

CHAPTER ONE

1. INTRODUCTION

1.1 ORIENTATION OF THE PROPOSED STUDY

The use of natural composites has been a part of man's technology since ancient time with the use of straw to reinforce mud bricks (straw bricks). However, in the past few decades, there has been rapid development and revolution of composite materials with enhanced properties (Bernadette and Tim, 2002). This versatile material system has become part of everyday life and are widely used in various application. Modern composite materials constitute a significant proportion of engineered materials ranging from everyday products to sophisticated niche applications. On the basis of enhanced properties of composites materials, they are able to account for the weaknesses and limitations of conventional materials (metals, polymers and ceramics), thereby making them useful in various applications (Brian Harris, 1999).

Metal-matrix composites (MMCs) are combinations of a metal or an alloy with a finite fraction (by volume or by weight) of second phase, generally ceramic, that is deliberately introduced into the metal in order to improve its properties. MMCs are a class of materials with potential for a wide variety of structural and thermal applications. They can be tailored to give improved strength, stiffness, thermal conductivity, abrasion resistance, creep resistance, or dimensional stability. The successful application of MMCs requires adequate corrosion resistance as well. Metal matrix imparts a metallic nature to the composite in terms of thermal and electrical conductivity, manufacturing operations and interaction with the environment (Bobic *et al.*, 2009). Metal Matrix Composites are usually developed using Al alloys, Mg, Cu and Zn-Al alloy among others.

Zn-Al based composites have continued to find relevance in several technological applications (Can Kurnaz, 2003). The Zn-Al alloys, which serve as the matrix for this class of MMCs, are known for their good combination of physical, mechanical and technological properties. High strength, excellent castability, good machinability, low melting point and good tribological properties, as well as low manufacturing cost are among its notable characteristics (Hekimoglu and Savaskan 2014; Zhu *et al.*, 2003). They have shown satisfactory service performance when used for the design of components such as bearings, dies, punches and seals which require high mechanical and wear resistance (Bobic *et al.*, 2009). The inability of Zn-Al based alloys to work effectively above operating temperatures of 80 degrees Celsius has been a sour limitation to its application for several other purposes.

The use of reinforcements such as silicon carbide (SiC) and alumina (Al_2O_3) has been explored in order to take advantage of Zn-Al alloys base properties and this has resulted in marked improvement in hardness, strength, specific strength, wear and creep resistance of Zn-Al based composites (Bobic *et al.*, 2014; Mitrovic' *et al.*, 2007). The problem of machinability of Zn-Al based composites has been improved on (without any deleterious effect on mechanical and tribological properties) by the use of graphite as complementing reinforcement to SiC and Al_2O_3 (Mitrovic' *et al.*, 2011).

Currently, Zn-Al alloys are being reinforced using waste products derived from industrial processes (red mud, fly ash, quarry dust) and agro based materials (rice husk ash, bamboo leaf ash, groundnut shell ash among others) (Alaneme and Adewale, 2013). All the aforementioned advantages are also exhibited by these categories of composites and have made Zn-Al alloy based composites top choice candidate for a wide range of engineering applications.

The development of reliable material property (mechanical properties, wear and corrosion behaviour) database for Zn-Al alloy based composites newly developed with the use of industrial waste as hybrid reinforcing materials (to either silicon carbide or alumina) is highly important in order to determine most suitable areas of application and limitations of the composites. To this end, there has been efforts to generate material properties data for a number of Zn-Al alloy based composites developed with the use of industrial waste reinforcement (Alaneme and Ajayi, 2015). Sparse information has been generated on the material property of Zn-Al alloy based composites reinforced with industrial waste such as fly ash, quarry dust among others but little work has been done in generating reliable material property data for Zn-Al alloy based composites reinforced with steel machining chips.

A success in this area has the potential of offering reduced composite production cost, additional channel for industrial waste recycling, while still maintaining the technical efficiency and performance levels of conventional Zn-Al based hybrid composites.

1.2 STATEMENT OF THE PROBLEM

The low creep, mechanical and wear resistance exhibited by Zn-Al alloy based composite at temperatures above 100 °C has been a major problem of concern in engineering, also the high cost of production of Zn-Al based composite reinforced with silicon carbide and alumina (which are produced outside Nigeria) which is to bridge the limitations of Zn-Al alloy has been of concern.

On account of this, the use of agro waste as secondary reinforcement in the development of Zn-Al alloy based composites was explored so as to reduce the production cost while maintaining high performance level of the composites. A limitation for the use of agro waste is the preparation of the waste which involves burning, conditioning and sieving before use as reinforcements, these preparation processes takes longer time. To this end, the use of industrial waste (which require less processing) as a secondary reinforcement in the development of composites is being explored.

Therefore, this study explore the use of steel machining chips as a suitable reinforcement in the development of low cost, high performance Zn-Al alloy based composites.

1.3 BACKGROUND OF THE RESEARCH

Researchers all over the world are focusing on ways to utilize either industrial or waste as a source of reinforcement in replacing the conventional reinforcement materials used for composites production. In view of this, several work had been done on Zn-Al alloy reinforced with light weight, high yield strength and low density materials.

Alaneme and Ajayi (2015) reported that the fracture toughness of the composites increases with increase in the weight percent of rice husk ash (and corresponding decrease in SiC weight percent) and some of the composite compositions containing rice husk ash had slightly higher % elongation values compared with the composite grade without rice husk ash.

According to the results obtained by Bobic *et al.* (2009), the corrosion resistance of MMCs with zinc-aluminium alloy Zn-27Al substrate increases with the enhancement of reinforcing phase content in composite material. The MMCs corrosion rate was found to decrease with time.

Prakash and Pruthviraj (2011) studied Hybrid Zn-Al-SiC-Graphite composites have prepared by liquid metallurgy route and concluded that the hybrid composites possess values of hardness, coefficient of friction in between that of Zn-Al-SiC and Zn-Al-graphite composites but possess excellent wear resistance when compared with the monolithic composites.

Jaglinski and Lakes (2011) successfully made composites with a zinc–aluminum (Zn-Al) alloy matrix and silicon carbide particles and observed that these composites exhibited a favorable combination of stiffness and viscoelastic damping. Composites of Zn-Al-SiC were stiffer than the alloy matrix and exhibited higher damping at acoustic frequencies than the alloy Matrix. Iglesias *et al.* (2013) observed that steel machining chips increased the wear resistance of metal alloys (Zn based alloy system) not only at room temperature but also at

elevated temperature and under severe contacts conditions. These investigations emphasize that the use of multiple reinforcements in Zn based matrix hybrid composites yields better tribological properties, However, little information is provided in terms of the mechanical properties and corrosion behaviour of Zn-Al alloy reinforced with steel machining chips, therefore, the need for this research, which is focused on studying the mechanical properties, corrosion and wear behaviour of Zn-Al alloy reinforced with steel machining chips.

1.4 AIM OF THE RESEARCH

The aim of this research is to assess the potentials of steel machining chips and graphite as reinforcements in the development of high performance, low cost Zn-27Al alloy based hybrid composites.

1.5 OBJECTIVES OF THE RESEARCH

The specific objectives of the research are to:

- a) produce Zn-Al based metal matrix hybrid composite reinforced with different weight ratios of graphite and steel machining chips using double stir casting method;
- b) assess the effect of the mix ratios of the reinforcements on the microstructure and percentage(%)porosity of the composites produced in (a);
- c) evaluate the mechanical (tensile properties, hardness and fracture toughness), wear and corrosion behaviour of the composites assessed in (b); and
- d) determine the best reinforcement mix ratio that yields enhanced mechanical, wear and corrosion properties in the composites produced.

1.6 JUSTIFICATION OF THE RESEARCH

The research and development of Zn-Al alloy based composites reinforced with conventional particulates such as silicon carbide and alumina is still at evolving stage in Nigeria based on recent investigations. This is due to the high cost of procurement of silicon carbide and alumina as reinforcement to be used in the production of Zn-Al alloy based composites. The proposed study intends to investigate the use of steel machining chips which is an industrial waste with low cost implications. The recycling of steel machining chips for other purposes is presently a problem creating waste management challenges. A research centered on investigating the potentials of productive use of steel chips as reinforcement of Zn-Al alloys is therefore worthwhile.

1.7 EXPECTED CONTRIBUTIONS TO KNOWLEDGE

It is envisaged that at the completion of this research, it will:

- a) Extend the Frontiers of knowledge in the science of metal matrix composite development.
- b) Profer a scientific basis for the utilization of steel machining chips (an industrial waste) for low cost development of Zn-27Al alloy matrix composites which serves as a viable alternative for recycling of industrial waste for productive use.

1.8 SCOPE OF THE RESEARCH

The scope of this research will cover:

- a) Zn-27Al based hybrid composite with a single reinforcement weight percent but with five weight ratios of graphite and steel machining chips will be produced.
- b) Tensile properties, hardness and fracture toughness of the produced composites will be the mechanical properties to be evaluated.
- c) Corrosion resistance test of the composites produced would be performed using single concentration of NaCl solution only.
- d) The wear resistance of the composites produced would be evaluated using the abrasion test method only.
- e) Microstructural analysis of the composites will be limited to the use of Optical and Scanning Electron Microscopes.

CHAPTER TWO

2. LITERATURE REVIEW

2.1 COMPOSITES OVERVIEW

A composite can be defined as material system made up of suitably arranged combination of two or more nano, micro, or macro constituents with an interface separating them that differ in form, chemical composition and are essentially insoluble in each other (Himanshu *et al.*, 2014). They are produced when two or more materials with different physical and chemical properties are combined to produce a new material that has improved properties when compared with the individual constituent materials.

Generally, a composite function is made up of two or more chemically distinct phases on a microscopic scale, separated by a distinct interface which are the matrix and the reinforcement and it is important to be able to specify these constituents (<http://www.owenscorning.com/composites>). In a metal matrix composite, three important features determine its characteristics that is, the matrix, the reinforcement, and the matrix/reinforcement interface.

Matrix is the monolithic material into which the reinforcement is embedded and it's a viscous material that hardens to give shape to the composite product and support for the reinforcement (www.scribd.com/). The matrix surrounds and binds together a cluster of fibres or fragments of a material with different properties (the reinforcement). It is completely continuous and usually a lighter metal such as Aluminium, Magnesium, Titanium or alloys. (<https://pt.scribd.com>). The matrix is required to fulfil several functions depending on the reinforcement such as to retain the composite mass in a solid form for a particulate aggregate and for fibre composites, the matrix can serve as load-transfer medium, separate the fibres from each other in order to realise the advantage of the fibre aggregates, it protects the reinforcing filaments from mechanical damage and environmental attack, it also increases the toughness of the composite through the quality of its grip on the fibres.

Reinforcement: - the reinforcement is the material that provides strength, rigidity and the ability to carry a load. It is also known as the discrete constituent.

The limitations of conventional materials brought about the production and use of composites and these limitations are presented in Table 2.1.

Table 2.1: Limitations of conventional materials (www.cantab.net/)

S/n	Property	Ceramic	Metal	Polymer
1.	Density	Low	High	Lowest
2.	Hardness	Highest	Low	Lowest
3.	Ductility	Low	High	High
4.	Wear resistance	High	Low	Low
5.	Corrosion resistance	High	Low	Low
6.	Thermal conductivity	Mostly low	High	Low
7.	Electrical conductivity	Mostly low	High	Low

Properties of composites: the properties of composites depend on factors such as properties of phases, geometry of dispersed phase (particle size, distribution, orientation) amount of phases.

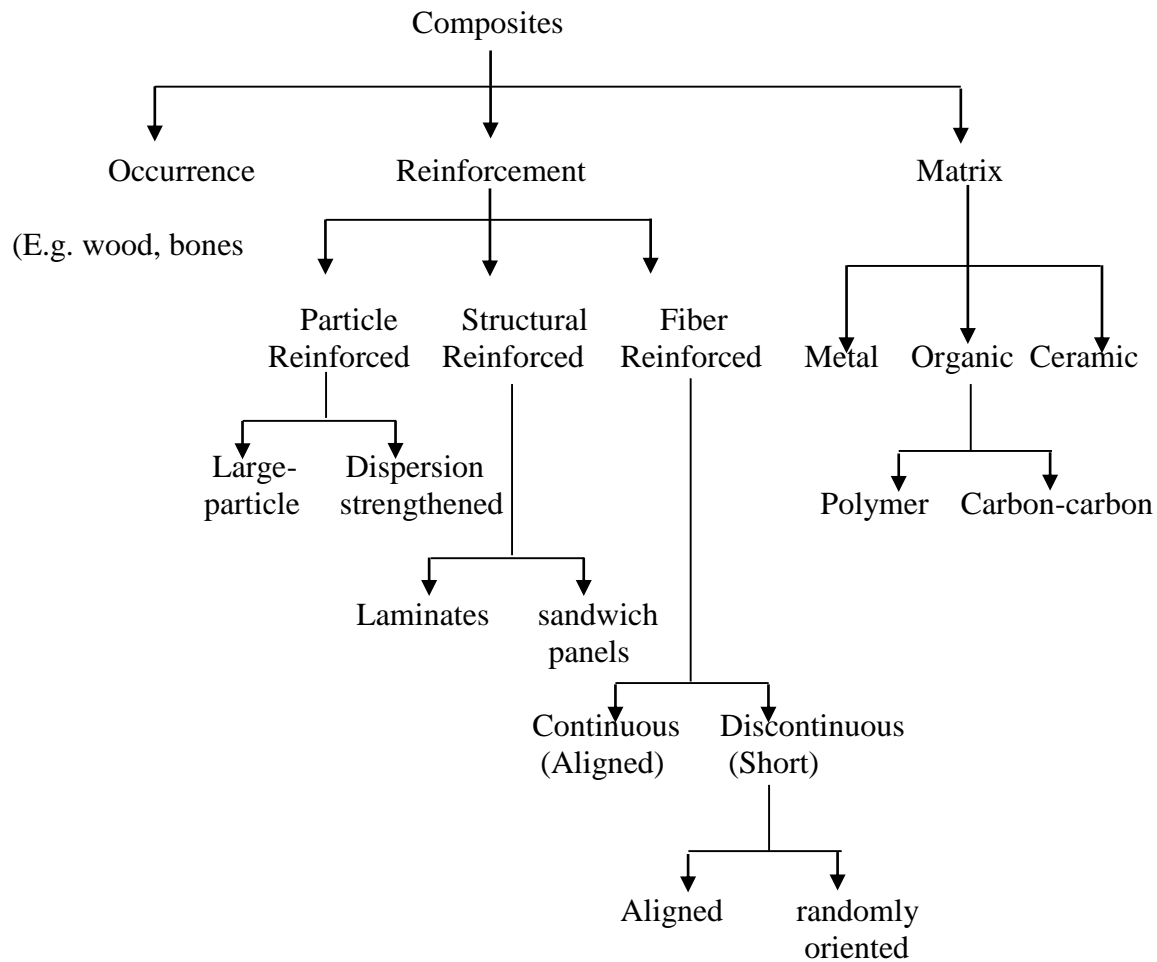


Figure 2.1 Classification of composites (Callister, 2007).

Some of the advantages of Composite materials includes light weight, corrosion resistance, part consolidation, dimensional stability, corrosion resistant, high strength-to-weight ratio, high impact strength, high electric strength (insulator), low maintenance, long-term durability, rapid installation. Some of the limitations or setbacks of composites materials includes high cost of fabrication and materials, more brittle than wrought metals, easily damaged, they cannot be easily disposed or reused, transverse properties may be weak.

Composites due to their remarkable characteristics has found applications in various aspects such as buildings, bridges; structures such as boat hull, swimming pool panels, race car bodies, shower stalls, bathtubs, storage tanks, imitation granite and cultured marble sinks and countertops; also in advanced examples perform routinely on spacecraft and aircraft in demanding environment.

2.2 CLASSIFICATION OF COMPOSITES

Ananda and Sudipt (2008) classified composite materials based on matrix and the structure of the material (reinforcement). This classification of composites is based on matrix and reinforcement.

2.2.1 Based on Reinforcement

The major composite classes based on reinforcement form are:

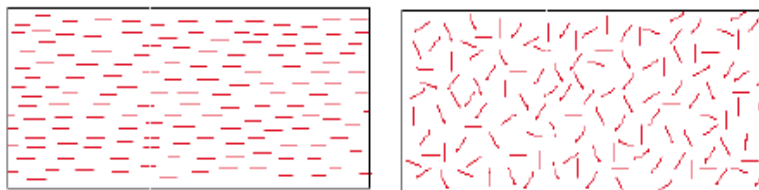
(a) Fiber Reinforced Composites

Fibre Reinforced Composites are composed of fibres embedded in matrix material. The matrix serves to bind the fibres together, transfer loads to the fibres and protect them against environmental attack due to handling, while the reinforcement transfer strength to the matrix constituent, thereby influencing and enhancing their properties as desired. The dimensions of the fibre determine its capability of contributing its properties to the composite.

The length, shape, orientation, and composition of the fibers and the mechanical properties of the matrix determines the performance of a fiber composite. Fiber composites can be characterized based on fiber length which is much greater compared to its cross-sectional dimensions into Short-fibre or long-fibre reinforced composites.

