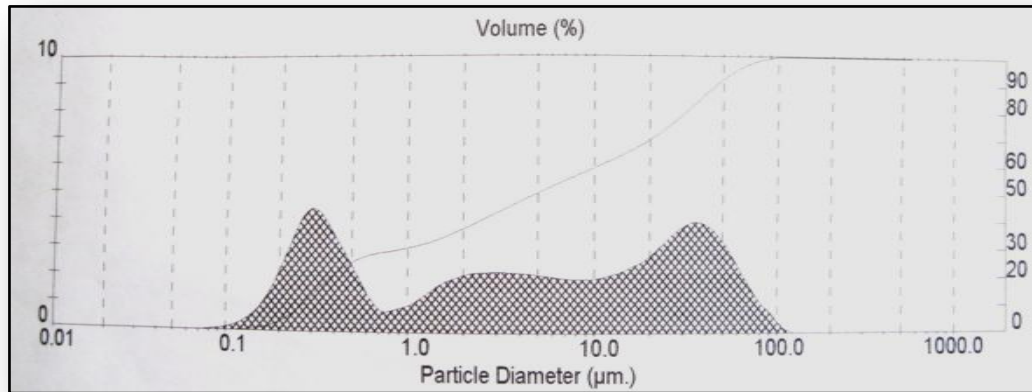


Fig.4.2 Particle size analysis of iron ore

Iron ore received from Khanda Bandha OMC Ltd. Orissa had a wide particle size distribution. The particle size of the ore as received condition, lies in the range from 0.1-100 µm. The SEM micro-graph of the iron ore is shown in fig. The average particle size is about 4.78µm.

4.2 Microstructure of cast composites

(a) Optical micrographs of MMCs



Fig.4.3 Al-10% iron ore composite

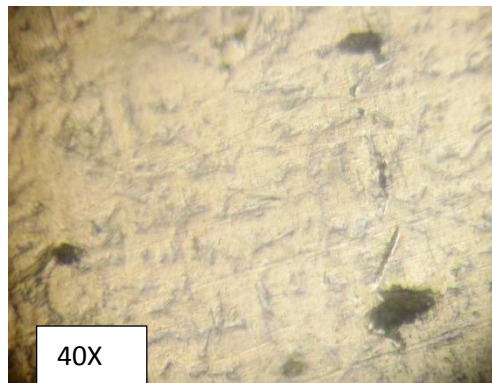


Fig.4.4 Al-2%Si-10% iron ore composite

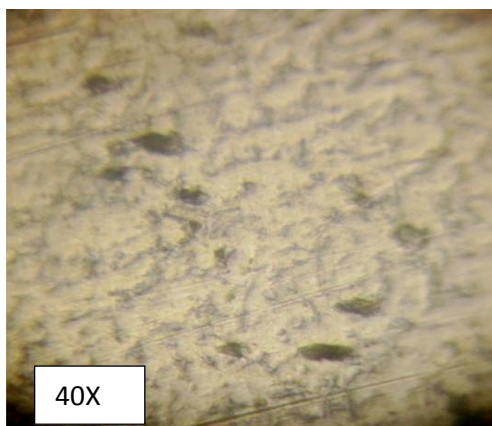


Fig.4.5 Al-2%Mg-10% iron ore composite

(b) SEM micrographs of the MMCs

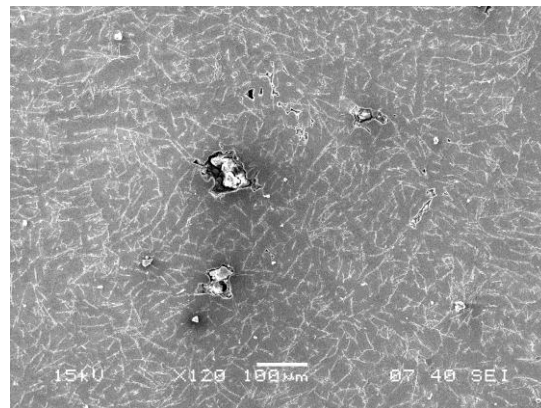


Fig.4.6 SEM of Al-10% Iron ore

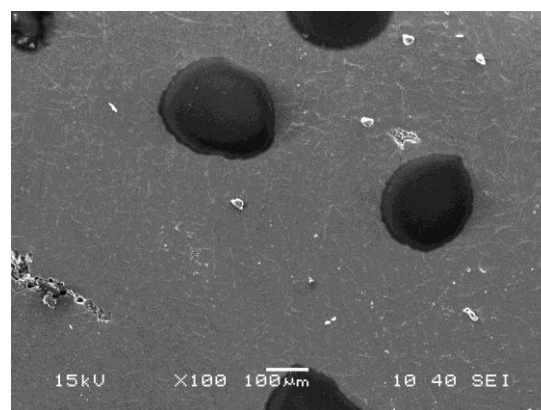


Fig.4.7 SEM of Al-2%Si-10% Iron ore

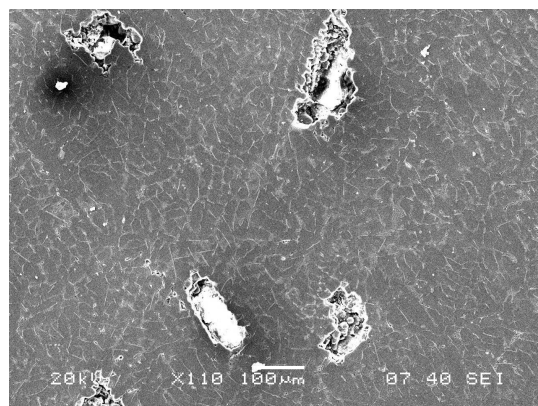


Fig.4.8 SEM of Al-2%Mg-10% Iron ore

The SEM micrographs of composites are shown in figure 4.6 to 4.8. The morphology, density, type of reinforcing particles, and its distribution have a major influence on the properties of particulate composites. The variables which govern the distribution of particles are solidification rate, fluidity, type of reinforcement, and the method of incorporation. It is necessary to distribute particles uniformly throughout the casting during production of particulate composites. The first task is to get a uniform distribution of particles in the liquid melt and then to prevent segregation/agglomeration of particles during pouring and progress of solidification. One of the major requirements for uniform distribution of particles in the melt is its wettability. Addition of magnesium and silicon improves the wettability.

