

MECHANICAL AND MICROSTRUCTURAL CHARACTERIZATION OF
ALUMINUM METAL MATRIX COMPOSITE REINFORCED WITH
PARTICULATE PERIWINKLE SHELLS

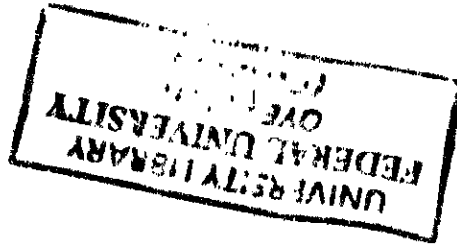
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PROJECT REPORT

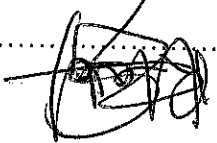
SUBMITTED IN PARTIAL FULFILLMENT FOR THE AWARD OF
DEGREE OF BACHELOR OF ENGINEERING (B.ENG) IN
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SEPTEMBER, 2016.



CERTIFICATION


I certify that this work was carried out by OLATAYE, DAMILARE JOHN with Matriculation Number MME/11/0425 under my supervision in the Department of Materials and Metallurgical Engineering, Federal University Oye-Ekiti. It is an original work for Bachelor Degree in Materials and Metallurgical Engineering and has not been submitted elsewhere for any other degree.

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DEDICATION

This project is dedicated to Almighty God and my parents, Elder and Deaconess J.O. Olaleye.

ACKNOWLEDGEMENTS

My profound gratitude is expressed to the Almighty God for giving me life, knowledge and resources to complete this study.

I express my deep unalloyed appreciation to my project supervisor, Engr. Reginald Umunakwe; the Head of Department, Materials and Metallurgical Engineering, Prof. Abayomi Oni; the non-teaching staffs and all the lecturers in Materials and Metallurgical Engineering Department Federal University Oye-Ekiti, for scholarly criticisms, meticulous teachings and sagacious advice throughout the study.

The fervent support of my parents, Elder and Deaconess Olaleye; brothers and sisters, Olaleye Tope, Olaleye Seun, Olaleye Damilola and Olaleye Tejumola; my uncles and aunts especially Chief Mrs. Fabunmi and Mrs. Ope Subulade; my cousins especially Mrs. Ogundipe, Mr. and Mrs. Lekan Fabunmi, Fabunmi Sanmi; my brother-in-law, Mr. Tope Ogundipe; my spiritual fathers and mothers, Mr. Aduragbemi, Mrs. Adebayo, Mr. Innocent and Mr. Adetunji and my sister's friends especially Ogunsuyi Tomilola is highly appreciated.

Thanks to the management of the following establishments; Federal University of Technology, Akure; Federal Polytechnic Ado-Ekiti; Engineering Materials Development Institute, Akure and University of Lagos, Lagos. The technical staff of the establishments listed above provided the professional advice and characterization machines that helped me to finish this research.

To my friends, course mates and colleagues whom I had good stay with during the course of this study; Akeju Oluwagbemi Oluwaseun, Aina Jonathan, Akeju Oluwagbogo, Olojo-Kosoko Adebowale, Aduloju Oluwatobi, Olugbemi Mayowa, Bomodeoku Kolawole, Josu Gigonu Michael and Adediji Omoniyi. Thanks for being the best. I appreciate you. God bless and keep you all.

ABSTRACT

This study is about the production and characterization of aluminium alloy-Particulate periwinkle shell composite. Composites were produced at 1%, 5%, 10% and 15% weight fraction of Particulate periwinkle shell (PPS) fillers and the aluminium alloy was cast neat at 0%PPS which served as the control.

The microstructures of the composites were studied with Optical Microscopy. Mechanical properties of the composites such as tensile properties (tensile strength, Young's modulus, energy at break and percentage elongation at fracture) and hardness were experimentally determined in the Engineering Materials Development Institute (EMDI) using Instron Universal Testing machine, optical microscopy and microhardness tester. The Optical microscopy analysis showed that interfacial interactions existed between the PPS and the aluminium alloy matrix. Results of the mechanical property tests showed increase in tensile strength, Young's modulus and hardness at low particle sizes while at high particle sizes, the results of the mechanical property test showed decrease in the mechanical properties due to poor wettability of the filler by the matrix, low surface area and high porosity at high particle sizes.