- i. Discontinuous fibre (short fiber): these are short fibres, particles or whiskers. It defines a reinforced composite whose properties vary with fibre length. In discontinuous fibre reinforced composites, the load transfer function of the matrix is more critical than in continuous fibre composites.

It is an important class of engineering materials because of their desirable properties including low density, high hardness, high compressive strength, wear resistance, etc.



a) Aligned

b) Random

Figure 2.2 Discontinuous fibre reinforced composite (Callister, 2007)

- ii. Continuous fibre (or filaments): these are filamentary materials whose lengths are greater than 100 μm . They are found in reinforced composites with which the length of the fibre is such that any further increase in length does not further increase the elastic modulus of the composite. It exhibit better orientation and due to its long dimension does not

encourage the growth of cracks initiation normal to the reinforcement which might lead to failure, making fibres effective in enhancing the fracture toughness resistance of the matrix.

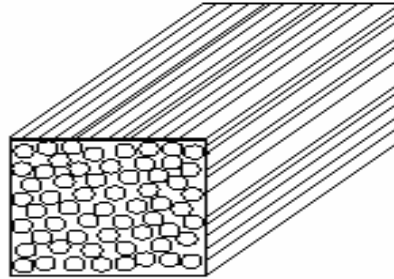


Figure 2.3 Continuous fibre reinforced composite (www.nptel.ac.in)

(b) Laminar Reinforced Composites

Composites in this class are composed of several layers of two or more metal materials occurring alternately or in a determined order more than once, and in as many numbers as required for a specific purpose held together by matrix. They are reinforced by repeating lamellar reinforcement of high modulus and strength.

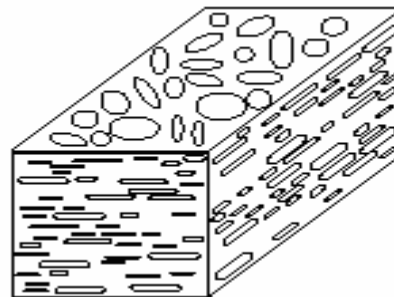


Figure 2.4 Laminar reinforced composite (www.nptel.ac.in)

(c) Particulate Reinforced Composites

Composites in this class, from microstructures, show particles of one phase strewn in the other or embedded in the matrix. The particles may be flakes or in powder form. Particle fillers due to the good wear resistance that hard fillers can give are widely used to enhance the properties of matrix materials (Clyne, 2000). They have high volume fraction of dispersoid, smaller sizes of particles and inter-particle spacing.

Their Strength depends on diameter of the particle, interparticle spacing, and volume fraction of reinforcement matrix.

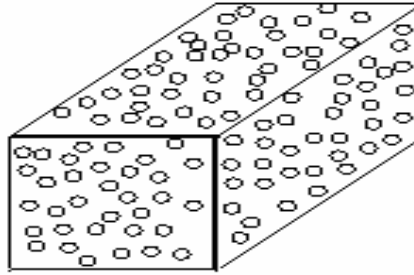


Figure 2.5 Particulate reinforced composite (www.nptel.ac.in)

2.2.2 Based on Matrix

The major composite classes based on the monolithic materials include:

- (a) Metal Matrix Composites (MMCs):- this is the type of matrix composites in which metal is the monolithic material, that is, metal is the major constituent reinforced with any other material, usually ceramics. MMCs are composites with at least two constituent parts, one being a metal and the other material might be an entirely different material. Metals found applications in engineering because they can be plastically deformed and strengthened by inhibiting the motion of dislocations by various methods. The monolithic material can include zinc, aluminium, iron, cobalt, magnesium, copper, reinforced with another material such as organic compound or ceramic. It is called hybrid composites when three materials are present, (www.nptel.ac.in).
- (b) Organic Matrix Composites (OMCs):- this can be classified into the polymer matrix and carbon matrix composites.
 - i. Polymer Matrix Composites (PMCs): polymer is the matrix, reinforced with another material. Polymers are embraced especially in aeronautical applications where avionic systems are to be secured from external electrical activity (for example, lightning strike) because of their electrical conductivity when reinforced with carbon fibres. Polymer Matrix composites are composed of a matrix from thermoset for example, unsaturated polyester or thermoplastic for example, Polystyrene. Embedded glass, carbon, steel or Kevlar fibres as dispersed phase, (www.nptel.ac.in).
 - ii. Carbon matrix composites or carbon-carbon composites: carbon is the matrix reinforced with another material. Its development is determined by the opportunity to develop its mechanical properties and reduce its brittleness. Its properties includes lubricating and electrical properties, high thermal conductivity and relatively low susceptibility to thermal shock, nuclear characteristics, high strength and resistance to chemical and

oxidative environment, high tensile moduli and tensile strengths that are retained to temperatures in excess of 2000 °C. Its setback is the tendency to high temperature oxidation. Carbon matrix composites are applied in rocket motors, as friction materials in aircrafts and high performance automobiles, advanced gas turbines, hot pressing moulds (Callister, 2007). Carbon matrix can be as conventional hot-pressed carbons and graphite, pyrolytic graphite, densified impermeable graphite in form (Harris, 1999).

- (c) Ceramic Matrix Composites (CMCs): this class of composites involves ceramic constituting the major part, reinforced with another material. Ceramics are resilient to oxidation and deterioration, improved high temperature creep behaviour, high temperature and severe stress applications except for their brittle nature. They may be fabricated by liquid phase sintering techniques or by hot isostatic pressing (Callister, 2007).

2.3 METAL MATRIX COMPOSITES (MMCs)

Metal Matrix Composites are recently drawing attentions of researchers because of the ability to alter their physical properties such as density, thermal expansion, high strength, fracture toughness, stiffness, thermal diffusivity and mechanical properties like tensile and compressive behaviour, creep, and tribological behaviour by varying the filler phase. Most metals and their alloys can be used as matrices and requires the reinforcements to be non-reactive and stable over a range of temperature. Example of the light metals that serves as matrices includes aluminium, titanium and magnesium.

Some of the advantages of metal matrix composites compared to monolithic metals includes higher strength-to-density ratios, better fatigue resistance, lower coefficients of thermal expansion, better wear resistance, high modulus, high toughness and impact properties, high surface durability and low sensitivity to surface flaws. Some of the disadvantages of metal matrix composites compared to monolithic materials also, includes higher cost of fabrication and of the reinforcement materials, low recyclability, and harder to fabricate than the resin-matrix composites.

Some of the areas where Metal Matrix Composites have found applications includes aerospace (commercial airliners, Space shuttle), military (Covers for missile guidance system), civil, electronics (electronic substrates), energy, automobile (bicycles, diesel engine pistons), sports (golf clubs), medicals, marine, cutting tools (Chawla, 1998).

Types of Metallic Matrix

Many different metals and alloys ranging from light alloys like aluminium, magnesium, titanium, to others like zinc, copper their alloys to mention but a few have been employed as matrix material for the production of Metal Matrix Composites (Bodunrin *et al.*, 2015).

i. Aluminium and its alloys

Aluminium from literature has been employed in the production of Metal Matrix Composites. It has approximate density of 0.1 lb/in^3 (2698 kg/m^3) and a low melting temperature of 1220°F (660°C) compared to other metals. Aluminium composites can be produced by casting and powder metallurgy among others. Aluminium composites has applications mostly in the Aerospace Industry because of their strength, toughness, resistance to corrosion and relatively low density.

ii. Titanium and its alloys

Titanium is a light metal with approximate density of 0.18 lb/in^3 (4317 kg/m^3) and it is about 60 % higher than aluminium alloys in density. Its merits include the ability to retain strength to high temperatures, good structural properties, good oxidation and corrosion resistance at temperatures of about 315°C (600°F). It is also applied in the Aerospace Industry because of its low density.

iii. Magnesium and its alloys

Magnesium is also light metal (lightest of the three) with density of 1.74 g/cm^3 and about 35 % lighter than aluminium. Magnesium is employed in engineering due to its damping capacity, castability and excellent machinability. However, its low strength and ductility has been major setback to its use. Hard ceramic reinforcement such as SiC, Al_2O_3 are often added to Mg to surmount these limitations, thereby resulting in extreme brittleness. It is applied in aircraft gearbox housings, chainsaw housings and electronic equipment.

2.3.1 Properties of MMCs

Metal–matrix composites possesses high strength, stiffness, thermal conductivity, abrasion resistance, creep resistance, or dimensional stability, fracture toughness, they can withstand elevated temperature in corrosive environment than polymer composites. Most metals and alloys could be used as matrices and they require reinforcement materials which needs to be stable over a range of temperature and non-reactive too.

2.3.2 Methods of producing MMCs

The various methods that can be employed in the production of Metal Matrix Composites include:

(a) Solid state methods

- i. Powder blending and consolidation (powder metallurgy): this involves the mixing of powdered metal and discontinuous reinforcement, thereby bonding through compaction, degassing, or thermo mechanical treatment. It can be employed in the production of fine grained composites with near-uniform reinforcement dispersal with highest possible reinforcement content (Viswanath *et al.*, 2015).

Advantages of Powder Metallurgy techniques include nominal wettability-related problems, incorporation of higher volume fraction of reinforcements, possibility of using reinforcement-metal combinations not viable by liquid-state routes.

Disadvantages or setbacks include cost-ineffectiveness, oxidization of powders which demands inert/protective gas conditions, and high porosity content which is inevitable in powder metallurgy techniques, leading to degradation in properties.

- ii. Foil diffusion bonding: involves sandwiching layers of metal foil with long fibres and then pressing through to form a matrix.
- iii. Mechanical Alloying: this is a traditional Powder Metallurgy process. During the process, a small quantity of the base powder is loaded into a sealed container, together with the grinding media, then blended (to mix the powders without inducing material transfer between the mixed components) through agitation at high speed for a predetermined amount of time and with the use of a process control agent usually lubricant (Methanol, stearic acid, and paraffin compounds). The matrix and reinforcement using this method can be alloyed together by inducing cold welding, fracturing, and re-welding of the powder particles (Al-Aqeeli and Suryanarayana, 2013; Suryanarayana, 2001,2011). The kinetic energy of the grinding balls depends on their mass and velocity. Main process parameters, influencing the quality of the composite includes ball-to-powder ratio, time and rotational speed of milling. Powders are compacted, degassed, and consolidated after milling.

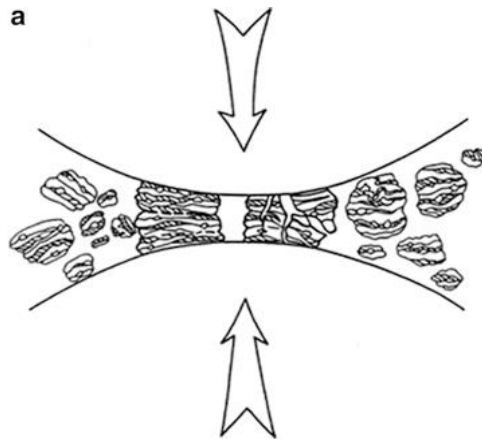


Figure 2.6 Mechanical alloying processing technique (Suryanarayana, 2001)

iv. Reaction Milling

Reaction milling defines a mechanical alloying process which is accompanied by a solid state reaction aimed to produce fine dispersion of oxides, nitrides, and carbides in the light alloy matrix. For the reaction to occur, the process control agent can be absent or introduced by a suitable milling atmosphere, i.e., oxygen, argon, nitrogen, or simply air (Suryanarayana, 2001).

v. Cryomilling

Cryomilling involves carrying out the milling phase at cryogenic temperatures or within a cryogenic medium, as liquid nitrogen (Lavernia and Witkin, 2006). A Process control agent like stearic acid can be used to avoid severe sticking. Severe recovery and recrystallization of fine microstructures occur as a result of the increase in temperature due to the attrition during traditional milling process.

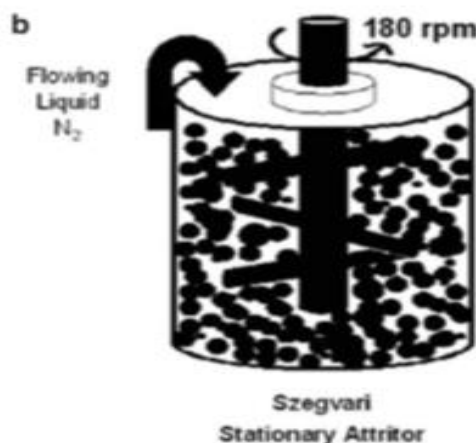


Figure 2.7 Schematic of cryomilling process (Lavernia and Witkin, 2006)

vi. Spark Plasma Sintering

Spark plasma sintering also known as field assisted sintering is an effective nonconventional sintering method for attaining fully dense materials with refined grain size (Saheb *et al.*, 2012). The densification is enabled by passing a pulsed DC current through a graphite die and composite powder compact. Also the heat generation is internal in disparity to the conventional hot pressing where heat is provided by external heating elements. The main drawback of conventional sintering is the occurrence of high porosity in a product, likewise, when the green compacts are hot pressed, hot extruded, or hot isostatically pressed, it often results in matrix grain growth that weakens mechanical properties. This process is effective for producing fully dense light metal nanocomposites.

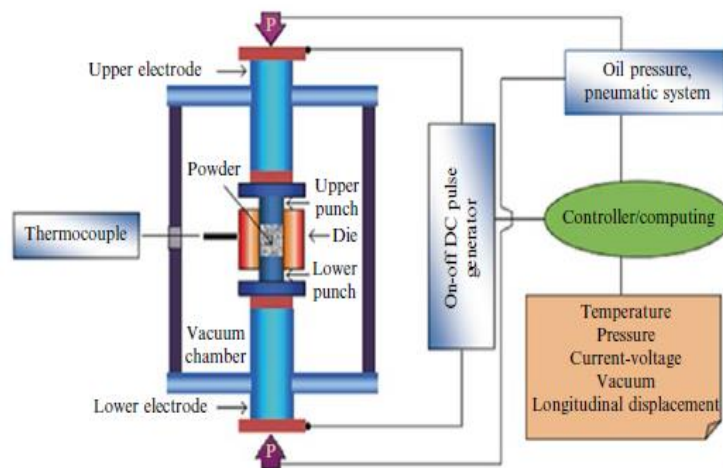


Figure 2.8 Spark plasma sintering (Saheb *et al.*, 2012)

vii. Microwave Sintering

Microwave heating can be defined as a volumetric heating process which involves the conversion of electromagnetic energy into thermal energy (Eugene and Gupta, 2007). It is in contrast to conventional sintering process (where heat is generated from the outer surface of the material) as heat is generated from within the materials and then radiated outwards due to the penetrative power of microwaves. Higher temperatures exist at the core of materials in this process whereas their surfaces experience lower temperatures (thermal gradient).

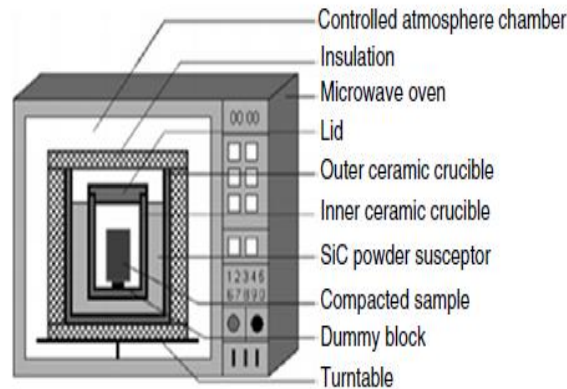


Figure 2.9 Bi-directional hybrid microwave-assisted rapid sintering (Gupta and Tun, 2009).

(b) Liquid state methods.

- i. Electroplating and electroforming: involves co-depositing a solution containing metal ions loaded with reinforcing particles.
- ii. Stir Casting- involves the stirring of discontinuous reinforcement into molten metal after which solidification is allowed.

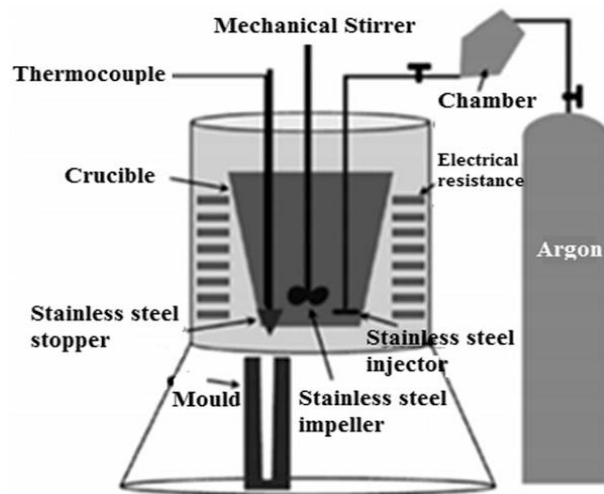


Figure 2.10 Stir casting method (Ezatpour *et al.*, 2014)

- iii. Pressure infiltration: involves the infiltration of molten metal into the reinforcement using of a kind of pressure such as gas pressure. This method can be used to synthesize composites with higher volume fraction of reinforcement (Viswanath *et al.*, 2015).
- iv. Centrifugal casting: Centrifugal casting is a process in which centrifugal force is induced immediately during casting to achieve optimal reinforcement placements. Centrifugal casting is applied in brake rotors, wherein the rotor face is expected to be of high wear

resistance when compared to the hub, as difficulties encountered in machining due to high hardness of the composite produced by normal casting methods are eliminated by centrifugal casting process (Chawla and Chawla, 2006).