4.3 Distribution of particles in particulate composites

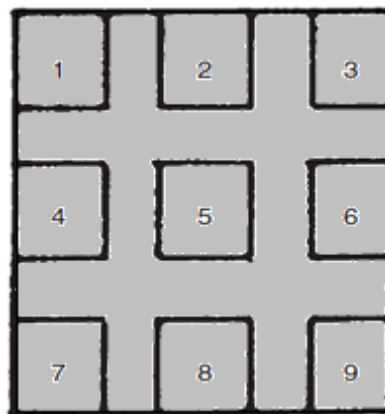
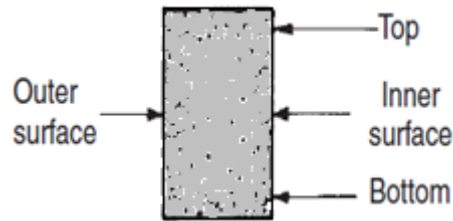
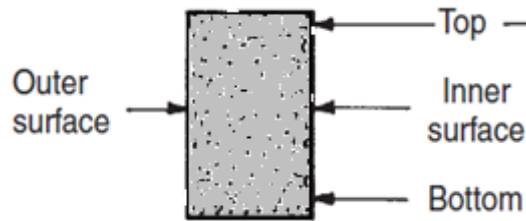


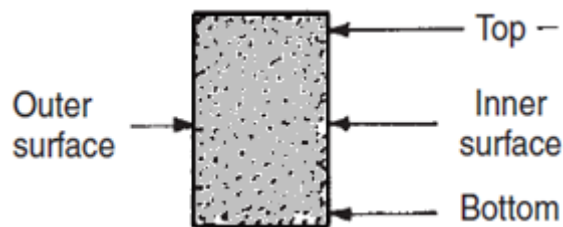
Fig 4.9(a) Different locations of selected sample



Al-10% iron ore composite



Al-2%Si-10% iron ore



Al-2%Mg-10% iron ore

Fig.4.9 (b) Macrographs showing distribution of particles

The micro structure of the samples, cut from the plate casting at different locations, were observed to study the particle distribution (fig 4.9.a). In the case of Al-10% iron ore, particles were not uniformly distributed throughout the casting. The particles were segregated at the top, bottom, and sides of the plates. The interior of the casting contained very few particles. This is due to the poor wettability and gravity regulated segregation of the particles. Whereas in the case of Al-2% Mg-10% iron ore and Al-2% Si-10% iron ore, particles were present more or less throughout the casting. The particle distribution strongly influences the physical and mechanical properties of the composites. The result shows that volume percentage of reinforcement increases with the addition of magnesium and silicon to the melt. It is probably due to better wetting conditions

4.4 X-Ray diffraction analysis

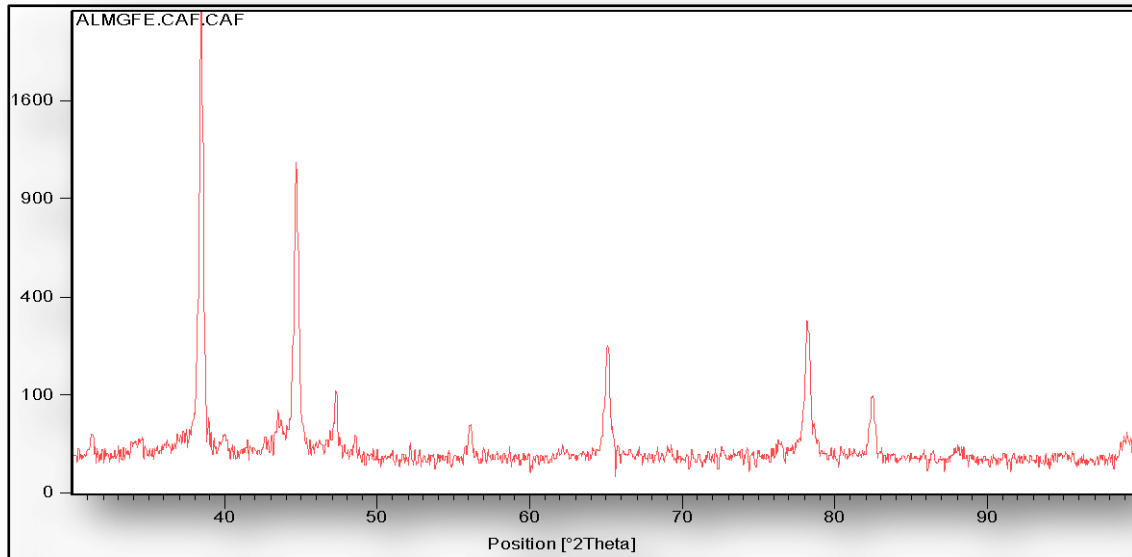


Fig.4.10 XRD pattern for Al-2%Mg-10% Iron ore composite

Table 2 Relative intensity and position [$^{\circ}2\theta$] of compounds in XRD pattern.