The increase in mechanical properties at small particle sizes is as a result of good wettability of the filler by the matrix, high surface area of the filler and less porosity. It was deduced from the study that aluminium alloy-Particulate periwinkle shell composite is suitable for engineering applications such as aeronautical and automobile applications due to their low weight to strength ratio. Also, it could be used in engineering structures, marine application, automotive bumpers and sporting goods due to the high strength and hardness.

Coating of the surface of PPS with big particle sizes with coupling agents could be done to improve interfacial adhesion and the particle size of PPS could be reduced to a nano particle to improve the mechanical properties and interfacial adhesion.

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CHAPTER ONE

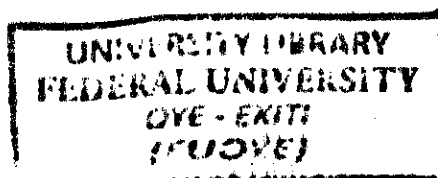
INTRODUCTION

1.1 Background to Study

Developing new engineering materials with light weight, low cost and good mechanical properties is the challenge engineers face most times since no material in nature possesses all these properties. Various approaches are used by researchers, engineers and scientists to develop these new materials from the existing materials in order to meet various service requirements. Some of the methods used to modify the properties of engineering materials are heat treatments, alloying and production of composites.

Composite materials are engineering materials produced by merging two or more materials having different compositional variations and depicting properties from those of the individual materials of the composite (Rajput, 2013). Examples of composite materials are vehicle tyres, cement concrete, plywood etc. According to Rajput (2013), the strength, heat resistance or stiffness of composite material is generally better than any of the individual components. Some of the limitations of composite materials are high cost of raw materials and fabrication, difficulties in reusing and disposing composites and difficulties in combining the reinforcement and matrix for formation of composite materials.

From the last few years, specific strength, weight and cost have become the important parameters in material selection in much industrial applications (Vengatesh and Chandramohan, 2014). Material engineers are facing the problem of developing materials that have low cost, light weight and enhanced mechanical properties for executing construction works. The low weight and excellent thermal conductivity properties of aluminium alloys have made them



preferred engineering material for automobile, aerospace, mineral processing industries and various high performing components that are being used for varieties of applications (Saravanan, 2015). Aluminium is relatively soft and weak so there is need for cold working, alloying, heat treating or production of composite.

Recent studies focus on the development of aluminium metal matrix composites. The applications of aluminium metal matrix composites include drive shafts, fan blades, and shrouds, springs, bumpers, interior panels, tires, brake shoes, clutch plates (Elmasry *et al.*; 2009).

Some of the materials that have been used to reinforce aluminium in Aluminium metal matrix composite are corn cob ash, silicon carbide, zircon, boron carbide, fly ash, copper and austenitic stainless steel fibers (Vijaya *et al.*; 2014). According to Vijaya *et al.* (2014), the most commonly used reinforcements for aluminium in aluminium matrix composite is silicon carbide (SiC) and aluminium oxide (Al_2O_3). Silicon Carbide increases the tensile strength, hardness, density and wear resistance of aluminium and its alloys. Periwinkle shells can be used to reinforce aluminium due to the fact that periwinkle shell is relatively cheap, very strong, hard and less dense than aluminium.

Survey of literatures shows that periwinkle shells have not been used as reinforcement in aluminium. Recent researches show that periwinkle shell has been used as partial replacement for fine aggregates in concrete (Otunyo *et al.*; 2014). Also, Obidiegwu *et al.* (2014) carried out a research on the influence of periwinkle shell addition on mechanical properties of gray cast iron. This survey makes periwinkle shell attractive as reinforcement in aluminium matrix composite.

This work therefore, represents a new design of composite materials containing aluminum as a matrix and particulate periwinkle shell (PPS) as the reinforcement.

1.2 Statement of the Problem

Most developing countries like Nigeria are not yet properly utilizing wastes for gainful engineering production. Periwinkle shells are wastes that constitute environmental pollution and sight disorder in areas where they are dumped. Therefore, it is necessary to develop various means in which the amount of waste in the environment can be reduced. The use of periwinkle shells as reinforcement in aluminium matrix composite is one of the ways of reducing environmental pollution. Microstructural study of the aluminium reinforced with PPS is fundamental in understanding mechanical behavior of aluminium matrix composites.