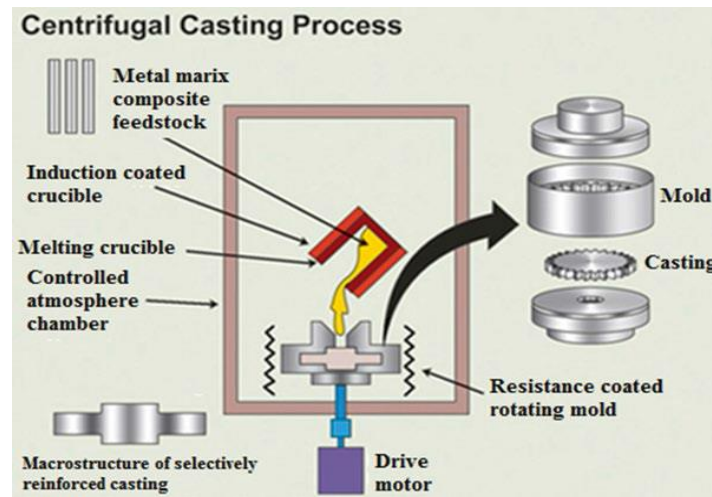


Figure 2.11 Schematic of centrifugal casting process (<http://www.adityainc.com/casting/centrifugal-casting.html>, 2014)

- v. Squeeze Casting- involves the injection of molten metal into a form with fibers pre-placed inside it.

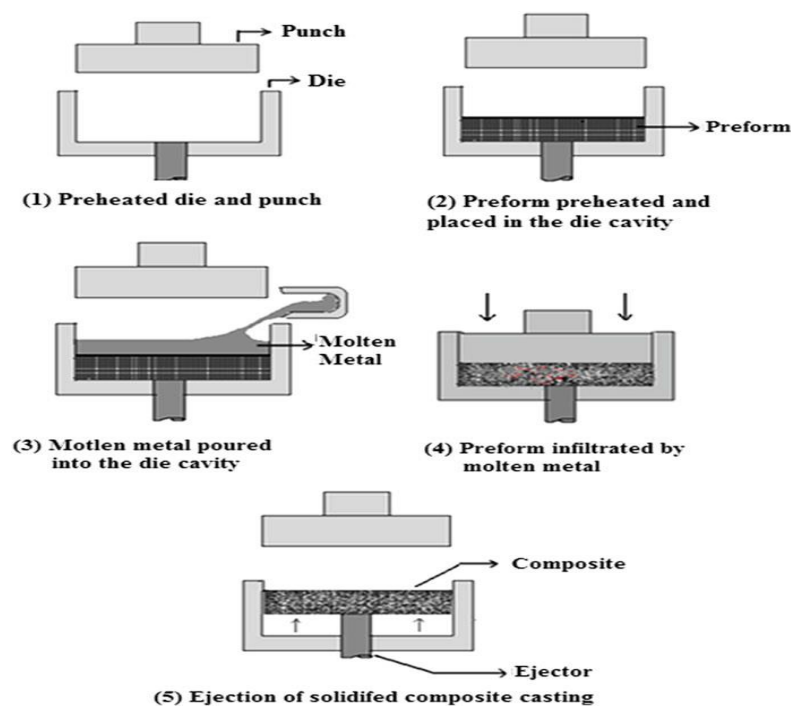


Figure 2.12 Schematic of squeeze casting/infiltration process (Ghomashchi and Vikhrov, 2000)

- vi. Spray deposition: involves the spraying of molten metal onto a continuous fiber substrate.
- vii. Disintegrated melt deposition technique: This technique as a liquid-state processing method has the combined merits of both gravity die casting and spray forming processes (Gupta and Sharon, 2011). DMD employs higher superheat temperatures and lower impinging gas jet velocity unlike the spray process. It is usually employed for particle reinforcements, and is quite successful for reinforcing nanoparticles. The process involves stirring of reinforcement particles with a predetermined stirring velocity and time using an impeller when the metal/alloy is in the molten state. The disintegration process of the composite melt ensures higher solidification rate and fine-grained structure.

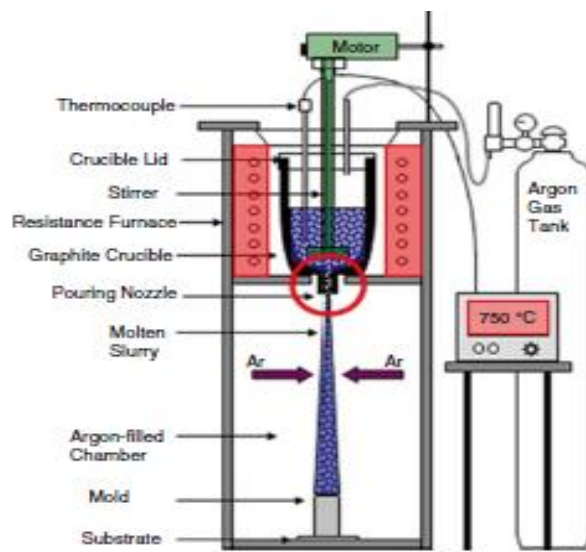


Figure 2.13 Disintegrated melt deposition technique (Gupta and Sharon, 2011).

- viii. Reactive Processing- involves a chemical reaction occurring with one of the reactants forming the matrix and the other the reinforcement.
- ix. Ultrasonic-assisted casting method: ultrasonic-assisted casting method is effective in extenuating particle cluster formation in composites that occurs (nanocomposites in particular) due to the low wettability and high tendency of agglomeration of nanoparticles (Donthamsetty *et al.*, 2009; Mula *et al.*, 2009). Agglomeration is usually encountered in conventional stirring methods such as mechanical stirring/vortex methods. In contrast, the ultrasonic-assisted method employs subjecting the melts with ultrasonic waves (frequency range: 18–20 kHz) during or after adding a reinforcing phase. This is followed by casting. A schematic of the setup is shown in Fig. 2.14 (Yang *et al.*, 2004).

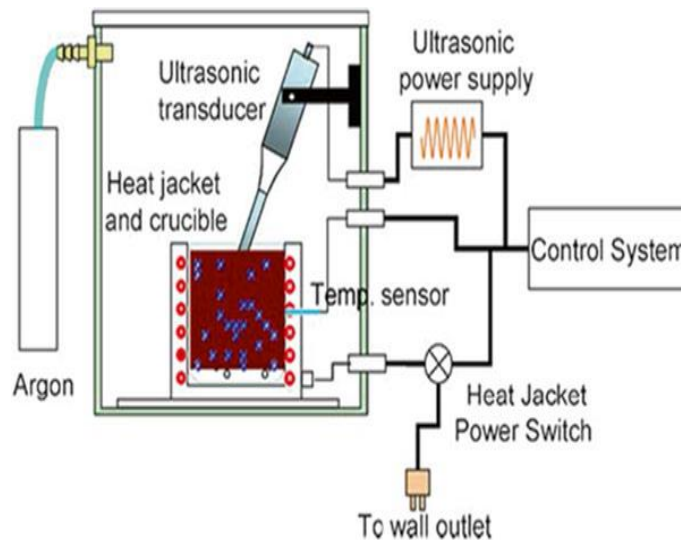


Figure 2.14 Schematic of Ultrasonic-assisted casting (Yang *et al.*, 2004)

(c) Semi-solid state method

This process involves the incorporation of particles in matrix metal is conducted when a matrix is in semisolid state (slurry). The process enables uniform dispersion of reinforcements, removes settling of denser reinforcement due to slurry state, eradicates porosity (that usually occurs due to voids generated at the molten metal/particle interface), (kandemir *et al.*, 2012; Nie *et al.*, 2011).

The various types of semi-solid state methods include:

- i. Semi-solid powder processing: involves the heating of a powder mixture to semi-solid state and then the application of pressure to form the composites.
- ii. Thixo-processing: it involves reheating and partial melting of a proper solid feedstock. The base material is generated by allowing a liquid melt to partially solidify under controlled conditions of low superheat and rapid cooling, usually with significant convection in the liquid. This is done to induce the formation of crystals in the slurry. The feedstock may be produced in a variety of ways such as with mechanical stirring during solidification as in rheocasting, continuous casting combined with magneto-hydrodynamic stirring for grain refining and ultrasonic treatment for grain refinement, (Abbasipour *et al.*, 2010; Gonasagren and Heinrich, 2012).
- iii. Rheo-processing: this processes does not require a special feedstock. The semisolid slurry is generated starting from the liquid state by cooling the molten metal during the casting process itself unlike the thixo-process. Rheo-processes are all easier to implement in a foundry as they involve standard equipment for melting, transport, treatment, degassing, and handling (Yasunori *et al.*, 1996).

- iv. **Compocasting:** Compocasting is a rheocasting process which involves the injection of reinforcement particles into semisolid state alloys. It is generally assumed to be a processing route which allows quite uniform distribution of reinforcing particles to be attained, and also enhance particle wettability (Kamali Ardakani *et al.*, 2014). Also the lower porosity which is usually observed in experimental studies is attributed to the better wettability between the matrix and the reinforcement particles as well as the lower volume shrinkage of the matrix.

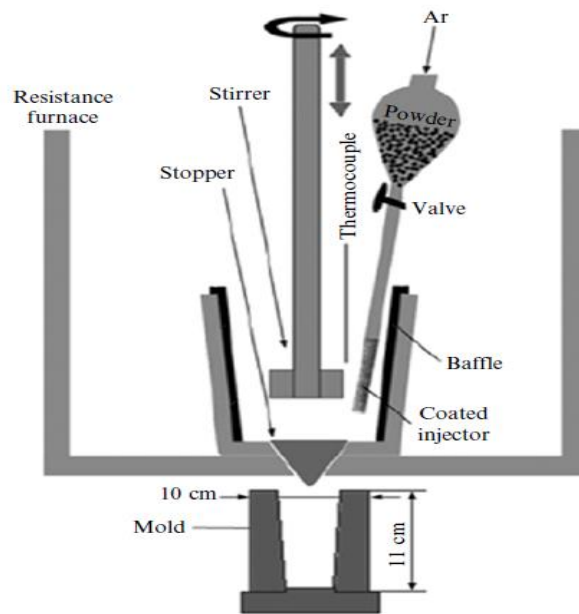


Figure 2.15 Compocasting experimental setup (Abbasipour *et al.*, 2010)

(d) Vapour deposition

- i. **Physical Vapour deposition-** involves passing of fiber through a thick cloud of vapourised metal, thereby coating it.

(e) Insitu-fabrication technique

- i. **Controlled unidirectional solidification** of a eutectic alloy can result in a two phase microstructure with one of the phases present in lamellar or fiber form, distributed in the matrix. E.g. Cu/Cr and Al/Al₂Cu.

2.3.3 Factors affecting successful production of MMCs

Himanshu *et al.* (2014), Ananda and Sudipt (2008) observed that there are some factors that needs to be considered when preparing MMCs by the stir casting method which includes:

- i. The difficulty of achieving a uniform distribution of the reinforcement material (uniform): Settling of the reinforcement particles during melt holding or during casting is one of the problems encountered in metal matrix composite processing. This arises as a result of density differences between the reinforcement particles and the matrix alloy melt. Factors that affects the proper dispersion of the particulate reinforcements in a matrix are pouring rate, pouring temperature and gating systems. Factors such as the geometry of the mechanical stirrer, position of stirrer in the melt, melt temperature, and the properties of the particles added also determines the distribution of particles in molten matrix. Also the distribution of particles in the final solid depends on strength of mixing, wetting condition of the particles with the melt, rate of solidification and relative density, (Girod *et al.*, 1987; Harnby *et al.*, 1985). The reinforcement distribution is influenced during several stages such as the distribution in the liquid as a result of mixing, the distribution in the liquid after mixing, but before solidification, and the redistribution as a result of solidification.

- ii. Wettability between the two main substances (should be optimised): wettability can be defined as the ability of a liquid to spread on a solid surface. It describes the extent of intimate contact between the liquid and the solid. It is a significant problem when producing cast metal matrix composites. The attainment of complete wetting becomes more difficult to achieve as the particle size decreases, due to the increase in the surface energy required for the metal surface to deform to a small radius as the particles begin to penetrate through it.

The basic means used to improve wetting are by increasing the surface energies of the solid, decreasing the surface tension of the liquid matrix alloy, and decreasing the solid-liquid interfacial energy at the particles-matrix interface.

Major approaches that has been taken to promote the wetting of the reinforcement particles with a molten matrix alloy includes the coating of the particles (using wettable metal), addition of alloying elements (usually reactive elements such as Mg, Ca, Ti, or Zr) to the molten matrix alloy, the treatment of the particles (Heat treatment), and ultrasonic irradiation of the melt (etching techniques, and heating in a suitable atmosphere can be used to clean the particle surface).

A clean surface provides a better opportunity for melt-particles interaction, and thus, enhances wetting.

- iii. Porosity in the cast metal matrix composites (should be minimal): Porosity of a composite occurs as a result of air bubbles entering the slurry either independently or as an air

envelope to the reinforcement particles. The size, volume fraction and distribution of porosity plays important role in controlling the mechanical properties of a composite material, thereby making the material detrimental to corrosion resistance. The three causes of porosity are gas entrapment during mixing, hydrogen evolution, and shrinkage during solidification.

Factors that influence the development of porosity are the process parameters of holding times, stirring speed, and the size and position of the impeller.

Some of the developed strategies to minimize porosity are compocasting in vacuum, extensive inert gas bubbling through the melt, casting under pressure, or compressing, and extruding, or rolling the materials after casting to close the pores.

- iv. Chemical reactions between the reinforcement material and the matrix alloy (should be avoided).

In conclusion, controlling the processing conditions as well as the relative amount of the reinforcement material, composite with a broad range of mechanical properties can be obtained.

2.4 STIR CASTING METHOD OF PRODUCING MMCS

Stir casting is a generally accepted and most economical method of producing MMCs. The process usually involves the production of melt of the selected matrix material, followed by the introduction of the reinforcement material into the melt. Also the distribution of the particulate reinforcement into the matrix-melt by mechanical stirring to obtain a suitable dispersion, followed by the solidification of the melt containing suspended dispersoids under some selected conditions to obtain the desired distribution of the dispersed phase in the matrix (Hashim *et al.*, 1999).

The major advantages of stir casting process is its applicability to mass production, low cost compared to other fabrication methods, its simplicity, and flexibility (Himanshu *et al.*, 2014). This method is attractive because, from principle it allows the use of conventional metal processing route and hence minimizes the final cost of the product and allows very large sized components to be fabricated.

One of the problems associated with the stir casting process is the segregation of reinforcing particles due to settling of particles during solidification (Himanshu *et al.*, 2014).

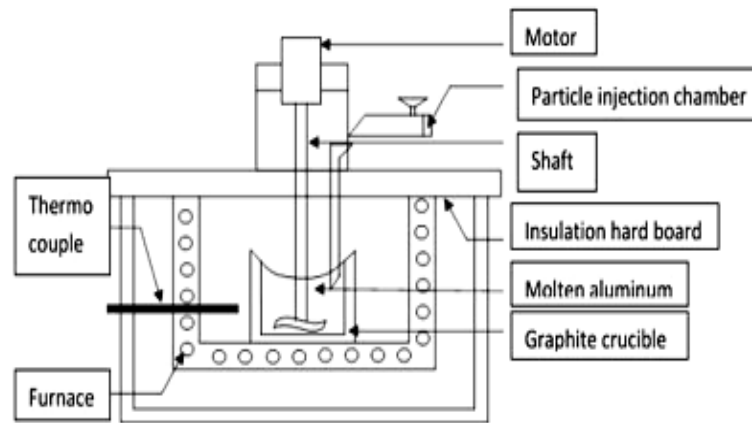


Figure 2.16 Stirring mechanism for the Fabrication of composites (Lloyd *et al.*, 1989).

Factors to consider in the production of cast MMCs includes the mechanical stirrer used (usually during melt preparation or holding) during stirring, the melt temperature, and the type, amount and nature of the particles.

The methods of introducing particles into the matrix melt to ensure proper dispersion of reinforcement in the melt is an important aspect in casting process. These methods includes

- i. Injection of the particles entrained in an inert carrier gas into the melt with the help of an injection gun, wherein the particles are mixed into the melt as the bubbles rise through the melt;
- ii. Addition of particles into the molten stream as the mould is filled;
- iii. Pushing particles into the melt through the use of reciprocating rods;
- iv. Spray casting of droplets of atomised molten metal along with particles onto a substrate;
- v. Dispersion of fine particles in the melt by centrifugal action;
- vi. Pre-infiltrating a packed bed of particles to form pellets of a master alloy, and redispersing and diluting into a melt, followed by slow hand or mechanical stirring;
- vii. Injection of particles into the melt while the melt is irradiated continuously with high intensity ultrasound; and
- viii. Zero gravity processing which involves utilising a synergism of ultra-high vacuum and elevated temperature for a prolonged period of time.