Pos. [$^{\circ}2\theta$.]	Rel. Int. [%]	Matched by
38.4671	100.00	Al ₂ O ₃ ; Fe ₂ O ₃ ; MgO; MgAl ₂ O ₄
44.6471	45.90	Al ₂ O ₃ ; Fe ₂ O ₃ ; MgO; MgAl ₂ O ₄ ; FeAl ₃ ; Fe
47.3013	3.87	Fe ₂ O ₃ ; FeAl ₃ ; Fe ₃ O ₄ ;
56.0456	0.78	Al ₂ O ₃ ; MgAl ₂ O ₄
65.1534	8.41	Al ₂ O ₃ ; MgAl ₂ O ₄ ; Fe
78.2038	12.04	Al ₂ O ₃ ; MgAl ₂ O ₄ ; (MgO) _{0.91} (FeO) _{0.09}
82.4715	3.57	Fe ₃ O ₄ ; MgAl ₂ O ₄ ; MgO; FeAl ₃ ; Fe

The added reactive oxides (iron ore) are expected to react with liquid metal and to produce reaction products of various types. The strengthening of the composites is influenced by the nature of the reinforcing phase. Hence there is necessity of identification of the compound produced by the chemical reaction, X-Ray diffraction studies have been used for this purpose. The XRD analysis of the Al-Mg-Iron Ore composite shows the presence of unreacted iron ore as well as some simple and complex oxides like $MgAl_2O_4$. The presence of Al_2O_3 , MgO and metallic iron indicates the possible reaction which has taken place between the iron ore and the aluminium-magnesium alloy.

4.5 SEM spot analysis

X-Ray micro analysis of the particles is given below. The analysis shows that the particles has widely varying composition. In figure 4.11 the small particles contains mostly Al, Mg and O. this appears to be a complex oxide which may have formed because of the reaction between Al_2O_3 and MgO . X-ray spot analysis at the boundary of the particles shows the buildup of iron along with Al and Mg. this is possibly because of the release of metallic iron from the iron ore on reaction with Aluminium and Magnesium. The analysis clearly indicates that there is reaction between iron ore and liquid aluminium and magnesium alloys producing oxides of aluminium and magnesium and releasing metallic iron. The elemental iron thus produced goes into the aluminium melt. The above observation is in conformity with the XRD analysis.

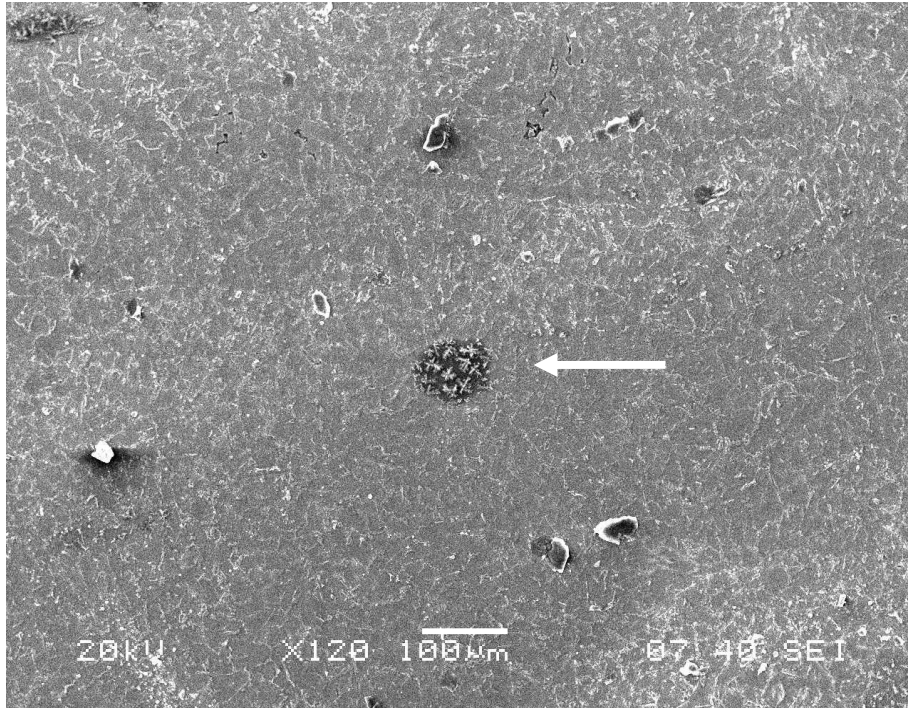


Fig.4.11 SEM micrograph showing the location for spot analysis 1
(Al-2% Mg-10% iron ore composite)

Table 3 spot analysis data 1

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
O K	55.02	1.0462	53.72	1.76	66.35
Mg K	12.31	0.8067	15.60	0.86	12.67
Al K	11.51	0.7068	16.64	0.90	12.19
Si K	6.27	0.6603	9.70	0.68	6.82
Ca K	2.42	0.9595	2.58	0.36	1.27
Ti K	1.03	0.8088	1.31	0.35	0.54
Fe K	0.37	0.8230	0.46	0.41	0.16
Totals			100.00		