1.3 Objectives of the Study

The general objective of this project is to conduct microstructural analysis, physical and mechanical tests on the aluminium 6063 and the aluminium- PPS metal matrix composite using optical microscope, universal testing machine and microhardness tester.

Specific Objectives

1. To study the effect of variation of PPS weight fractions on the microstructure, density and some mechanical properties of the aluminium reinforced with PPS
2. To examine the effect of variation of the particle size of the PPS on density and mechanical properties and microstructure of aluminium- PPS metal matrix composite and ascertain the suitability of the composite for engineering applications.

1.4 Justification of the Study

This study is valuable in understanding the potentials of periwinkle shell as reinforcement in aluminium metal matrix composite. The study is useful to scientists, researchers and engineers

because it will help to suggest ways of improving the physical and mechanical properties of the aluminium alloys which will increase the areas of applications of aluminium alloys. In Nigeria where youths are confronted by unemployment, composite production can offer employment opportunity to these unemployed youths due to low energy and machinery requirements for production. In order to describe the behaviours of composites and the effect of forces on the composites, it is vital to have the knowledge of microstructure and mechanical properties of particles filled composites. Proper understanding of the microstructure and mechanical properties of composites will help to ascertain the engineering application of composite in structures, industries, electronics, oil and gas, and other industrial production.

1.5 Scope and Limitation of the Study

Experimental approaches used in this study involve composite production, microstructural analysis using optical microscope, density using Archimedes' principle and determination of the mechanical properties of the composite materials. The composites were produced at 1%, 5%, 10% and 15% weight fractions of PPS filler at the particle sizes of 75 μ m and 150 μ m. The periwinkle shells were washed, dried and milled before they were used to reinforce the aluminium 6063 alloy. The production of the composites was done through die casting. Afterwards, optical microscope was used to view the particle and matrix interfacial interaction. The problems faced in the research were milling of the periwinkle shells to particles and handling of particulate periwinkle dust.

1.6 Limitations

Due to the time and cost of research, this research will be restricted to tensile testing and hardness testing, density measurement and metallographic using optical microscopy. The result

of the tensile test will include the ultimate tensile strength, percentage elongation, elastic modulus, breaking strength and the energy at break of the composite.

1.7 Definition of Terms

Composites

Composite materials are engineering materials produced from merging two or more materials having different compositional variations and depicting properties from those of the individual materials of the composite (Rajput, 2013). Examples of composite materials are vehicle tyres, cement concrete, plywood etc.

Metal Matrix Composites

Metal matrix composites are composites that have ductile metal as its matrix. Metals that are reinforced with hard ceramic particles or fibres have improved strength and stiffness, improved creep and fatigue resistance and increased hardness, wear and abrasion resistance (Harris, 1999).

Aluminium Metal Matrix Composite

Aluminium Matrix Composite is developed by reinforcing aluminium alloy with fillers such as ceramic particles and the fillers provide aluminium matrix composite with better mechanical properties than unreinforced aluminium alloys (Babalola *et al*; 2014).

Periwinkle Shell

Periwinkles are small marine snail which is housed in a v-shaped spiral shell and is found in many coastal communities in Nigeria. Periwinkle shells are the shells of periwinkles that are discarded as wastes after the consumption of periwinkles (Abdullahi and Sylvester, 2015).

Ultimate Tensile Strength (MPa)

Tensile strength or ultimate strength is the maximum amount of tensile stress that a material can absorb before breaking (Askeland, 1994).

Hardness

Hardness can be defined as the resistance of a material to localized plastic deformation such as indentation; it indicates wear resistance and resistance to scratching, abrasion and indentation. (Askeland, 1994).

Metallography

Metallography is the scientific discipline of examining and determining the constitution and the underlying structure of (or spatial relationships between) the constituents in metals, alloys and materials (sometimes called materialography).

CHAPTER TWO

LITERATURE REVIEW

2.1 Overview of Composite

Composite materials are engineering materials produced by merging two or more materials having different compositional variations and depicting properties from those of the individual materials of the composite (Rajput, 2013). Examples of composite materials are vehicle tyres, cement concrete, plywood etc. Matrix is the base material surrounding other materials and has the highest percentage in the composite material. Other materials which reinforce the properties of the base materials are called reinforcements (Rajput, 2013). The reinforcements provide strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement.