Stirring during stir casting method is important for the synthesis of composites as it helps in transferring particles into the liquid metal, and maintaining the particles in a state of suspension.

2.4.1 Double Stir Casting Method

This method involves heating of the matrix material to temperature above its liquidus temperature, cooling the melt to a temperature between the liquidus and solidus points to a

semi-solid state, then addition and mixture of preheated reinforcement particles followed by heating the slurry again to a fully liquid state and mixed thoroughly. It also known as two-step mixing.

In double stir casting the resulting microstructure has been found to be more uniform as compared with conventional stirring. The advantage of double-stir casting method is its ability to break the gas layer around the particle surface (because of the abrasive action due to the high melt viscosity) which otherwise impedes wetting between the particles and molten metal (Himanshu *et al.*, 2014).

2.5 STRENGTHENING MECHANISM IN MMCs

Strengthening mechanism is considered to control the performance of materials and it takes place by the reinforcement carrying much of the applied load. It can be divided into direct and indirect strengthening as observed in MMCs.

Direct strengthening in particulate reinforced metals can be defined as an extension of the conventional composite strengthening mechanisms used to define the behavior of continuous fiber reinforced composites.

Indirect strengthening occur from the punching of thermally induced dislocation formed at the reinforcement/matrix interface. This is due to the high thermal mismatch between the high expansion of metallic matrix and the low expansion ceramic in a metal matrix composite where a high stiffness ceramic reinforcement is used. Indirect strengthening increases with increase in the percentage weight of reinforcement and decrease in particle size (Chawla and Shen, 2001). The strengthening mechanisms (Gupta and Jayalakshmi, 2015) include:

- i. Hall–Petch effect (grain refinement): the introduction of reinforcement (fibers/particles) in a molten matrix can act as heterogeneous nucleation sites during solidification, thus giving rise to refined and equiaxed grains. When secondary processes such as hot extrusion are undertaken, grain growth during recrystallization is hindered due to the grain boundary pinning effect by the reinforcements.
- ii. Orowan strengthening: This strengthening arises due to obstacle posed by closely-spaced hard particles to the dislocation motion. Highly dispersed reinforcements significantly increase the yield strength.
- iii. Enhanced dislocation density: The matrix and reinforcement materials usually have different CTE, due to which thermal stresses are generated during processing (for example, during cooling in casting process). Such stress levels are large enough to

increase dislocation density, especially at the matrix/reinforcement interface resulting in higher yield strengths.

- iv. Load bearing effect: Matrix transfers the load to the reinforcement under external loads. Reinforcements have strong interfacial bonding with matrix and so good load bearing capacity is exhibited.
- v. Solid-state hardening methods used in alloys: this involve producing a material in a metastable state which may subsequently revert to a more stable but unstrengthened condition if sufficient thermal energy is provided. The strengthening of metallic alloys is achieved through grain size refinement and dispersion of particles.
- vi. Precipitation hardening to strengthen alloys: alloys such as the strong aluminium alloys and alloys depending on phase transformations of the martensitic type like steels and heavily coldworked metals depend simply on the presence of a high dislocation density, and will all soften at elevated temperatures.
- vii. Age-hardened matrix materials: involves the thermally-induced dislocations which are formed upon quenching from the solution treatment to serve as heterogeneous nucleation sites for precipitate formation during the aging treatment. There is preferential distribution of precipitates in the particle/matrix interface region with the higher density of dislocations causing an 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 due to a larger amount of interfacial area exists for dislocation punching to take place. The difference in strengthening between unreinforced and composite could be attributed primarily to load transfer to the reinforcement

2.5.1 Dispersion Strengthening Mechanism of Composites

Dispersion strengthened composite is a class of particle reinforced composites based on strengthening mechanism. It involves the distribution of particles which are relatively smaller and usually of size ranges of 0.01-0.1 μm . The strengthening mechanism involves the matrix bearing the major portion of the applied load, while the dispersoids (metallic, non-metallic, or oxides materials) impede the motion of dislocations. The dispersion in composites extremely blocks the dislocation motions thereby increasing the yield strength and the modulus of elasticity and reducing the ductility. Smaller size particles had better properties than bigger one.

Oxide dispersion strengthened (ODS) steel is the most auspicious type of dispersed phase due to its excellent creep and irradiation resistance on the basis of uniformly distributed nano-oxide particles with a high density which are extremely stable at high temperatures under a neutron irradiation environment for a prolonged operating period. ODS can be used for military, aerospace, and plant industries including fusion reactor systems (Kim *et al.*, 2015). Examples includes thoria dispersed Ni-alloys with high temperature strength, and dispersion of extremely small flakes of alumina in aluminium matrix (Callister, 2004).

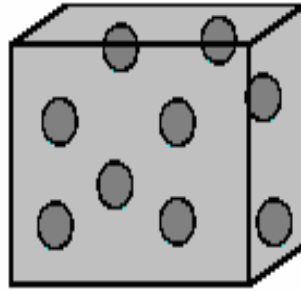


Figure 2.17 Particulate reinforced composite (Dispersion strengthened), (Callister, 2004)

2.5.2 Strengthening Mechanism of Particulate Composites

Particulate composites is the second class of particle reinforced composites based on strengthening mechanism. It is the one that contains large amount of comparatively coarse particles and can be used with the three classes of materials. It is one in which particle-matrix interactions cannot be treated on an atomic or molecular level i.e. the particle reinforced the matrix phase. The degree of reinforcement or improvement of mechanical properties depends on strong bonding at the matrix-particle interphase. Particulate composites are designed to produce unusual combinations of properties such as abrasion resistance, toughness, etcetera, rather than to improve strength. The degree of strength depends greatly on the amount of particulate present (volume fraction), the shape and size of the particulates, the level of distribution (Prabu *et al.*, 2006). Examples are the dispersion of SiC in aluminium for metals, the reinforcement of vulcanised rubber with carbon black for polymers, and the addition of cobalt or nickel in tungsten carbide or titanium carbide for ceramics and concrete. The particulate composites found application in making pistons and brakes for Automobiles (Callister, 2004).

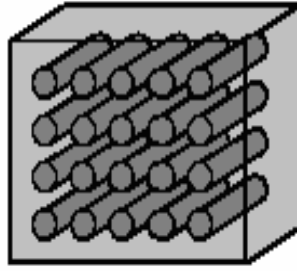


Figure 2.18 Particulate reinforced composite (Particulate composite), (Callister, 2004)

2.5.2 Particle size and shape

Particle size and shape contributes to the degree of strengthening in particulate reinforced composites. Considering some previous articles on particle size and shape, Sankaranarayanan *et al.* (2014) investigated Hybridizing micro-Ti with nano-B₄C particulates to improve the microstructural and mechanical characteristics of Mg-Ti composite. They observed that the nano-B₄C hybridized Mg-Ti composite showed improved strength and ductility under tensile and compressive loads, improved localized recrystallization and refined microstructure. Aravindan *et al.* (2015) investigated Evaluation of physical and mechanical properties of AZ91D/SiC composites by two step stir casting process. They observed the effect of changes in particle size and volume fraction of the SiC particles on physical and mechanical properties of the composites as the alloy reinforced with the finer SiC exhibits superior properties while the coarse SiC exhibits better properties when compared with unreinforced magnesium alloy (AZ91). Also Mimoto *et al.* (2011) investigated the mechanical and strengthening mechanism of pre Titanium powder composite reinforced with carbon nanoparticles. They found out the improvement in the mechanical properties of the composite was due to grain refinement.

2.6 ZINC-ALUMINIUM ALLOYS (ZA ALLOYS) OVERVIEW

Zinc-based alloys with high amount of aluminum (designated as ZA alloys) comprise a family of die-casting alloys that have proven themselves in a wide variety of demanding applications. The members of the ZA casting alloys are ZA-8, ZA-12 and ZA-27 alloy. These alloys combine high strength and hardness, good machinability with good bearing properties and wear resistance. Zn-Al based composite is a Metal-matrix composites (MMCs) which possess both high damping and high stiffness are of interest in achieving better performance for structural damping applications. They are used in the production of Metallic foams

(porous metals with high porosity ranging from 40 to 98 vol. %). The metallic foams are used as shock and impact absorbers, dust and fluid filters, engine exhaust mufflers, porous electrodes, high-temperature gaskets, heaters and heat exchangers, flame arresters, catalyst supporters. These alloys combine high strength and hardness, good machinability with good bearing properties and wear resistance. One major limitation to the practical use of Zn-alloys is their poor mechanical/structural resistance at elevated temperatures (above 100 °C), a consequence of the low melting point and poor dimensional stability of Zn (Iglesias *et al.*, 2013). They are produced using the stir casting method.



Figure 2.19 Applications of ZA-27 alloy (Bobic *et al.*, 2009)

2.6.1 Applications of ZA alloys

The good combination of physical, mechanical and technological properties, high strength, lowest density, excellent bearing, excellent corrosion resistance, high design stress capability at elevated temperatures excellent castability, good machinability (Jaglinski and Lakes, 2011), good tribological properties, low manufacturing cost and ease of fabrication are among its notable characteristics (Alaneme and Ajayi, 2015). These characteristics has helped them in showing satisfactory service performance, that is, to be useful as industrial casting alloys and are for many applications (Alaneme and Ajayi, 2015; Hekimoglu and Savaskan, 2014).

Some examples of ZA-27 alloy application are presented in Fig. 2.19



Medical

Sports

Engineering Construction

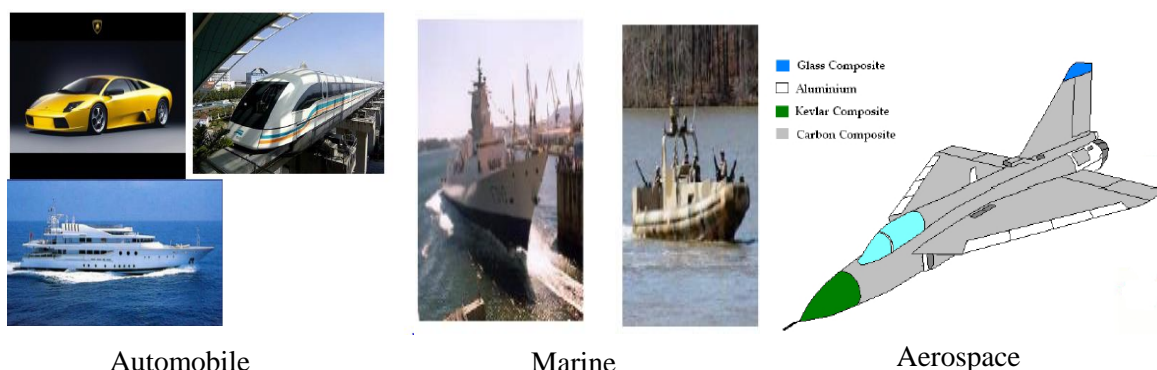


Figure 2.20 Application of ZA-27 alloy (Chawla, 1998).

Other applications of Zn-Al alloys when used for the design of components and industrial applications includes bearings and dies, Punches and seals (Bobic *et al.*, 2009), heat transfer conductors, high conductivity electrical contractors. (Prakash and Pruthviraj, 2011), industrial fittings and hardware, pressure tight housings, thrust washers and wear plates, electrical switchgear and hardware, hose couplings and connectors, fire fighting hardware, pneumatic and hydraulic cylinder components, industrial machine hardware, electrical conduit fittings, door hardware and lock components, pulleys and sheaves, non-sparking mine hardware, decorative hardware, electronic instrument chassis, hardware and covers (Prasad, 2007).

2.6.2 Mechanical, Corrosion and Wear behaviour of ZA alloys

(a) Mechanical properties

The mechanical properties of a composite depend on many factors such type of reinforcement, measure of reinforcement, shape, size (Himanshu *et al.*, 2014).

Considering previous articles on mechanical properties:

Alaneme and Ajayi (2015) investigated microstructure and mechanical behavior of stir-cast Zn-27Al based composites reinforced with rice husk ash, silicon carbide, and graphite. Hardness and tensile strength were observed to diminish while fracture toughness increased with rise in the weight percent of rice husk ash and decrease in SiC for both the 7 and 10 wt.% reinforced composite. Also Alaneme *et al.* (2013) studied mechanical and corrosion behaviour of SiC, bamboo ash reinforced Al-Mg-Si alloy hybrid composite and reported the ultimate tensile strength, hardness and yield strength values of the composite decreased, while fracture toughness increased with increase in weight percent of bamboo leaf ash. Alaneme *et al.* (2014) investigated the mechanical and corrosion behavior of Zn-27Al based composites reinforced with groundnut shell ash and silicon carbide. They observed that hardness, UTS, and elongation (though not consistent as others) of the composite decreased, while, the fracture toughness was enhanced with increase in groundnut shell ash content.

Also Alaneme and Sanusi (2015) investigated microstructural characteristics, mechanical and wear behaviour of Aluminium matrix hybrid composites reinforced with alumina, rice husk ash and graphite and reported that hardness decreased with increase in the weight ratios rice husk ash and graphite, also that the composites with 0.5wt.% graphite and 50 % rice husk ash has higher tensile strength and toughness than in cases without graphite. Himanshu *et al.* (2014) studied the mechanical and tribological behaviors of stir cast aluminum matrix composites and concluded that tensile strength increased while the hardness decreased on addition of graphite to the aluminum. Fatile *et al.* (2015) also studied fabrication characteristics and mechanical behavior of fly ash-alumina reinforced Zn-27Al hybrid composite using stir-casting technique and observed that the hardness, ultimate tensile strength, and percent elongation of the hybrid composites decreased with increase in fly ash content, also that the fracture toughness of compositions containing fly ash was greater to that without fly ash. Sajjadi *et al.* (2011) investigated mechanical properties of the stir cast Al (A356)/ Al₂O₃p composite. The hardness and compressive strength of the composite were reported to be amplified with increasing weight percent of Alumina with reducing particle size. Su *et al.* (2012) studied tensile strength of nano particle alumina reinforced Al (2024) matrix composite using 3 step casting method. The yield strength and tensile strength of the composite was observed to be higher to that of pure matrix alloy.

(b) Corrosion Resistance

Corrosion can be defined as the deterioration of a material due to leaching, dissolution, and chemical or electrochemical reaction with the environment (Mahmoud *et al.*, 2012).

Corrosion can occur uniformly (for homogeneous single phase materials) or irregularly (due to the segregation of reactive phases and impurities at the grain boundaries). The reinforcements distributed within the matrix material of a Metal Matrix Composites are in form of fibres, particles and whiskers (short fibres) usually combined with two or more materials that have different corrosion potentials and corrosion features. Reinforcements may interact electrochemically, chemically or physically with the matrix thereby leading to accelerated corrosion (Mahmoud *et al.*, 2012). Considering related articles:

Alaneme *et al.* (2013) studied the fabrication characteristics and mechanical behaviour of rice husk ash-alumina reinforced Al-Mg-Si alloy matrix hybrid composites and corrosion resistance of the hybrid composite was reported to increase with increase in wt.% of RHA in alloy matrix. Also Alaneme *et al.* (2013) investigated the mechanical and corrosion behaviour of SiC, bamboo ash reinforced Al-Mg-Si alloy hybrid composite and reported the corrosion

resistance of the hybrid composite was superior in basic solution as compared to acidic solution. Mahmoud *et al.* (2012) studied mechanical and corrosion behaviours of Al/SiC and Al/Al₂O₃ metal matrix nanocomposites fabricated using powder metallurgy route and observed that both Al/SiC and Al/Al₂O₃ showed better corrosion resistance in NaCl solution. The Al/ Al₂O₃ MMNCs displayed lower corrosion rates than the Al/SiC nanocomposites. The corrosion resistance decreased with increase size and volume fraction of the nanoparticles. David *et al.* (2013) investigated the synthesis and characterization of Al6061-Fly Ash-SiC composites by stir casting and compocasting methods and observed that impeding the formation of the aluminium carbide phase by fly ash helped to improve the corrosion performance of aluminium hybrid composites. Bobic *et al.* (2009) also studied corrosion of aluminium and Zn-Al Alloys Based Metal-Matrix Composites. The corrosion resistance ZA-27 alloy substrate was reported to increase with the development of reinforcing phase content in composite material, that is, the corrosion rate was found to decrease with time.