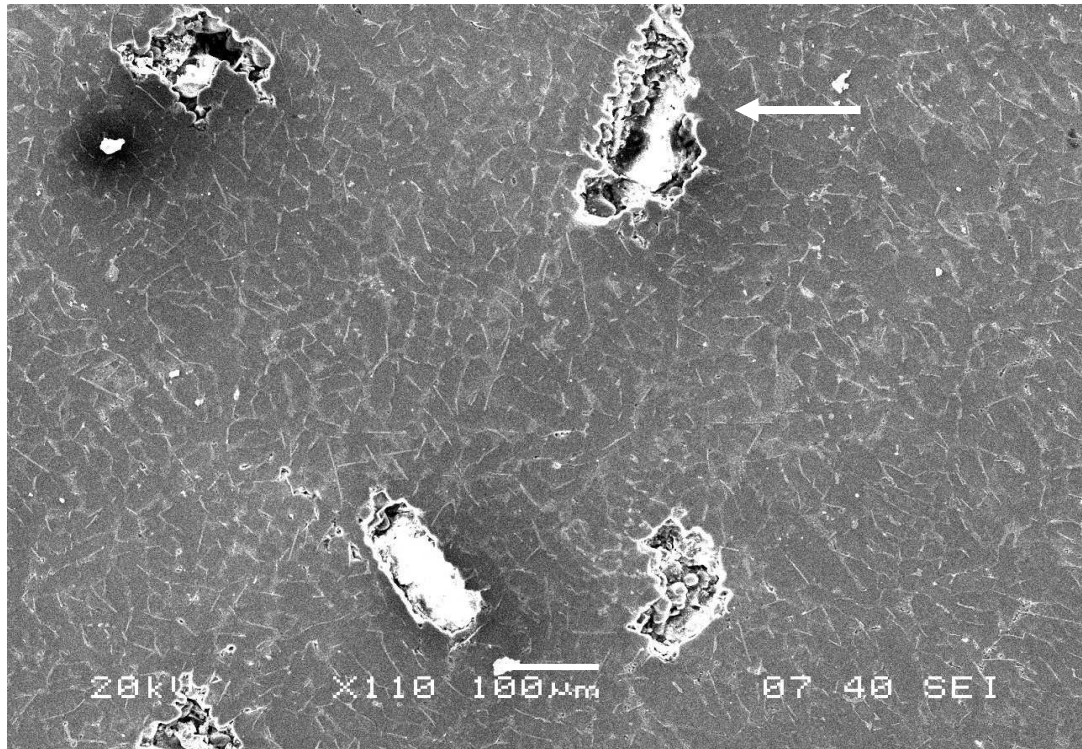


FIG 4.12 SEM micrograph showing the location for spot analysis 2
(Al-2% Mg-10% iron ore composite)

Table 4 spot analysis data 2

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
O K	32.96	1.3715	49.54	9.08	63.70
Mg K	7.18	0.7442	19.87	5.20	16.82
Al K	6.40	0.6321	20.86	5.28	15.90
Fe K	3.96	0.8401	9.72	6.68	3.58
Totals			100.00		

4.6 Mechanical Properties

Hardness

Table 5 Vickers hardness test

Sample	Average Hardness (Hv)
As cast Al	44
Al-5% iron ore	53
Al-2%Mg-5% iron ore	71
Al-10% iron ore	56
Al-2%Si-10% iron ore	84
Al-2%Mg-10% iron ore	93

The above table shows that there is significant improvement in the hardness with addition of iron ore. The increase in hardness value is more in case of Al-2%Mg alloys with reinforcement as compared to 2% Si addition. This is may be due to the higher volume percentage of the reinforcement. In the case of alloys added with Mg there is a possibility of MgO and $MgAl_2O_4$ formation and possibly these are much finer and give greater strengthening in the composite.

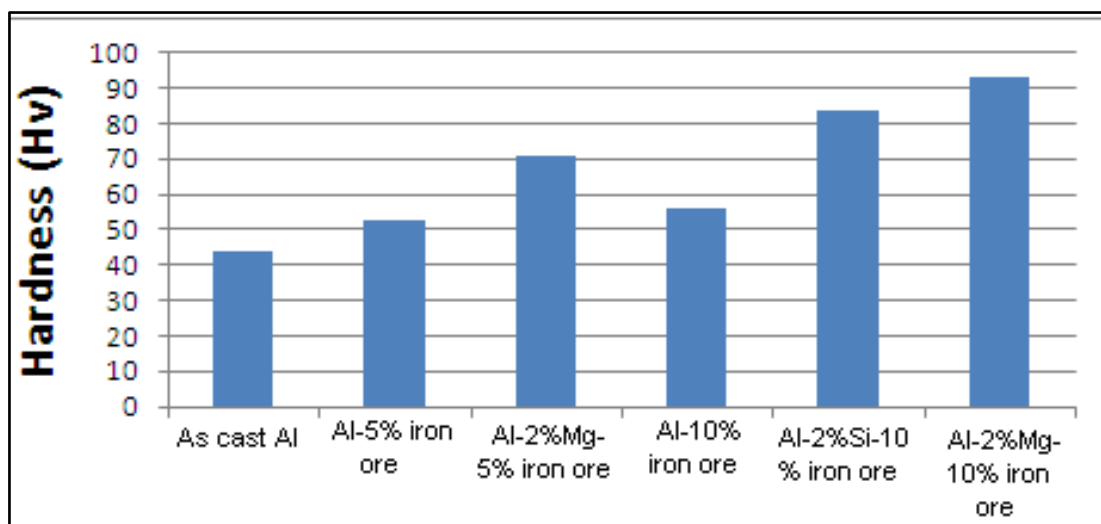


Fig.4.13 Graph showing variation in hardness with composition of MMCs

Tensile properties of composites

Table 6 Tensile properties of composites

Sample	Yield strength (MPa)	U.T.S. (MPa)	Percent elongation
6063 Al alloy (theoretical)	131.00	18.00
Al-10% iron ore	82.48	243.70	6.107
Al-2%Si-10% iron ore	140.70	249.10	3.968
Al-2%Mg-10% iron ore	277.41	383.20	2.589

From the above table it is clear that addition of iron ore leads to improvement in the ultimate tensile strength of the aluminium alloy. The addition of Magnesium improves the strength of the composites significantly.

The composites prepared in the present study are reinforced with particles of variety of simple and complex oxides as seen from the XRD analysis given above. The size range of the particles is also very wide as compared to the composites investigated earlier. The size varies from the very fine ones of 0.1 μ m to the coarse one of 100 μ m. The size range of the iron ore particles present indicates that the composite prepared can be considered as dispersion strengthened as well as particle reinforced composite. Thus the strengthening of composite can be due to dispersion strengthening as well as due to particle reinforcement. Dispersion strengthening is due to the incorporation of very fine particles, which help to restrict the movement of dislocations, whereas in particle strengthening, load sharing is the mechanism. The improvement in strength may also result from the dissolution of iron released on aluminothermic reduction of iron oxides present in iron ore. Though, it is very difficult to quantify the contribution of these factors in improvement of strength.

4.7 Dry sliding wear behavior

Wear behavior of different composite was studied with different parameter like sliding velocity and applied loads. There result and discussion are given in the following subsections:

Effect of sliding distance- Figure shows the relationship between weight loss and sliding distance..