According to Trishul *et al.* (2015), the use of composites started many centuries ago. In the Christian Bible, the Book of Exodus recorded that straws were used to produce rigidity and strength in mud walls. Historical examples include the use of bamboos as a reinforcing material in mud walls in houses by Egyptians (1500BC) and laminated metals in the forging of swords (1800AD). In the 20th century, modern composites were used in the 1930's, where glass fibres reinforced resins. Boats and aircrafts were built out of these glass composites commonly called fibre glass (Trishul *et al.*; 2015).

Since the 1970's the applications of composite materials have widely increased due to development of new fibres such as carbon, boron and aramids and new composite systems with matrices made of metal and ceramics.

2.1.1 Classification of composite

Composite can be classified based on the type of matrix used and also based on the reinforcement.

(a) Classification of composites based on the type of material used for the matrix

- i. Metal matrix composite (MMCs)
- ii. Polymer matrix composite (PMCs)
- iii. Ceramic matrix composite (CMCs)
- iv. Carbon matrix composite

(i) Metal matrix composite

Metal matrix composites are composites that have ductile metal as matrix. Metals that are reinforced with hard ceramic particles or fibres have improved strength and stiffness, improved creep and fatigue resistance and increased hardness, wear and abrasion resistance (Harris, 1999). Also, these metals may be utilized at higher service temperatures than unreinforced metals or competing reinforced plastics (Callister, 2007). According to Harris (1999), these properties offer potential for development of materials that can be used in a range of pump and engine applications, including compressor bodies, vanes and rotors, piston sleeves and inserts, connecting rods, and so forth. These metal matrix composites have some advantages over the polymer-matrix composites. Some of the advantages include higher operating temperatures, non-flammability, and greater resistance to degradation by organic fluids. Metal-matrix composites are much more expensive than PMCs and this makes their (MMC) use is somehow restricted (Callister, 2007).

In metal matrix composite, super alloys, as well as alloys of aluminum, magnesium, titanium, and copper, are employed as matrix materials while the reinforcement may be in the form of particulates, both continuous and discontinuous fibres, and whiskers; concentrations normally range between 10 and 60 vol% (Callister, 2007). Some of the materials of the continuous fibre are carbon, silicon carbide, boron, aluminum oxide, and the refractory metals. On the other hand, discontinuous reinforcements consist primarily of silicon carbide whiskers, chopped fibres of aluminum oxide and carbon, and particulates of silicon carbide and aluminum oxide.

According to Callister (2007), some matrix–reinforcement combinations are highly reactive at elevated temperatures. Consequently, high-temperature processing or subjecting the MMC to elevated temperatures during service may cause composite degradation. The common way of resolving this is by applying a protective coating to the reinforcement or by modifying the matrix alloy composition.

(ii) Polymer matrix composite

Polymer-matrix composites (*PMCs*) are the composites that have a polymer resin as the matrix with fibre as reinforcement. Polymers make ideal materials as they can be processed easily, possess lightweight, and desirable mechanical properties (Callister, 2007). There are two main kinds of polymers and they are thermosets and thermoplastics. Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on heating. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering Thermosets very flexible. Thus, they are most

suited as matrix bases for advanced conditions fibre reinforced composites. Thermosets find wide ranging applications in the chopped fibre composites form particularly when a premixed or moulding compound with fibres of specific quality and aspect ratio happens to be starting material as in epoxy, polymer and phenolic polyamide resins

Examples of polymer-matrix composites are:

a) Glass fibre reinforced polymer (GFRP) composites: optic fibre

Fibreglass reinforce polymer composite is simply a composite consisting of glass fibres, either continuous or discontinuous, contained within a polymer matrix; this type of composite is produced in the largest quantities (Callister, 2007).

According to Callister (2007), glass fibre is popular as reinforcement because:

- i. It is easily drawn into high-strength fibres from the molten state.
- ii. It is readily available and may be fabricated into a glass-reinforced plastic economically using a wide variety of composite-manufacturing techniques.
- iii. As a fibre it is relatively strong, and when embedded in a plastic matrix, it produces a composite having a very high specific strength.
- iv. When coupled with the various plastics, it possesses a chemical inertness that renders the composite useful in a variety of corrosive environments.