(c) Wear behavior

Alaneme and Sanusi (2015) investigated microstructural characteristics, mechanical and wear behaviour of Aluminium matrix hybrid composites reinforced with alumina, rice husk ash and graphite. They observed the presence of graphite helped to lessen the wear rate of the composites by providing a solid lubricating layer between the composite and the rubbing hard counter surface, i.e. wear resistance decreased with increase in the graphite content from 0.5 to 1.5 wt.%. Also, Alaneme *et al.* (2013) studied the fabrication characteristics and mechanical behaviour of rice husk ash - alumina reinforced Al-Mg-Si alloy matrix hybrid composites. The wear rate of the hybrid composite was reported to increase with increase in wt.% of the rice husk ash in alloy matrix. Himanshu *et al.* (2014) investigated the mechanical and tribological behaviors of stir cast aluminum matrix composites. It was observed that there was decrease in friction coefficient in case of tribological behavior. Iglesias *et al.* (2013) studied steel machining chips as reinforcements to improve sliding wear resistance of metal alloys: Study of a model Zn-based alloy system, and the wear resistance of the Kirksite matrix reinforced with steel machining chips was reported to be due to the steel chips showing insignificant wear during the sliding with difference to significant material removal from the matrix. Kumar *et al.* (2013) also investigated tribological behaviour of dual reinforced aluminium based metal matrix composite incorporated with zircon sand and silicon carbide. It was reported that the wear resistance of the dual reinforced composite was higher than both single reinforced composite and pure alloy at low and high loads.

Mitrovic *et al.* (2012) investigated tribological potential of hybrid composites based on Zinc and Aluminium Alloys reinforced with SiC and Graphite Particles. It was reported that the hybrid composites displayed better wear resistance and lower coefficient of friction when compared to pure alloys. Miloradovic *et al.* (2013) investigated tribological behaviour of Z-27Al/10SiC/1gr hybrid composite. The wear volume of the composite was observed to increase with increase in normal load and sliding speed. Prasad *et al.* (2011) also studied dry sliding wear behaviour of Al/flyash/graphite hybrid composite and reported an improved tribological characteristic and reduced wear losses from the composites. Pruthviraj (2011) studied wear characteristics of chilled Zn-Al alloy reinforced with silicon carbide particulate composites and observed that the wear resistance of the MMCs produced improved with increasing dispersoid content and copper chill was reported to have a major effect on wear because of its high Volumetric Heat Capacity.

2.7 STIR CASTING METHOD OF DEVELOPING ZA ALLOY MATRIX COMPOSITES

Stir casting as earlier discussed under MMCs is the most economical and adaptable route for ZA alloy matrix composites due to its simplicity, low processing cost, flexibility, and high production rate among others, used to synthesize discontinuously reinforced composites. It is an effective method of fabricating ZA alloy composites with the reinforcement (up to 25 wt.%) getting near uniform distribution in the matrix with less agglomeration and cleaner interface (Viswanath *et al.*, 2015). Fatile *et al.* (2015) studied the fabrication characteristics and mechanical behaviour of stir cast ZA-27 matrix hybrid composites with weight percent of reinforcements and via two-step stir casting method. Some of the measure taken to assess the performance of the produced composites includes the density measurement, estimated percentage porosity, tensile testing, micro hardness measurement and optical microscopy. Also the hardness, ultimate tensile strength, and percent elongation of the hybrid composites were shown to decrease with increase in fly ash content.

2.7.1 Stir Casting process parameters

The nature of the matrix and reinforcing materials influence the processing route and the final properties of MMCs as most of the considered parameters during MMCs design are linked with the reinforcements, (Alaneme and Aluko, 2012). Considerations includes the type, shape, size, hardness, elastic modulus and distribution of the reinforcements in the matrix, (Das *et al.*, 2014).

2.7.2 Reinforcements

Reinforcements are the materials which are embedded into a substance in order to improve some desired properties of the materials. The addition of reinforcements to a matrix is because conventional materials are not strong which may be due to the presence of defects of various kinds and because of this cannot be used for structural and load-bearing purposes. The choice of reinforcements becomes more daring with increase in the melting temperature of matrix materials.

Ceramic materials are the reinforcement usually used for MMCs. There are three main classifications of reinforcing materials used in the development of MMCs which includes: synthetic ceramic particulates (for example, silicon carbide, alumina), industrial wastes (for example, machining chips, fly ash, graphite, quarry dust particles) and agro waste derivatives (for example, rice husk, bread seed, corn cob), (Bodunrin *et al.*, 2015).

Considering the earliest work that involved the use of synthetic ceramic particulates Al_2O_3 as a reinforcement, 50 μm long and a few microns in diameter of tiny filamentary crystals had great strength and stiffness for composite materials. Also in the combination of alumina and rice husk ash hybrid reinforced Aluminum matrix composites favorable levels of ductility and fracture toughness comparable to that of single alumina reinforced composites were observed (Alaneme *et al.*, 2013). Silicon carbide among others due to its high stability and wettability is the most popularly used ceramic reinforcement. Silicon carbide has been observed to improve strength (Wang *et al.*, 2012), wear resistance of MMCs (Lim *et al.*, 2003) and creep resistance (Viswanath *et al.*, 2015). Examples of synthetic ceramic particulates are Silicon carbide (SiC), alumina (Al_2O_3), boron carbide (B_4C), tungsten carbide (WC), graphite (Gr), carbon nanotubes (CNT) and silica (SiO_2), (Bodunrin *et al.*, 2015).

Considering industrial wastes, machining chips which are produced in different shapes and morphologies enabling new reinforcement topologies has emerged as an attractive option of reinforcement. Chips improve mechanical properties by strengthening as a result of microstructure refinement occurring by large strain deformation in machining. Also machining chips offer better solutions for chip recycling and reuse than the high energy intensive methods of melting and reprocessing (Iglesias *et al.*, 2013). Examples of industrial wastes include Fly Ash, Red mud, Machining Chips among others (Bodunrin *et al.*, 2015).

Also, there is the use of agro waste derivatives as reinforcement because of their low cost, accessibility, low density, and reduced environmental pollution. This is due to the limited

availability of synthetic reinforcing materials in most developing countries, the high cost of procurement in countries where these reinforcing materials are available or most developing countries are not as industrialized as developed countries so the use of industrial waste (fly ash) is quite scarce as these wastes are limited. The specific strength, percent elongation and fracture toughness of the 2 wt.% RHA containing hybrid composite was higher than that of the single Al_2O_3 reinforced and other hybrid composite compositions worked on (Alaneme *et al.*, 2013), though the properties obtained are inferior to that offered by synthetic reinforcement. Examples of Agro waste derivatives from early articles includes bagasse ash (BA), bamboo leaf ash (BLA), bean shell waste ash (BSWA), corn cob ash (CCA), maize stalk ash (MSA), palm kernel shell ash (PKSA), rice husk ash (RHA).

The use of a single reinforcement in Zn-Al may sometimes lead to the deterioration in the values of its physical properties and this brought about the use of use of two different types of reinforcement in MMC production are called Hybrid MMCs, (Prakash and Pruthviraj, 2011).

Hybrid MMCs with two synthetic ceramic materials can be; an agro-waste derivative combined with synthetic ceramic materials; and industrial waste combined with synthetic reinforcement (Bodunrin *et al.*, 2015).

Considering Hybrid MMCs with synthetic and industrial waste reinforcement from a number of review papers, (Prasat and Subramanian, 2013) studied the tribological properties of AlSi10Mg/fly ash/graphite hybrid metal matrix composite. They observed the mechanical properties improved in the hybrid composite compared to unreinforced alloy and alumina-graphite composites. Also the wear resistance was enhanced due to load bearing capability of the fly ash (industrial waste) and the lubricating effect of graphite. David *et al.* (2013) synthesized and characterized aluminium hybrid composites reinforced with SiC and FA via stir casting technique and compocasting. The Mechanical properties were observe to improve due to high dislocation as a result of thermal mismatch between the reinforcement and the matrix along with large surface area of the hard ceramic phase, which bears the load transferred by the matrix when subjected to loading conditions. The hybrid composites possess values of hardness, coefficient of friction in between that of Zn-Al-SiC and Zn-Al graphite composites but possess excellent wear resistance when compared with the monolithic composites.

Also considering hybrid MMCs with synthetic and agro waste derivatives as reinforcement from early works, it was observed that the agro waste has some advantages which includes low cost, low density and accessibility. Alaneme *et al.* (2013) studied the fabrication characteristics and mechanical behaviour of rice husk ash- alumina reinforced Al-Mg-Si alloy

matrix hybrid composite produced via stir casting and observed there was slight decrease in the mechanical properties compared with the single reinforced Al-Mg-Si/Al₂O₃ composites. Prasad *et al.* (2014) also investigated stir cast aluminium hybrid composite containing equal amount of rice husk ash and silicon carbide from 2 % to 8 % in step of 2 and observed the homogenous distribution of the reinforcement in the matrix. Also that the mechanical properties increased with increase in the reinforcement.

CHAPTER THREE

3. MATERIALS AND METHODS

3.1 MATERIALS

The various raw materials selected for the production of the Zn-Al hybrid composite are the commercial pure Zinc, commercial pure Aluminium, Medium steel machining chips (75 μm), and graphite.



a)



b)



c)



d)

Figure 3.1: (a) Medium Steel Machining chips; (b) Commercial pure Zinc; (c) Commercial pure Aluminium; and (d) Graphite

3.2 EQUIPMENT

The equipment employed for the production are presented in Table 3.1

Equipment	Location
Crucible Furnace Auto LAB Potentiostat Hardness Testing Machine Taber Abrasion Machine Pulverizer	Federal University of Technology, Akure (FUTA)

Ball mill	
Universal Tensile Testing Machine	Obafemi Awolowo University, Ife (OAU)
Scanning Electron Microscope	Engineering Materials Development Institute (EMDI)
Optical Microscope	

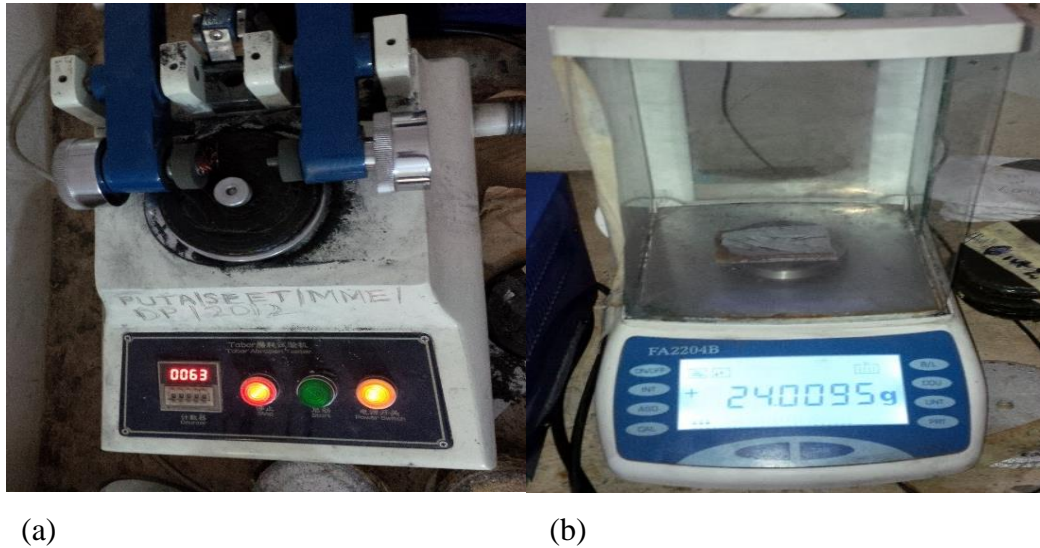


Figure 3.2 (a) Taber Abrasion Machine (b) Weighing balance

3.3 RESEARCH DESIGN

This research involved both qualitative and quantitative approaches. This implies that the study was by mixed research approach. Series of experiment were carried out to determine the specimen's mechanical properties, corrosion and wear resistance of the composites and these were studied quantitatively. Other experiments carried out to examine the microstructure of the composites required qualitative assessment approach. The experiments conducted for quantitative assessment are outlined in Table 3.2

The experimental design for the proposed research is as presented in Table 3.2

Compositions	Hardness Test	Tensile Test	Wear Test	Fracture Toughness	Corrosion NaCl	Total
Composition 1	3	3	3	3	3	15
Composition 2	3	3	3	3	3	15

Composition 3	3	3	3	3	3	15
Composition 4	3	3	3	3	3	15
Composition 5	3	3	3	3	3	15
Total						75

3 repeat tests were carried out for each experiment conducted that is, 3 tests per specimen, making a total of 75 tests that was carried out for quantitative assessments.

3.4 EXPERIMENTAL PROCEDURE

3.4.1 Charge calculation

The charge calculation of both the matrix and reinforcements are presented in Table 3.3

s/n	Composition	Zinc	Aluminium	SMC	Graphite
1.	Zn-27Al (control)	2313.77	855.77	0	0
2.	Zn-27Al + 7wt.%SMC	2200.48	813.88	226.88	0
3.	Zn-27Al + 6wt.%SMC +1wt.% graphite	2049.84	758.17	52.84	158.51
4.	Zn-27Al + 5wt.%SMC +2wt.% graphite	2080.31	769.43	107.25	107.25
5.	Zn-27Al + 4wt.%SMC +3wt.% graphite	2092.56	773.96	161.83	53.93

3.4.2 Mould preparation

The moulding sand with little quantity of bentonite and water were mixed together then the moulding board was placed on the foundry floor and the drag placed on it. The pattern was placed within the drag (moulding flask) directly on the board. Dry facing sand was sprinkled over the board and pattern to provide a non-sticky layer and the moulding sand was then riddled in to cover the pattern with the fingers, after which the drag was completely filled. The sand was then firmly packed in the drag by means of hand rammers in such a way that it is neither too hard (which could lead to blow holes due to trapping of gases within the mould cavity) nor too soft (as erosion by the molten metal when poured may occur). After the

ramming is the striking off of excess sand using the strike-off bar. Vent holes are made in the drag to the full depth of the flask as well as to the pattern using vents rod to facilitate the removal of gases during pouring and solidification. The finished drag flask is then turned over to the bottom board exposing the patterns. The cope half of the flask is then placed on the drag and aligned with the help of pins. Dry parting sand is sprinkled all over the drag and the patterns. Sprue pins for making sprue passages were located at a small distance from the pattern, the operation of filling, ramming and venting of the cope proceed in the same manner as performed in the drag. The sprue pins were removed first with care and a pouring basin was scoped out at the top to pour the liquid metal, then the pattern was removed from the drag and facing sand was applied all over the mould cavity and runners which gave the casting a good surface finish.



(a)



(c)



(b)



(d)

Figure 3.3 (a) – (d) Moulding Flasks

3.4.3 Melting and Casting

This encompasses the production process of the composite. The production of the composites was performed using double stir casting process in accordance with Alaneme and Adewale (2013). Charge calculation was used to determine the amount of reinforcements (steel machining chips and graphite) required to prepare the five compositions as in Table 3.3.

The melting was done using the gas-fired crucible furnace (fitted with a temperature probe). The commercially pure Aluminium was charged in first and heated to about 670 °C until the Aluminium melted completely. The temperature of the furnace was then reduced to about 500 °C before Zinc was introduced into the aluminium melt. After the Zinc has melted completely, the melt was then allowed to cool in the furnace to a semi solid state and it was stirred for about 5 minutes in order to obtain homogenization of the molten metal. The medium steel machining chips and the graphite were then charged into the melt and stirring of the slurry was performed manually for 7-10 minutes. The slurry was placed again inside the furnace and allowed to superheat. The second stirring was performed for about 3 minutes by manual stirrer for homogenization of the mixture, after which the slurry was poured into the mould cavity (i.e. casting), which makes the process a double stir casting process. The cast was then removed from the mould after complete solidification had taken place.

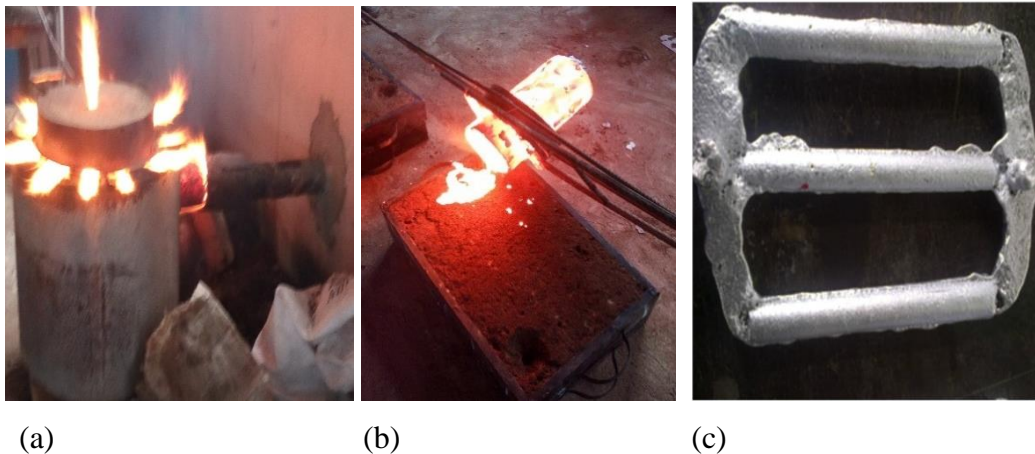


Figure 3.4: (a) - (c), Melting and Casting process

3.4.4 Mechanical properties evaluation

(a) Hardness Test

Hardness is the resistance of a material to deformation, while, hardness testing measures the strength of a material by determining its resistance to penetration. Hardness test is very useful in the selection of materials since it indicates how well a material will wear and can easily be

machined. The hardness values of the samples were determined according to the provisions in ASTM E18-16 (2016) using Rockwell Hardness Tester. Before the test was carried out the mating surfaces of the indenter, plunger rod and test samples were cleaned by removing dirt, scratches and oil and the testing machine was calibrated using the standard block. An indentation load of 60 kgf was then applied on flat smoothly polished specimens of the composites for 10 seconds and the hardness readings was evaluated following standard procedures. Four repeat tests were carried out on each sample per composition and the average value was taken as a measure of the hardness of the specimen. (Laboratory testing inc.)