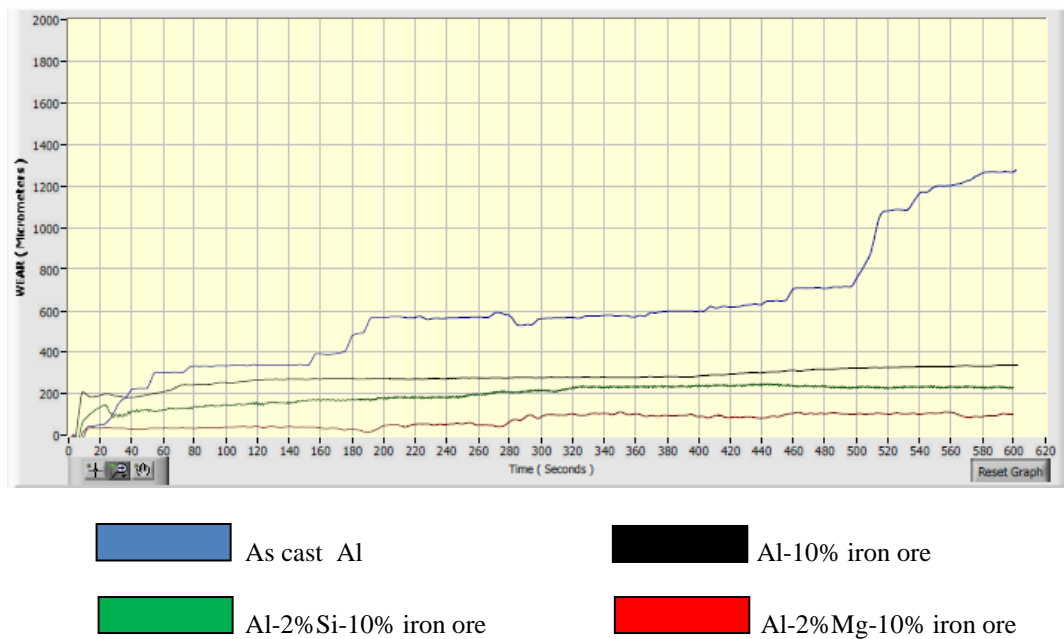
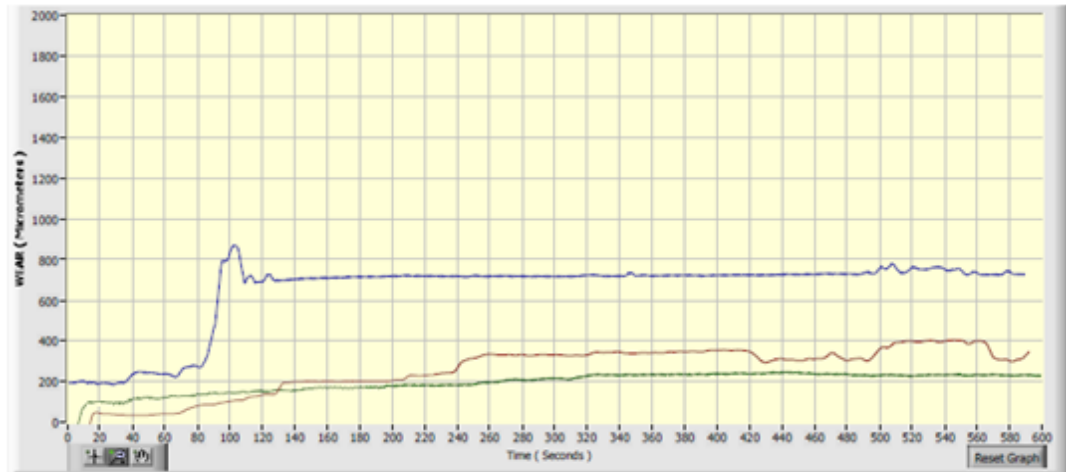


Fig.4.14 Wear behavior with different composition under normal load of 10N

Figs. 4.14 and 4.15 shows wear curves of MMCs specimen with 10% iron ore, 10% iron ore with 2% Si and 10% iron ore with 2% Mg content with normal load of 10N and 20N. All the MMCs showed a very small initial nonlinear wear regime. After a certain sliding distance, the wear has increased linearly with time indicating steady state wear regime. The transition from initial wear regime to steady state regime has taken place within few minutes (2–3 min) of commencement of the test. From the graph (Fig.4.14) it is evident that the wear resistance of composite is much greater than the commercially pure aluminium. Bulk wear decrease with addition of magnesium.

Incorporation of iron ore content significantly reduces wear. This evidence from the amount of wear observed for commercially pure aluminum and composite with 2%Mg10%iron ore

content. This is because of the presence of hard iron ore particle which will increase the overall bulk hardness.



Al-10% iron ore Al-2%Si-10% iron ore Al-2%Mg-10% iron ore

Fig.4.15 Wear behavior with different composition under normal load of 20N

In the initial wear regime, the reinforced particles act as load carrying elements and as inhibitors against plastic deformation and adhesion of matrix material. In the later stages of wear regime, the worn particles get dislodged from their positions in the matrix and get mixed with the wear debris. The wear debris containing matrix material, worn particles and iron from the disc get pushed into the craters formed by dislodging of particles and act as load bearing elements.

Effect of variation in sliding velocity

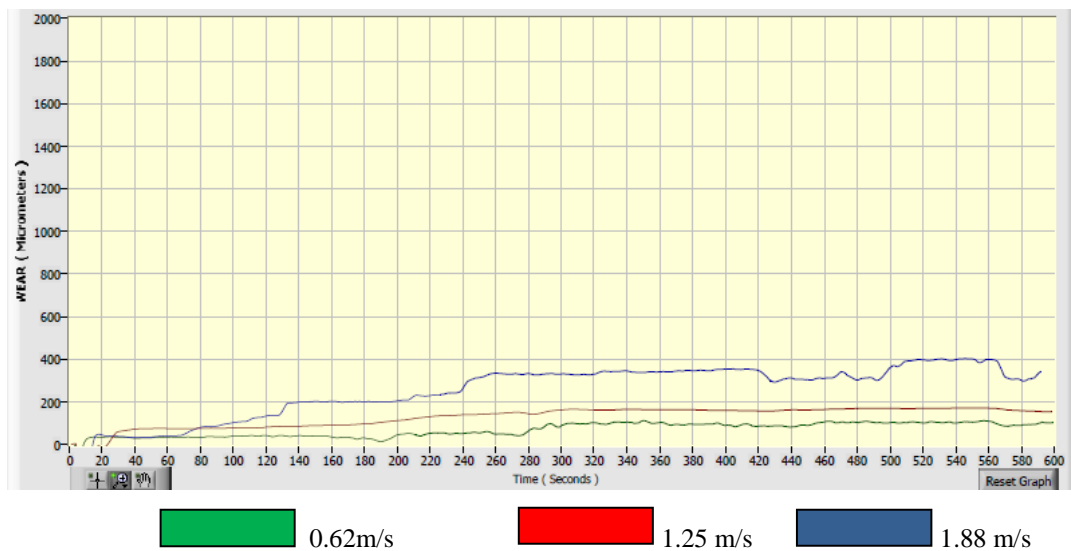


Fig.4.16 Wear behavior with variation in sliding velocity at constant load (20N)

Variation in sliding velocity was achieved by varying rotational speed of the disc with different rpm keeping track diameter constant. The wear of the composites are significantly increased with increase in sliding velocity.

Effect of variation in normal load.

Fig.4.17 shows the relation between applied load and wear of the MMC (Al-2%Mg-10%Iron ore) produced for a particular velocity of 1.25 m/sec. The amount of wear has been increased with increase in normal load. Like other Al composites, Al- iron ore composites also have an increasing trend of wear with applied load due to deformation and generation of cracks within oxide films that might acts as a three body wear on removal of the particles, thereby increase the wear rate at higher loads.

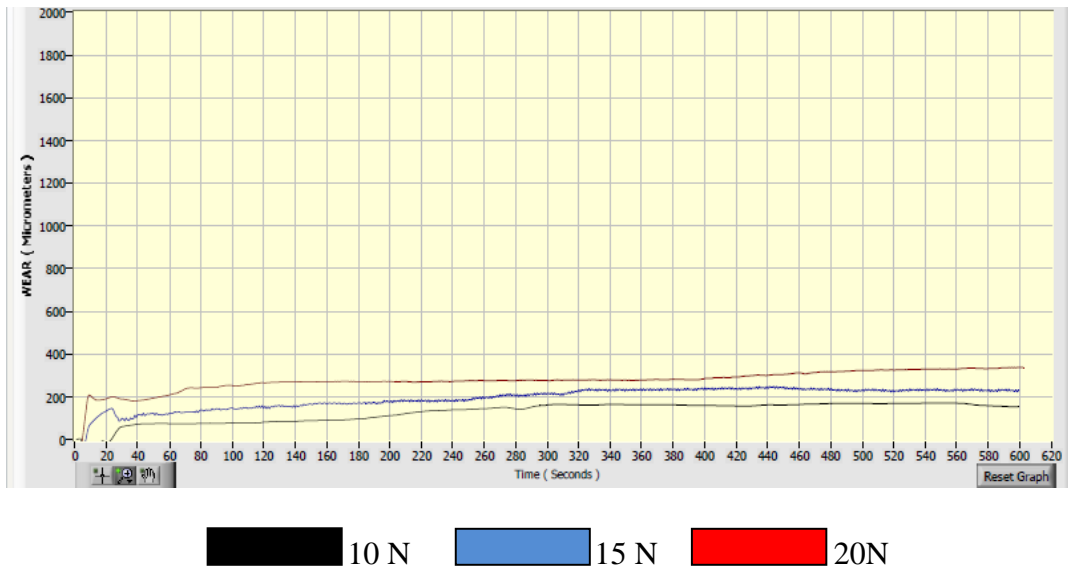


Fig.4.17 Wear behavior with variation in normal load at constant velocity.

Frictional characteristics

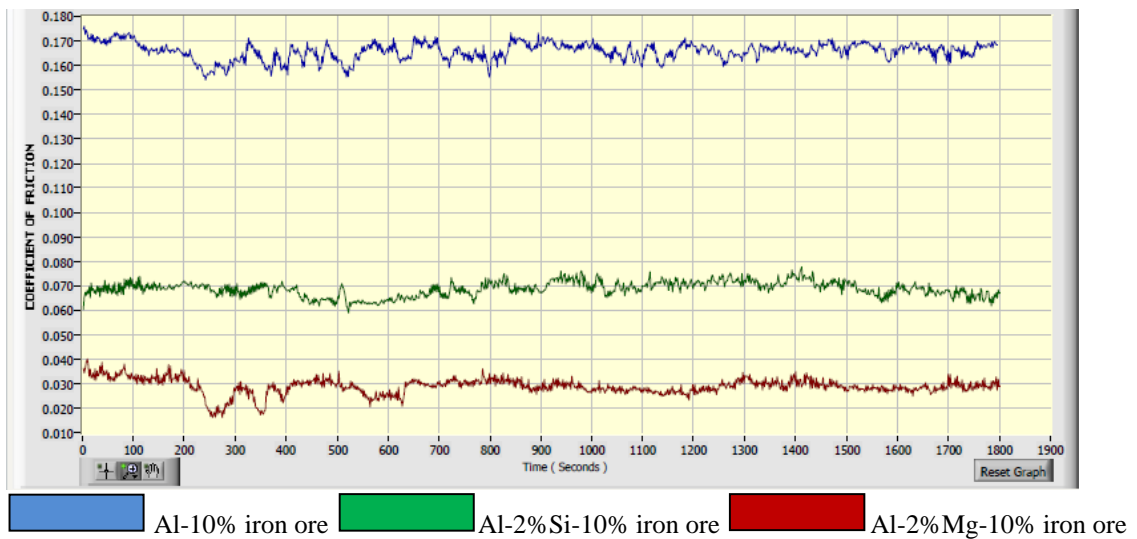
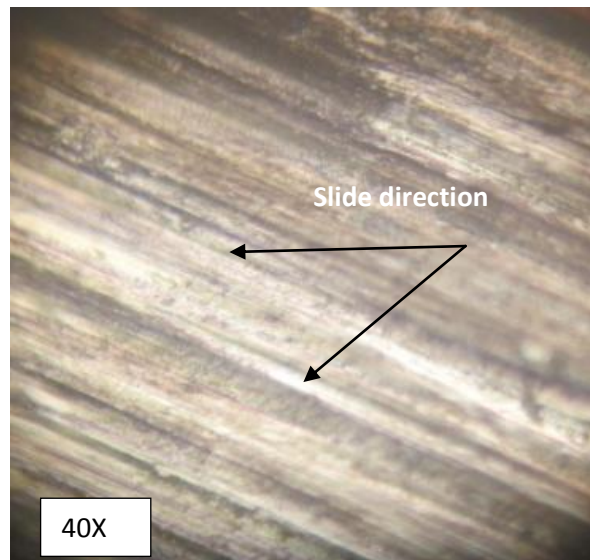