Figure3.5: Hardness testing machine

(b) Tensile Testing

Tensile test is a destructive test process that provides information about the tensile strength, yield strength and the ductility of the material (Laboratory testing inc.).

A universal testing machine (UTM) operated at nominal strain rate of 10^{-4} /s (quasi-static strain rate) was used for the tensile testing of the composite samples which were machined with a gauge length of 30 mm and diameter of 4.4 mm. the samples were pulled in tension to fracture and stress strain curves were generated for each sample in accordance with ASTM E8M-15a (2015) standard. The tensile properties evaluated from the stress-strain curves are ultimate tensile strength, yield strength, % elongation and tensile toughness.



Figure3.6: A tensile test piece.

(c) Fracture Toughness (K_{IC}) Evaluation

Fracture toughness is the ability of a material containing a crack to resist fracture.

The fracture toughness of the Zn-27Al composites was determined using circumferential notch tensile (CNT) testing approach in accordance with Alaneme *et al.* (2013).

The composites for the test were machined having gauge length of 30 mm, specimen diameter of 4.4 mm (d₁), notch diameter of 3.6 mm (d₂), and notch angle of 60° were noted. The specimens were then subjected to tensile loading to fracture using the Universal tensile testing machine. The fracture load (p_f) obtained from the CNT specimens load-extension plots were used to evaluate the fracture toughness using the empirical relations in accordance with Alaneme and Ajayi (2015).

$$k_{IC} = 0.454 \sigma_{NTS} \times D^{1/2} \quad (3.1)$$

Where; D is the gauge length of the specimen,

σ_{NTS} is the tensile strength of the notched specimen

Using the above relation, the value of fracture toughness for each samples were determined.



Figure 3.7: A circumferential notch tensile (CNT) specimen

3.4.5 Wear behaviour evaluation

The wear test of the specimen according to ASTM G 99 -05 (2016) standard was determined using the Taber abrasion tester model 5135. The wear test was conducted using samples prepared in form of discs 100 mm diameter and 10 mm thick. The samples were placed on the turntable platform of the wear machine and gripped at a constant pressure by two abrasive wheels lowered onto the sample surface. In operation, the turntable rotates at 150 rpm with the samples which drive the abrasive wheels in contact with its surface. Rotation of the turntable cause the wheels to drive in reversed direction about a horizontal axis which is tangentially displaced from its axis. The rubbing action between the sample and the abrasive wheel during the rotating motion of the machine, results in the generation of loose composite debris from the sample surface. A complete circle was traversed by the action of the abrading wheels on the sample to form which reveals the abrasive resistance at every angles in relative to the strand of the material. A pattern of crossed arcs caused by the abrasion marks now formed a circular length of approximately 30 cm². The test was conducted for 15 min; and the sample weights before and after the tests are recorded.

The Taber wear index method was used to evaluate the rate of wear and the formula is given below;

$$I = \frac{[(A-B) \times 1000]}{C} \quad (3.2)$$

Where: I is wear index,

A is weight of specimen before abrasion,

B is weight of specimen after abrasion, and

C is number of test cycles (150) rpm.



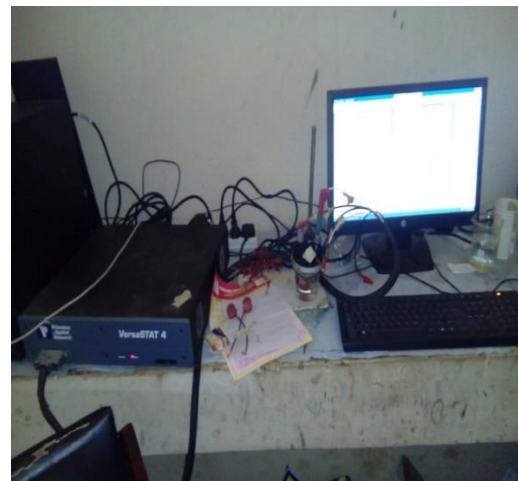
Figure 3.8: wear test specimen sample.

3.4.6 Corrosion behaviour evaluation

Corrosion testing was conducted using potentiodynamic polarization electrochemical methods in accordance with ASTM G48-11 (2015) standard. Corrosion behavior of the samples was investigated in 0.3M H₂SO₄ and 3.5 wt.% NaCl solutions at room temperature (25 °C) using an AutoLab potentiostat. The surface morphology of the composites after immersion in the corrosion solutions was assessed using the Field Emission Gun Scanning Electron Microscope (FEG-SEM).



(a)



(b)

Figure 3.9: (a) Corrosion samples (b) Autolab Potentiostat

3.4.7 Micro-structural examination

A Zeiss Metallurgical Microscope with accessories for image analysis was used for optical microscopic investigation of the composites produced. The specimens for the test were metallographically polished and etched before microscopic examination was performed. The

specimens for the test were machined from rods into cylindrical shapes of dimensions 10 mm diameter and 10 mm length. The specimens were ground under water on a rotating disc using emery papers of increasing grits up to 1200grits and then polishing was done using polishing cloth and polishing paste with Al_2O_3 particles. The Polished samples were etched in dilute aqua regal (HCl and HNO_3) before they were viewed under microscope.



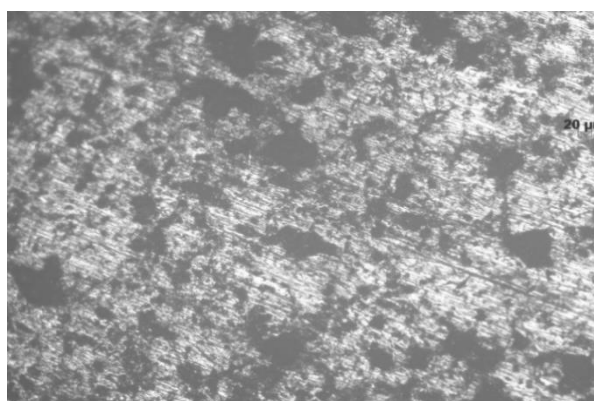
Figure 3.10: A metallurgical microscope

CHAPTER FOUR

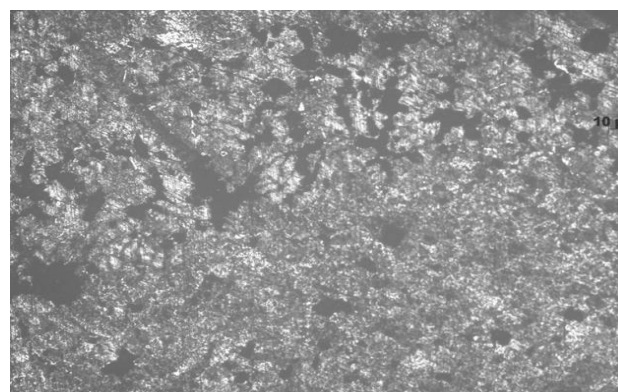
4. RESULTS AND DISCUSSIONS

4.1 MIROSTRUCTURAL EXAMINATION

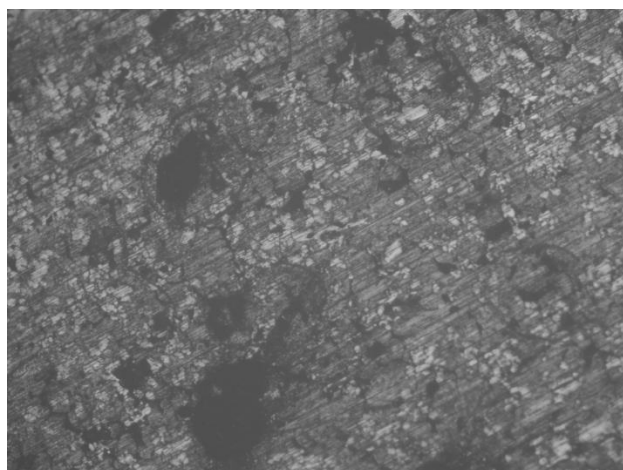
Figure 4.1(a, b, c) shows the optical micrographs of selected compositions of the Zn–27Al alloy matrix composites produced. The microstructures appear very similar, consisting of the reinforcing particles almost evenly dispersed within the Zn–27Al alloy matrix, although some particles agglomeration could also be observed. The grain structure and morphology for all the composite compositions appeared to have the same features.



(a)



(b)



(c)

Figure 4.1: Optical Micrographs of (a) Composition B(x100) (b) Composition D(x50) and (c) Composition E(x100)

4.2 MECHANICAL BEHAVIOUR

The results of the mechanical properties evaluated for the composites are presented in Figures 4.2-4.6.

Figure 4.2 compares the Hardness values of the composites produced. It is observed that the hardness of the composite with steel machining chips alone decreased while the hardness of the other composite grades having both steel machining chips and graphite as reinforcements increased. Composition C with 6 wt.% SMC and 1 wt.% graphite has the highest hardness value, while Composition B with 7 wt.% SMC has the lowest hardness value. Therefore, the hardness of the composite increased with the addition of graphite.

Figure 4.3 compares the ultimate tensile strength for the Zn–27Al based composites produced. It is observed generally that the ultimate tensile strength for the composites increased with the addition of the reinforcements. Although the increment in the ultimate tensile strength did not follow a consistent trend, but Composition E (with 4 wt.% SMC + 3 wt.% graphite) has the highest value of ultimate tensile strength.

Figure 4.4 compares percentage elongation for the Zn–27Al based composites produced. It is observed generally that the percentage elongation of the composites reduced with addition of the reinforcements. It can be deduced that the %elongation of the composites reduced with increase in weight percent of graphite but Composition B (with 7 wt.% SMC) still showed the lowest %elongation.

Figure 4.5 compares elastic modulus for the Zn–27Al based composites produced. It is observed generally that the elastic modulus of the composites increased with the addition of the reinforcements, although there is variation in the increment. Composition D (with 5 wt.% SMC+ 3 wt.% graphite) has the highest elastic modulus value.

Figure 4.6 compares fracture toughness for the Zn–27Al based composites produced. It is also noted generally that the fracture toughness increased with the addition of the reinforcements but in a varying trend with Composition E (with 4 wt.% SMC + 3 wt.% graphite) having the highest value of fracture toughness. However, it can be deduced that the fracture toughness increased with increase in weight percent of graphite and decrease in weight percent of steel machining chips.

It can be deduced from the mechanical test results that Composition E (with 4 wt.% SMC + 3 wt.% graphite) is the composite grade with the optimum combination of the mechanical properties and therefore the most suitable for stress bearing and structural applications.

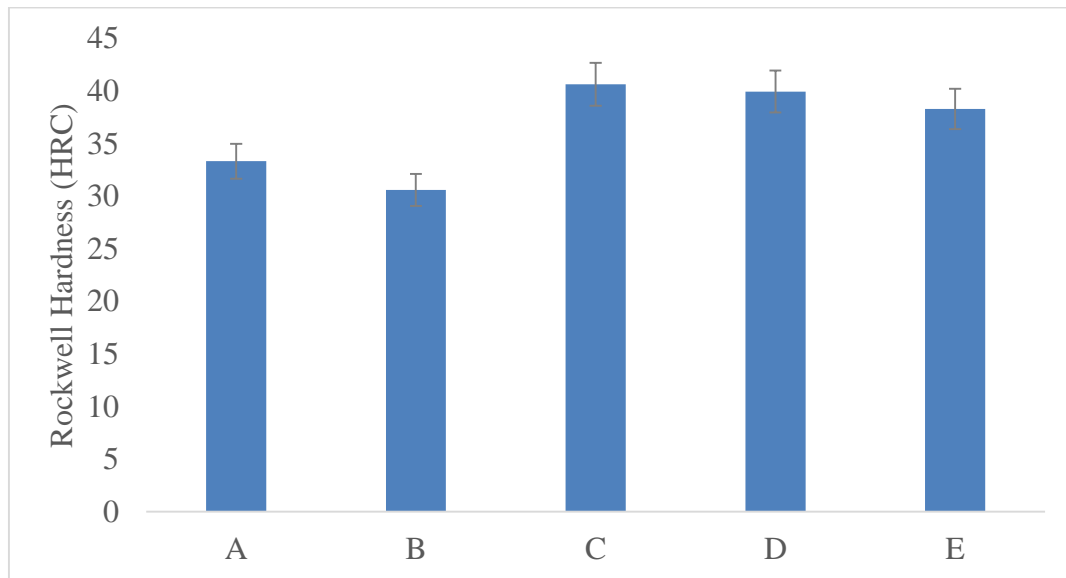


Figure 4.2: Variation of Hardness for the Zn-27Al based composites produced

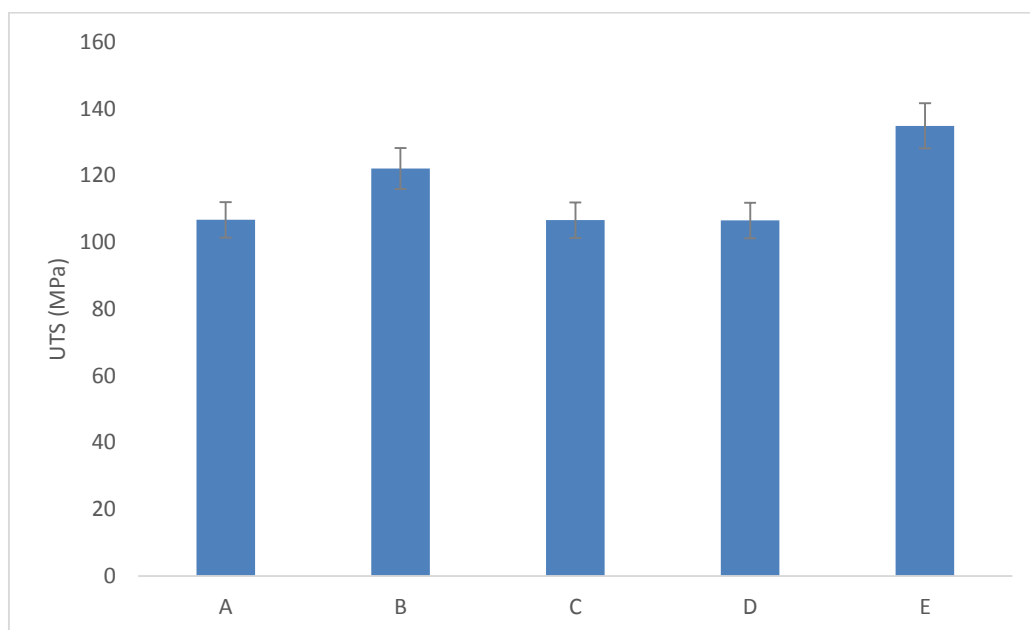


Figure 4.3: Variation of ultimate tensile strength for the Zn-27Al based composites produced.

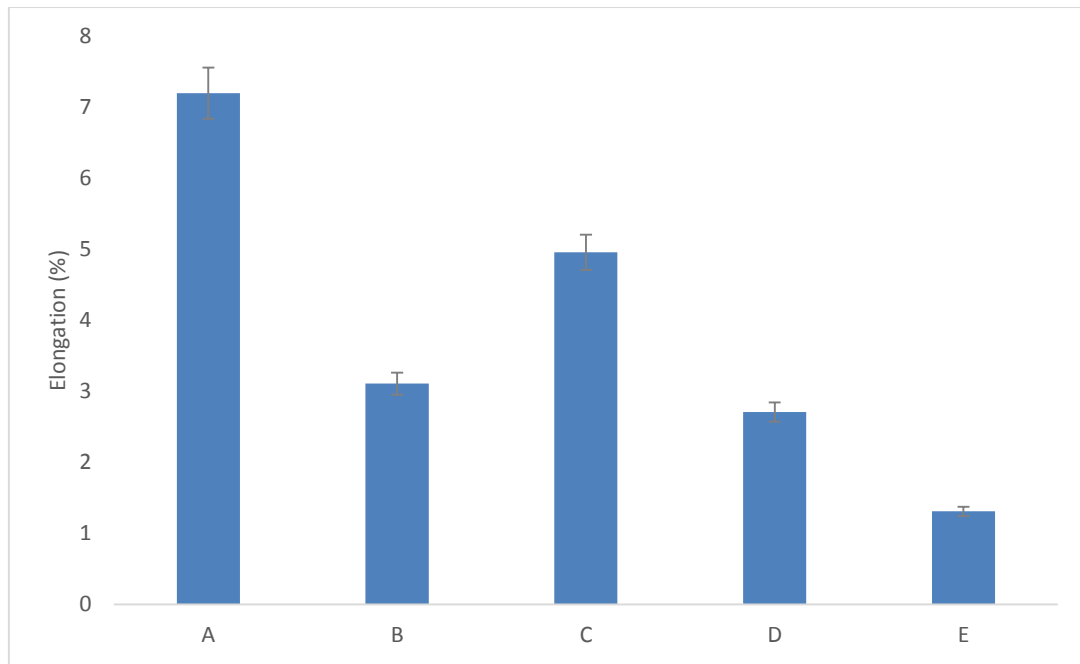


Figure 4.4: Variation of percentage elongation for the Zn-27Al based composites produced.