Fig.4.18 Variation of coefficient of friction with composition of the composite

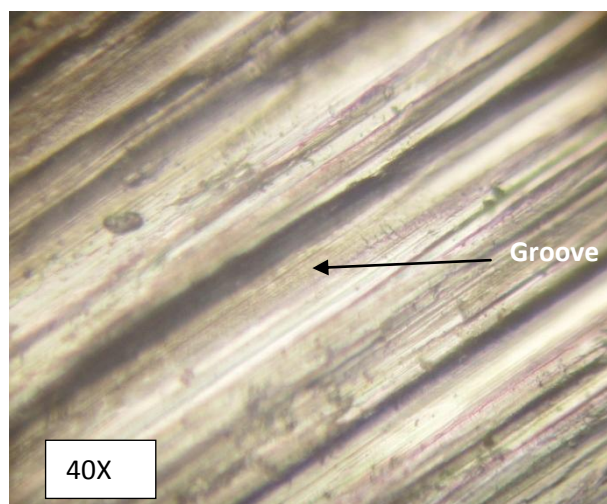
The coefficient of friction has decreased with increased iron ore as evident from Fig.4.18. In the test, normal load of 10N and sliding velocity of 1.25 m/sec were used. Al-2%Mg-10% iron ore showed lowest coefficient of friction (as low as 0.02), whereas Al-10% Iron ore showed the highest coefficient of friction(as high as 0.175) .The presence of reinforcing particles will reduce the friction by providing point contact between counter face and pin. The volume of the reinforcing particle is highest in case of Al-2%Mg-10% iron ore composite and lowest in the case of Al-10% iron ore.

4.8 Wear mode and mechanism

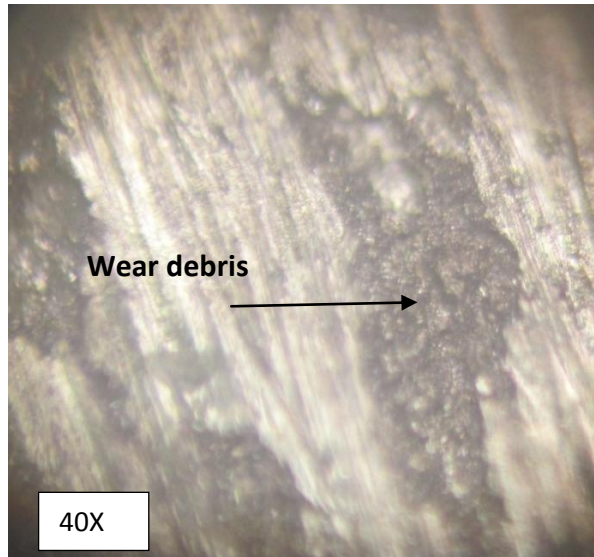
There is a continuous removal of material from the surface, whenever there is a sliding motion between two surfaces in contact. The extent may vary depending on various factors like type of material in contact, the applied load, sliding velocity, temperature, moisture, etc. In the present work, both the surfaces are different and the material undergoes deformation upto sub-surface level starting from asperities present.



(a) micrograph showing grooves and scratch marks

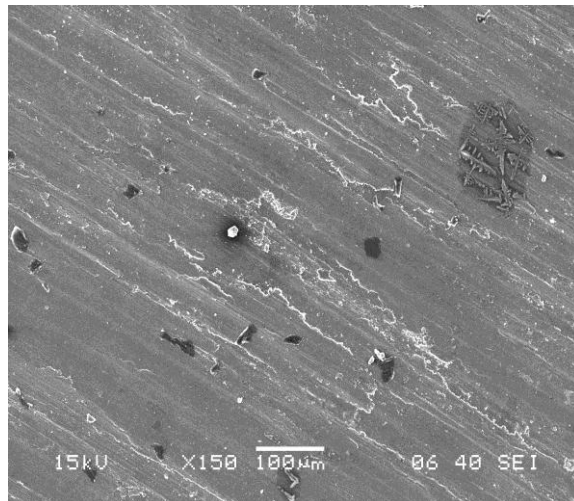


(b) Deep and large grooves under higher loads

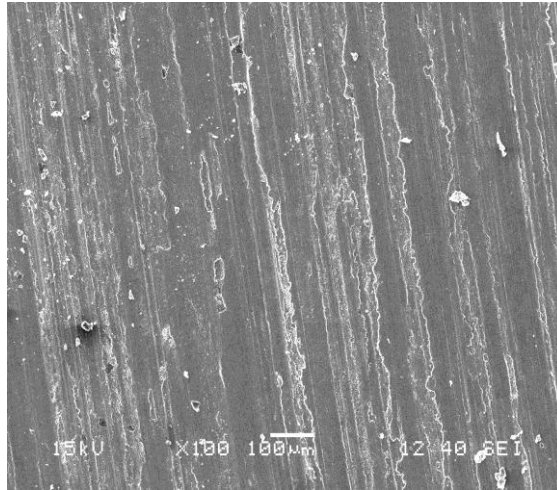


(c) Wear debris filling the valleys in the wear surface

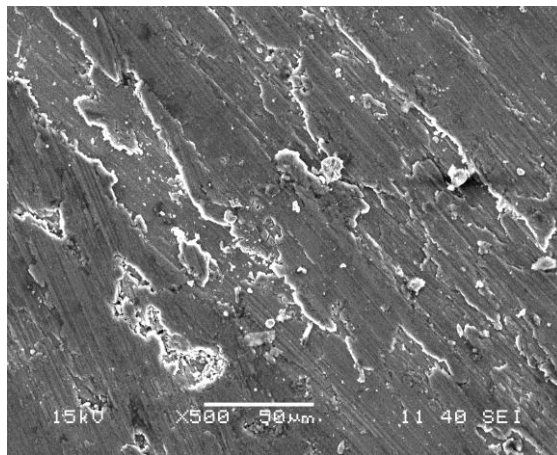
Fig.4.19 Optical micrograph of worn surface of Al-2%Mg-10% iron ore.



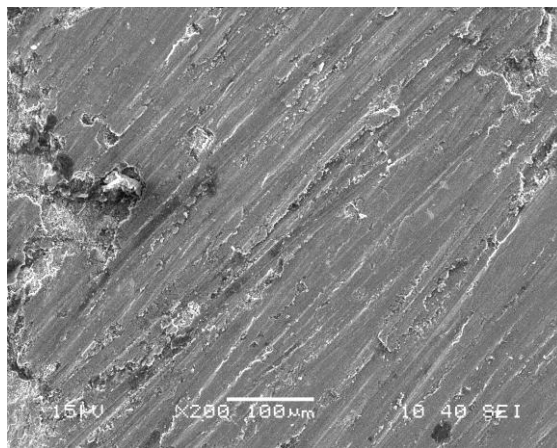
(a) Al-iron ore 200rpm 10N



(b) Al-iron ore 300rpm 10N



(c). Al-Mg-iron ore at 300rpm 20N



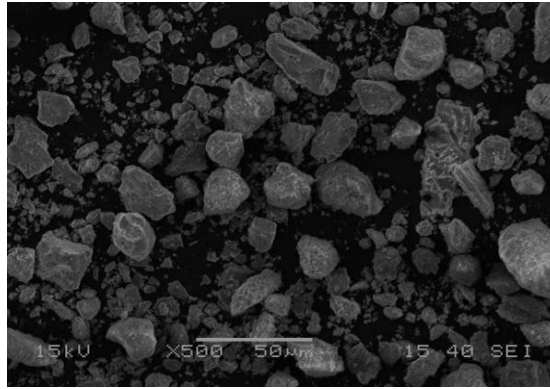
(d). Al-Mg-iron ore at 400rpm 20N

Fig.4.20 SEM micrograph of worn surface of composites

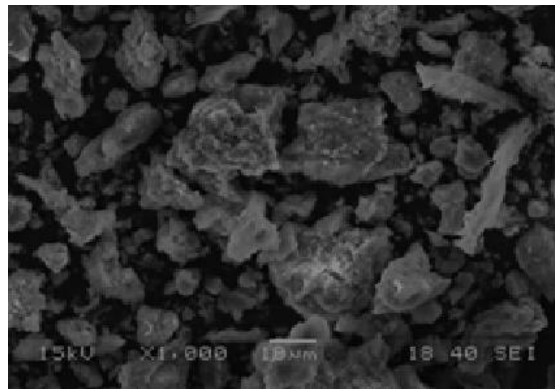
The study of the worn surfaces of aluminum–iron ore composites under optical and scanning electron microscopy shows that at lower load, the worn surface has relatively less ploughing and cutting, as shown in fig.4.20a. However, at higher loads and higher sliding velocities, fractured particles are frequently present on the worn surface (fig.20c and 20d)

The study of wear tracks at different applied loads and different sliding velocities shows broken oxide films on wear tracks, deep grooves and delamination of surface. The wear rate was observed to decrease continuously with increase in hardness of the composite. Wear properties of Al-Mg-Iron ore composites are found to be superior than that of as cast Aluminium and Al-Iron ore composites

The results clearly indicates the accumulation of strain in the sliding surface and subsurface level which causes plastic deformation and nucleation and growth of microcracks which finally becomes the source of wear failure and hence loss of material at the interface. Optical and scanning electron microscopic examination of the worn pin surfaces identified different wear mechanism operating under various sliding conditions such as abrasion, oxidation and delamination.



(a)



(b)

Fig.4.21 SEM micrograph of wear debris at different loads (a) 10N and (b) 20N

The characteristics of the wear debris generated during the wear tests have been studied in detail. The scanning electron micrograph of the wear debris in fig.4.21 shows fine particles at low load and large flaky particles at higher loads. The debris varies markedly in color, size, shape and composition. The material during wear testing, due to sliding motion eventually produces wear debris. If the stress produced in the material during sliding motion is more than the fracture stress of the particles, they lose their ability to support the load. The direct contact of the aluminium matrix with the counter face imposes large plastic strains on the pin surface. This gives rise to crack formation. Delamination of the wear surfaces also contributes to debris formation.

The following conclusions may be drawn from the project work:

1. Iron ore upto 10% by weight can be successfully added to aluminium by vortex method to produce composites that can be die cast.
2. There is appreciable reaction between the iron ore and the melt producing finer particles of complex oxides.
3. The oxide particles and the unreacted particles of iron ore gives sufficient improvement in strength and hardness of the composite.
4. Strengthening of composites is due to particle reinforcement, dispersion strengthening and solid solution strengthening.
5. Addition of Mg and Si improves the wettability of iron ore with aluminium melt and thus increase the amount of reinforcing phase in the composite.
6. The wear resistance has improved significantly by addition of iron ore.
7. The effect of increased reinforcement on the wear behaviour of the metal matrix composites is to increase the wear resistance and to decrease the coefficient of friction.
8. The metal matrix composites shows better wear resistance due to its superior load bearing capacity.
9. Increase in normal load and sliding velocity increases the magnitude of wear and frictional force.
10. Different wear mechanisms were observed at different normal loads, compositions and sliding velocities.

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