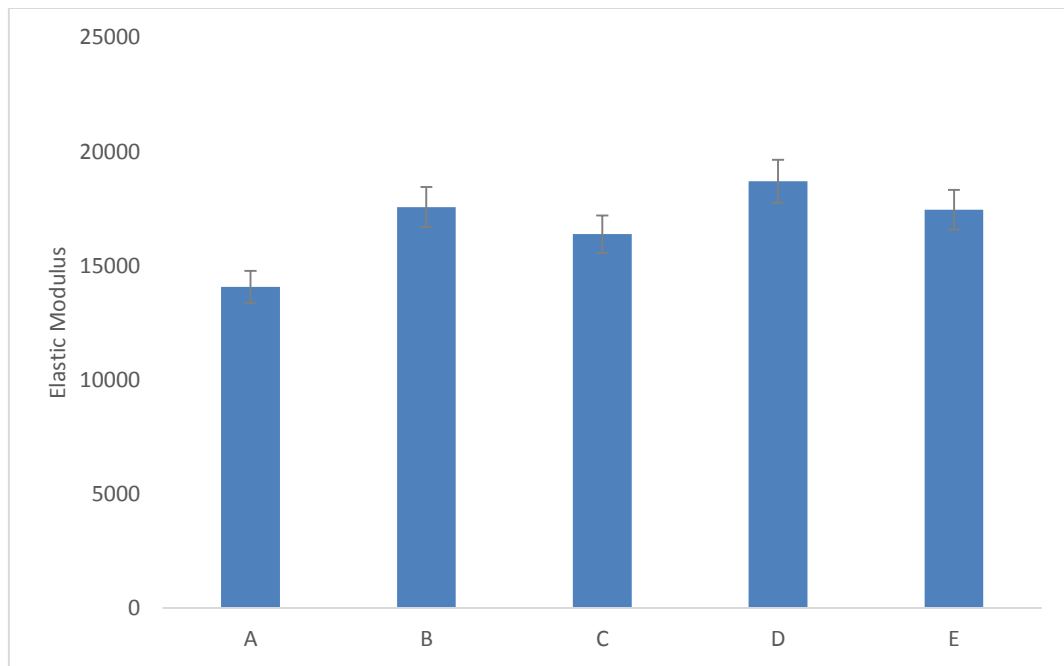


Figure 4.5: Variation of elastic modulus for the Zn-27Al based composites produced.

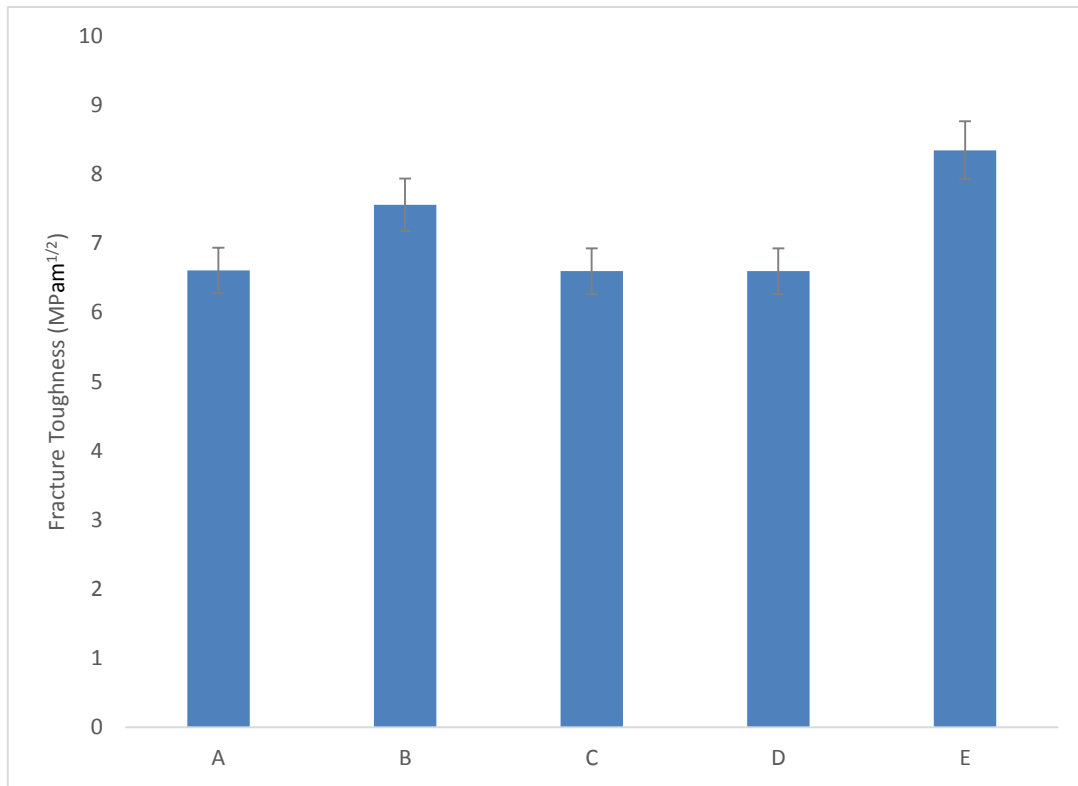


Figure 4.6: Variation of fracture toughness for the Zn–27Al based composites produced.

FRACTOGRAPHIC ANALYSIS

Figure 4.7 shows the fractographs of some selected compositions of the composites produced. Figure 4.7(a) containing 7 wt. % SMC shows greater dominance of dimple features and some regions of grain facet, which indicates mixed mode fracture. Figure 4.7(b) containing 6 wt. % SMC+ 1wt. % graphite shows a combination of cleavage and dimple rupture but greater dominance of fibrous features than granular. These manifestation shows that the fracture of the sample is a characteristics of mixed mode fracture.

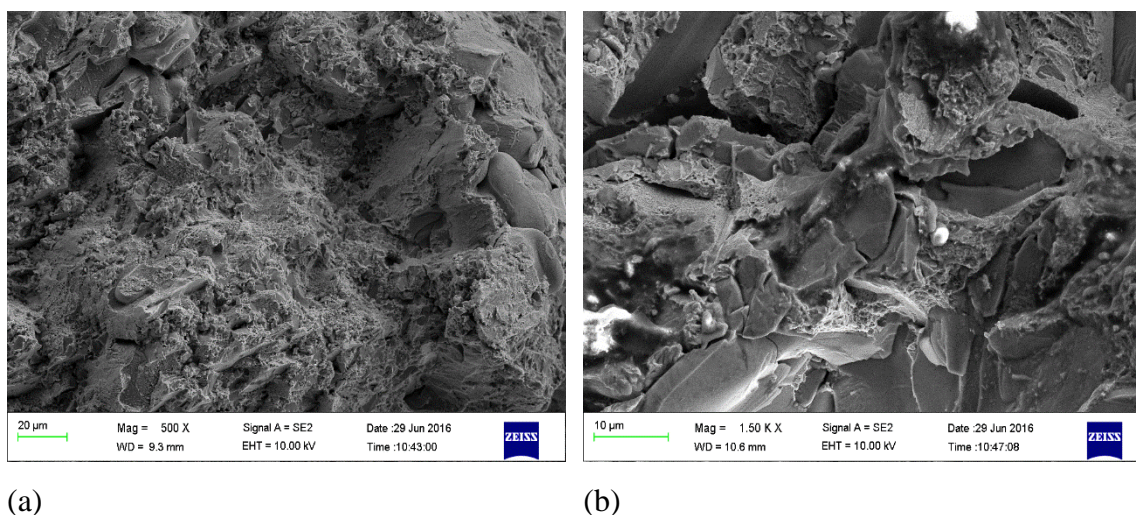


Figure 4.7: SEM fractographs of (a) Composition B and (b) Composition C.

4.3 CORROSION BEHAVIOUR

Tafel plots extrapolations from the electrochemical studies of the Zn-27Al matrix composites produced in 3.5 wt.% NaCl solution is presented in Figure 4.8. It is observed that the composites exhibited similar polarization and passivity characteristics apart from composition D with polarization curve displaced to higher potentials. However, the corrosion current densities (I_{corr}) and corrosion potentials (E_{corr}) indicate clear distinct corrosion behavior between the reinforced composite series and the unreinforced Zn-27Al alloy. It is observed from Table 4.1 that the corrosion current density and the corrosion rate were relatively more intense for the unreinforced Zn-27Al alloy in comparison with the reinforced Zn-27Al alloy matrix composites with composition D having the lowest I_{corr} value and corrosion rate. This indicates that the steel chips reinforced Zn-27Al alloy matrix composites are more resistant to corrosion in 3.5 wt.% NaCl solution compared with unreinforced Zn-27Al alloy in the same environment. The E_{corr} values are supportive of the I_{corr} trends as it is observed that the steel chips reinforced composites have higher corrosion potentials compared with the unreinforced Zn-27Al alloy. This indicates that the reinforced Zn-27Al alloy matrix composites have a lower thermodynamic tendency to corrode in 3.5 wt.% NaCl solution compared to the unreinforced Zn-27Al alloy, that is, the steel chips reinforced Zn-27Al alloy matrix composites are more thermodynamically stable in 3.5 wt.% NaCl solution.

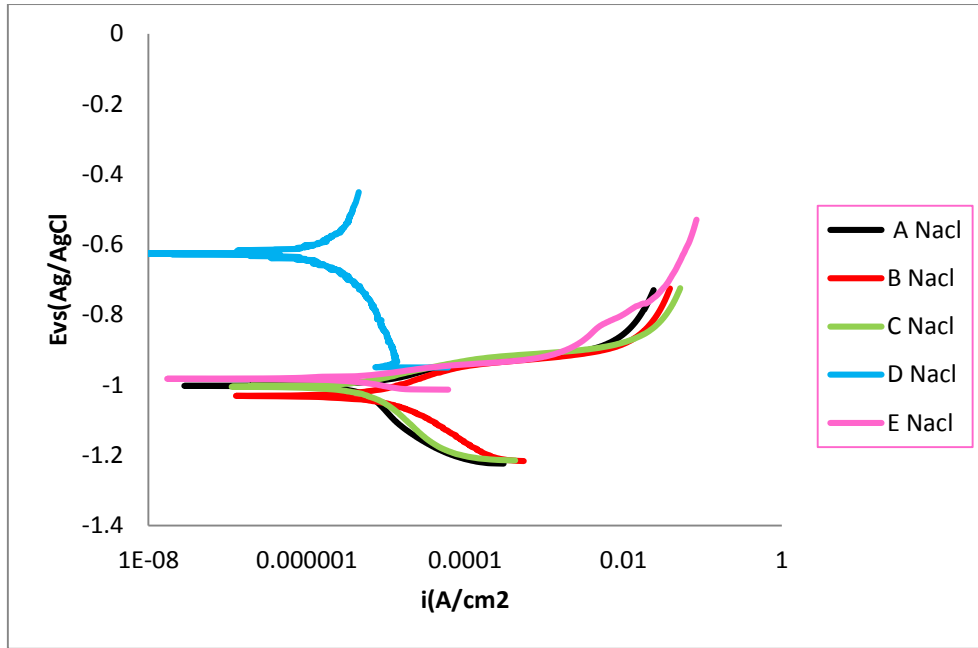
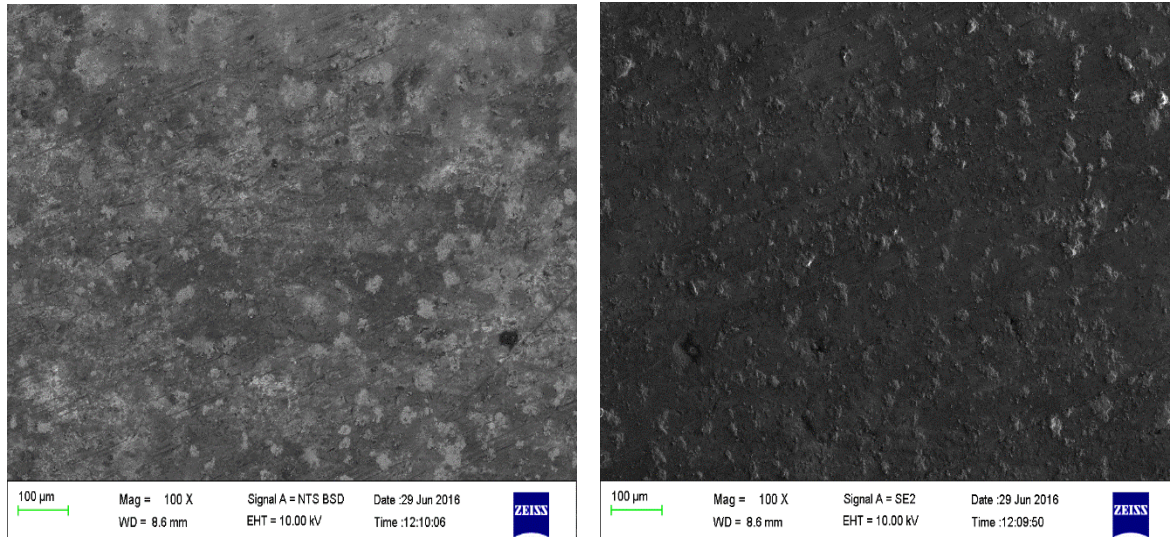


Figure 4.8: Polarization curves of the unreinforced Zn-27Al alloy and Zn-27Al matrix composites produced in 3.5 wt.% NaCl solution.

Table 4.1: Electrochemical data for the unreinforced Zn-27Al alloy and Zn-27Al matrix composites produced in 3.5 wt.% NaCl solution.

Sample Compositions	E_{corr} (V)	I_{corr} (μA)	Corrosion Rate (mmpy)
A	-1.002	6.542	0.098032
B	-1.031	4.111	0.061599
C	-1.003	5.498	0.082387
D	-0.624993	0.872	0.013062
E	-0.980965	3.415	0.051168

Figure 4.9(a) and (b) shows the SEM image of the surface morphology of a representative sample of the unreinforced Zn-27Al alloy sample (Composition A) and the reinforced Zn-27Al alloy matrix composite (Composition E) after the electrochemical test in 3.5 wt.% NaCl solution. It was observed that the corrosion proceeds uniformly over the surface of the samples, exposing the particulates. This suggests that uniform corrosion is the primary corrosion mechanism.



(a)

(b)

Figure 4.9: Representative SEM Micrographs showing surface morphology of (a) Composition A in 3.5 wt. % NaCl solution (b) Composition E in 3.5 wt. % NaCl solution.

4.4 WEAR BEHAVIOUR

It was observed from Figure 4.9 that the wear index (a measure of wear rate) does not follow a particular trend. However, composition B containing 7 wt. % SMC has the lowest wear index, that is, it has the lowest wear rate. Composition C has the highest wear index, that is, it has the lowest wear resistance. SMC can be observed to increase the wear resistance while the presence of graphite tends to reduce the wear resistance of the composite. This indicates that steel machining chips can serve as reinforcement in ZnAl based composites intended for wear resistance applications.

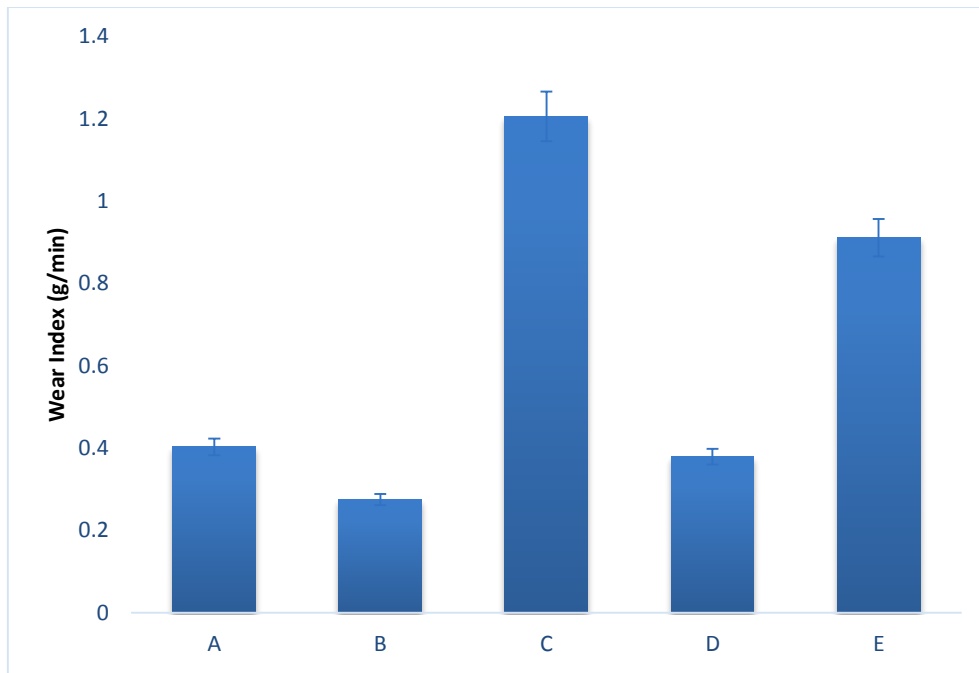


Figure 4.10: Variation of wear rate for the unreinforced Zn-27Al alloy and Zn-27Al hybrid composites.

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The mechanical properties, wear and corrosion behaviour of Zn-Al alloy matrix composites reinforced with graphite and steel machining chips has been investigated. The results have shown clearly that graphite and steel machining chips can serve as a cost effective and technically reliable reinforcement for the development of Zn-Al alloy matrix composites. Specifically:

1. The mechanical properties (hardness, ultimate tensile strength, elastic modulus and fracture toughness) of the composites generally increases with increase in weight percent of the reinforcements while the % elongation unreinforced Zn-27Al alloy has higher % elongation compared with the reinforced composites produced.
2. The wear resistance did not follow a consistent trend but the composite grade with the highest weight percent of steel machining chips was observed to have improved wear resistance.
3. The corrosion susceptibility in 3.5 wt.% NaCl solution were observed to be relatively more intense for the unreinforced Zn-27Al alloy grade compared with the steel chips and graphite reinforced Zn-27Al alloy matrix composites.
4. The corrosion mechanisms for the corrosive medium followed the same trend that revealed uniform corrosion and the fracture mechanism demonstrated by the composites is mixed mode fracture.

5.2 RECOMMENDATION

I suggest that the use of metallic moulds and other weight percent ratios of the reinforcements should be considered for the production of the composites. Also, creep behaviour and coefficient of thermal expansion of the composites should be investigated.

5.3 CONTRIBUTIONS TO KNOWLEDGE

On completion of this research, it has been able to:

- a) Extend the Frontiers of knowledge in the science of metal matrix composite development.
- b) Proffer a scientific basis for the utilization of steel machining chips (an industrial waste) for low cost development of Zn-27Al alloy matrix composites which serves as a viable alternative for recycling of industrial waste for productive use.

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APPENDIX

Table 1: Hardness values the composites

Sample Designation	Composition	H1	H2	H3	H4	Average Hardness
1	Control sample	33.9	33.7	32.4		33.3333
2	Zn-27Al + 7wt.%SMC	20.6	30.6	28.0	23.0	30.6
3	Zn-27Al + 6wt.%SMC +1wt.% graphite	37.2	39.8	41.5	34.7	40.65
4	Zn-27Al + 5wt.%SMC +2wt.% graphite	44.3	40.8	37.9	39.1	39.95
5	Zn-27Al + 4wt.%SMC +3wt.% graphite	33.8	39.2	37.4		38.3

Table 2: Mechanical test results

S/n	S/D	composition	UTS (MPa)	%E (%)	E (MPa)	Strain Energy Absorbed	Average Hardness	$\sigma_{NTS.I}$	K _{IC}
1.	A1	Control sample	178.86	6.10	8731.94	2.23		106.68	6.61
2.	A2	Control sample	257.30	7.20	14069.14	4.67			
3.	B1	Zn-27Al + 7wt.%SMC	102.06	1.14	17570.70	0.17	30.6	122.06	7.56
4.	B2	Zn-27Al + 7wt.%SMC	95.86	3.11	16948.55	0.88	30.6	74.07	
5.	C1	Zn-27Al + 6wt.%SMC	20.33	3.03	0	0.09	40.65	71.99	6.60

		+1wt.% graphite							
6.	C 2	Zn-27Al + 6wt.%SMC +1wt.% graphite	51.62	1.67	6093.33	0.19	40.65	91.12	
7.	D 1	Zn-27Al + 5wt.%SMC +2wt.% graphite	138.13	2.78	18700.9 5	1.08	39.95	70.16	6.6 0
8.	D 2	Zn-27Al + 5wt.%SMC +2wt.% graphite	113.92	1.11	15617.8 7	0.24	39.95	106.52	
9.	E 1	Zn-27Al + 4wt.%SMC +3wt.% graphite	70.39	0.81	14123.4 0	0.09	38.3	43.64	8.3 5
10 .	E 2	Zn-27Al + 4wt.%SMC +3wt.% graphite	134.85	1.31	17452.3 4	0.31	38.3	134.85	

Table 3: Tensile properties of the composite compositions.

S/n	SD	UTS (Mpa)	E (%)	E	$\sigma_{NTS.I}$	KIC	Hardness (HRC)
1	A	178.86	7.2	14069.14	106.68	6.61	33.33
2	B	102.06	3.11	17570.7	122.06	7.56	30.60
3	C	158.5	4.96	16385.05	106.6	6.60	40.65
4	D	138.13	2.71	18700.95	106.52	6.60	39.95
5	E	134.85	1.31	17452.34	134.85	8.35	38.30

Table 4: Wear loss calculation

Samples	Initial weight loss (g)	Final weight loss (g)	$W.I = \frac{[(A-B) \times 150]}{C}$ (grams/min)
A	179.45	179.39	0.40
B	108.34	108.30	0.28
C	166.85	166.66	1.21
D	191.53	191.47	0.38
E	152.57	152.44	0.91

Where;

W.I is the wear index

A is the weight of specimen before abrasion

B is the weight of specimen after abrasion

C is the number of test cycles (150rev/min)

CHAGRE CALCULATION

Volume of the Rod (pattern) = $\pi r^2 h$ but $r = 1\text{cm}$, $h = 20\text{cm}$, $d = 2\text{cm}$

$$22/7 \times (1.0)^2 \times 20 = 62.8319\text{cm}^3$$

Volume of Disc = $\pi r^2 h$

$$= \pi \times (5)^2 \times 0.5 = \pi \times 25 \times 0.5 = 39.270\text{cm}^3$$

Density of Zn = 7.13g/cm^3

Density of Al = 2.7g/cm^3

But on 100% of the Zn – 27 Al Alloy

We have

Zn = 73%

Al = 27%

$$\text{Density of the Alloy (Zn-27Al)} = (7.13 \times 0.73) + (2.7 \times 0.27)$$

$$(5.2049) + (0.729)$$

$$5.9339 \text{ g/cm}^3$$

Since the percent of Zn = 0.73% wt

$$\text{Al} = 0.27\% \text{ wt}$$

$$73\% \text{ -Zn, } 27\% \text{ -Al} = 1.0 = 100\%$$

But reinforcement is = 7% wt

Subtracting the reinforcement from the Alloy = $100 - 7\% \text{ wt} = 93\% \text{ wt}$ of (Zn-27Al)

FOR VARIOUS COMPOSITION

Composition A = Control sample (No Reinforcement)

Composition B = 7wt% Steel Machining Chips (SMC)

Composition C = 6wt% SMC + 1wt% graphite

Composition D = 5wt% of SMC + 2wt% graphite

Composition E = 4wt% of SMC + 3wt% graphite

Using

Overall formula

Density of composite = (Density of Zn * Mass fraction of Zn) + (Density of Al * Mass fraction of Al) + (Density of SMC * Mass fraction of SMC) + (Density of graphite * Mass fraction of graphite)

Density of ZA-Alloy = (Density of Zn x Mass fraction of Zn) + (Density of Al x Mass fraction of Al)

$$= (7.13 \times 0.73) + (2.70 \times 0.27) = 5.9339 \text{ g/cm}^3$$

For composition A (No reinforcement) = 100% Zn-27Al

$$= (7.13 \times 0.73) + (2.70 \times 0.27) = 5.9339 \text{ g/cm}^3$$

ℓ = mass/vol

Mass = $\ell \times \text{vol}$

Mass for rod = 5.9339×62.8319

$$= 372.8382 \text{ g}$$

Mass for disc = $5.9339 \times 39.270 = 233.0243 \text{ g}$

For composition B

7wt% SMC + 0wt% graphite

$$\begin{aligned}\text{Density} &= 5.5185 + (27.85 \times 0.07) \\ &= 6.0680 \text{ g/cm}^3\end{aligned}$$

ℓ = mass/vol

Mass = ℓ x vol

$$\text{For rod mass} = 6.068 \times 62.8319 = 381.2640\text{g}$$

For disc mass

Volume of Disc = $\pi r^2 h$

$$\begin{aligned}&= \pi \times (5)^2 \times 0.5 = \pi \times 25 \times 5 = 39.270\text{cm}^3 \\ &= 6.068 \times 39.270 = 238.2904\text{g}\end{aligned}$$

For composition C

6wt% SMC + 1wt% graphite

$$\begin{aligned}\text{Density} &= 5.5185 + (7.85 \times 0.06) + (2.2 \times 0.01) \\ &= 5.5185 + 0.471 + 0.022\end{aligned}$$

$$\ell_{\text{comp3}} = 6.0115 \text{ g/cm}^3$$

Mass = ℓ x vol

$$\text{For rod (mass)} = 6.0115 \times 62.8319 = 377.7157\text{g}$$

$$\text{For disc (mass)} = 6.0115 \times 39.270 = 236.0716\text{g}$$

For Composition D

5wt% of SMC + 2wt% graphite

$$\begin{aligned}\text{Density} &= 5.5185 + (7.85 \times 0.05) + (2.2 \times 0.02) \\ &= 5.5185 + 0.3925 + 0.044 = 5.955 \text{ g/cm}^3\end{aligned}$$

Mass = ℓ x vol

$$\text{For rod (mass)} = 5.955 \times 62.8319 = 374.164\text{g}$$

$$\text{For disc (mass)} = 5.955 \times 39.270 = 233.8529\text{g}$$

For Composition E

4wt% of SMC + 3wt% graphite

$$\text{Density} = 5.5185 + (7.85 \times 0.04) + (2.2 \times 0.03)$$

$$5.5185 + 0.314 + 0.066 = 5.8985 \text{ g/cm}^3$$

$$\text{Mass} = \ell \times \text{vol}$$

$$\text{For rod (mass)} = 5.8985 \times 62.8319 = 370.614\text{g}$$

$$\text{For disc (mass)} = 5.8985 \times 39.270 = 231.6341\text{g}$$

Composition	Mrod (g)	Mass for 3 rods	MD(g)	Mass for 2 disc	Mass rod S.L x 2	Mass disc S.L x 2
Comp A	372.89	1118.67	233.05	466.10	2237.34	932.20
Comp B	381.32	1143.96	238.33	476.64	2287.92	953.32
Comp C	377.7157	1065.66	236.0716	444.02	2131.32	888.04
Comp D	374.1640	1081.50	233.8529	450.62	2163.00	901.24
Comp E	370.6140	1087.86	231.6341	453.28	2175.72	906.56

For composition A

$$\text{S.L (mass rod)} = 2237.34\text{g}$$

$$\text{MR (Al)} = 27/100 \times 2237.34 = 604.08\text{g}$$

$$\text{MR (Zn)} = 73/100 \times 2237.34 = 1633.26\text{g}$$

For SMC 0:0 Graphite

For disc

$$\text{Mass S.L} = 932.20\text{g}$$

$$\text{M (Al)} = 27/100 \times 932.20 = 251.69\text{g}$$

$$\text{M (Zn)} = 73/100 \times 932.20 = 680.51\text{g}$$

For composition B

$$\text{S.L (mass rod)} = 2287.92\text{g}$$

$$\text{Therefore MR} = 7/100 \times 2287.92 = 160.15\text{g}$$

$$\text{S.L-MR} = 2127.77\text{g}$$

$$\text{MR (Al)} = 27/100 \times 2127.77 = 574.50\text{g}$$

$$\text{MR (Zn)} = 73/100 \times 2127.77 = 1553.27\text{g}$$

For SMC 1:0 graphite

$$M_{\text{SMC}} = 100/100 \times 160.15 = 160.15\text{g}$$

For disc

$$\text{Mass S.L} = 953.32\text{g}$$

$$\text{But } 7/100 \times 953.32 = 66.73\text{g}$$

$$M_{\text{SMC}} = 100/100 \times 66.73 = 66.73\text{g}$$

$$953.32 - 66.73 = 886.59\text{g}$$

$$\text{For M (Al)} = 27/100 \times 886.59 = 239.38\text{g}$$

$$\text{Mg (Zn)} = 73/100 \times 886.59 = 657.21\text{g}$$

Composition C

$$\text{S.L (mass rod)} = 2131.32\text{g}$$

$$\text{Therefore MR} = 7/100 \times 2131.32 = 149.19\text{g}$$

$$\text{S.L-MR} = 1982.13$$

$$\text{Also MR (Al)} = 27/100 \times 1982.13 = 535.18\text{g}$$

$$\text{MR (Zn)} = 73/100 \times 1982.13 = 1446.95\text{g}$$

For SMC 1:3 graphite

$$M_{\text{SMC}} = 25/100 \times 149.19 = 37.30\text{g}$$

$$M_{\text{G}} = 75/100 \times 149.19 = 111.89\text{g}$$

$$\text{But, } 1982.13 - 149.19 = 888.04\text{g}$$

For disc

$$\text{Mass S.L} = 888.04\text{g}$$

$$\text{But } 7/100 \times 888.04 = 62.16\text{g}$$

$$M_{\text{SMC}} = 25/100 \times 62.16 = 15.54\text{g}$$

$$M_{\text{G}} = 75/100 \times 62.16 = 46.62\text{g}$$

$$888.04 - 62.16 = 825.88$$

$$\text{For } M_{\text{SMC}} (\text{Al}) = 27/100 \times 825.88 = 222.99\text{g}$$

$$\text{Mg (Zn)} = 73/100 \times 825.88 = 602.89\text{g}$$

Composition D

$$\text{S.L (mass rod)} = 2163\text{g}$$

$$\text{Therefore MR} = 7/100 \times 2163 = 151.41\text{g}$$

$$\text{S.L-MR} = 2011.59$$

$$\text{Also MR (Al)} = 27/100 \times 2011.59 = 543.13\text{g}$$

$$\text{MR (Zn)} = 73/100 \times 2011.59 = 1468.46\text{g}$$

For SMC 1:1 graphite

$$M_{\text{SMC}} = 50/100 \times 151.41 = 75.705\text{g}$$

$$M_{\text{G}} = 50/100 \times 151.41 = 75.705\text{g}$$

For disc

$$\text{Mass S.L} = 901.24\text{g}$$

$$\text{But } 7/100 \times 901.24 = 63.09\text{g}$$

$$M_{\text{SMC}} = 50/100 \times 63.09 = 31.545\text{g}$$

$$M_{\text{G}} = 50/100 \times 63.09 = 31.545\text{g}$$

$$901.24 - 63.09 = 838.15$$

$$\text{For } M_{\text{SMC}} (\text{Al}) = 27/100 \times 838.15 = 226.30\text{g}$$

$$M_{\text{G}} (\text{Zn}) = 73/100 \times 838.15 = 611.85\text{g}$$

For composition E

$$\text{S.L (mass rod)} = 2175.72\text{g}$$

$$\text{MR} = 7/100 \times 2131.32 = 152.30\text{g}$$

$$\text{S.L-MR} = 2023.42$$

$$\text{Also MR (Al)} = 27/100 \times 2023.42 = 546.32\text{g}$$

$$\text{MR (Zn)} = 73/100 \times 2023.42 = 1477.10\text{g}$$

For SMC 3:1 graphite

$$M_{\text{SMC}} = 75/100 \times 152.30 = 114.23\text{g}$$

$$M_{\text{G}} = 25/100 \times 152.30 = 38.07\text{g}$$

For disc

Mass S.L = 906.56g

But $7/100 \times 906.56 = 63.46\text{g}$

$M_{\text{SMC}} = 75/100 \times 63.46 = 47.60\text{g}$

$M_{\text{G}} = 25/100 \times 63.46 = 15.86\text{g}$

$906.56 - 63.46 = 843.10$

For $M_{\text{SMC}} (\text{Al}) = 27/100 \times 843.10 = 227.64\text{g}$

$M_{\text{G}} (\text{Zn}) = 73/100 \times 843.10 = 615.46\text{g}$

Final Tabular Analysis

Composition	Zn (g)		Al (g)		SMC (g)		Graphite (g)	
	R	D	R	D	R	D	R	D
Comp A	1633.26	680.51	604.08	251.69	-	-	-	-
Comp B	1553.27	647.21	574.50	239.38	160.15	66.73	-	-
Comp C	1446.95	602.89	535.18	222.99	37.30	15.54	111.89	46.62
Comp D	1468.46	611.85	543.13	226.30	75.71	31.55	75.71	31.55
Comp E	1477.10	615.46	546.32	227.64	114.23	47.60	38.07	15.86
TOTAL	10736.96		3971.21		511.80		319.69	