The limitation of (GFRP) is that they are not stiff and they do not display the rigidity that is necessary for some applications (e.g., as structural members for airplanes and bridges). Most fibreglass materials are limited to service temperatures below 200⁰C. At higher temperatures; most polymers begin to flow or to deteriorate. Service temperatures may be extended to approximately by using high-purity fused silica for the fibres and high-temperature polymers such as the polyimide resins (Callister, 2007).

According to Callister (2007), the applications of fibreglass are in automotive and marine bodies, plastic pipes, storage containers, and industrial floorings.

b) Carbon fibre-reinforced polymer (CFRP) composites

Carbon is a high-performance fibre material that is the most commonly used reinforcement in advanced (i.e., non fibreglass) polymer-matrix composites.

The reasons for this are as follows:

- i. Carbon fibres have the highest specific modulus and specific strength of all reinforcing fibre materials.
- ii. They retain their high tensile modulus and high strength at elevated temperatures; high temperature oxidation, however, may be a problem.
- iii. At room temperature, carbon fibres are not affected by moisture or a wide variety of solvents, acids, and bases.
- iv. These fibres exhibit a diversity of physical and mechanical characteristics, allowing composites incorporating these fibres to have specific engineered properties.
- v. Fibre and composite manufacturing processes have been developed that are relatively inexpensive and cost effective.

Carbon-reinforced polymer composites are now being used extensively in sports and recreational equipment (fishing rods, golf clubs), filament-wound rocket motor cases, pressure vessels, and aircraft structural components—both military and commercial, fixed wing and helicopters (e.g., as wing, body, stabilizer, and rudder components) (Callister, 2007).

c) Aramid fibre-reinforced polymer composites.

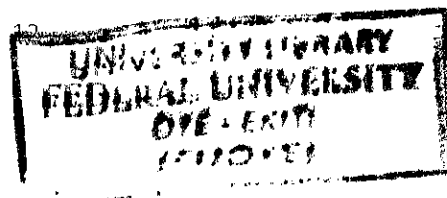
Aramid fibres are high-strength, high-modulus materials that were introduced in the early 1970s. According to Callister (2007), aramid fibres are desirable for their outstanding strength to weight ratios, which are superior to metals.

The aramid fibres are most often used in composites having polymer matrices; common matrix materials are the epoxies and polyesters. Since the fibres are relatively flexible and somewhat ductile, they may be processed by most common textile operations. Typical applications of these aramid composites are in ballistic products (bulletproof vests and armor), sporting goods, tires, ropes, missile cases, pressure vessels, and as a replacement for asbestos in automotive brake and clutch linings, and gaskets.

(iii) Ceramic matrix composites (CMCs)

CMCs have ceramics as their matrix. Ceramics can be described as solid materials which exhibit very strong ionic bonding in general and in few cases covalent bonding. High melting points, good corrosion resistance, stability at elevated temperatures and high compressive strength, render ceramic-based matrix materials a favourite for applications requiring a structural material that does not give way at temperatures above 1500⁰C. Naturally, ceramic matrices are the obvious choice for high temperature applications.

Ceramic-matrix composites may be fabricated using hot pressing, hot isostatic pressing, and liquid phase sintering techniques. Relative to applications, SiC whisker-reinforced aluminas are being utilized as cutting tool inserts for machining hard metal alloys; tool lives for these materials are greater than for cemented carbides (Callister, 2007).



(iv) Carbon matrix composite

The carbon fibre reinforced carbon-matrix composite is one of the most advanced and promising engineering material. It is often termed a carbon–carbon composite; as the name implies, both reinforcement and matrix are carbon. These materials are relatively new and expensive and, therefore, are not currently being utilized extensively. Their desirable properties include high-tensile moduli and tensile strengths that are retained to temperatures in excess of resistance to creep, and relatively large fracture toughness values. Furthermore, carbon–carbon composites have low coefficients of thermal expansion and relatively high thermal conductivities; these characteristics, coupled with high strengths, give rise to a relatively low susceptibility to thermal shock. Their major drawback is a propensity to high temperature oxidation (Callister, 2007).

According to Callister (2007), the carbon–carbon composites are employed in rocket motors, as friction materials in aircraft and high-performance automobiles, for hot-pressing molds, in components for advanced turbine engines, and as ablative shields for re-entry vehicles.

(b) Classification of composites based on the type reinforcement

i. Particulate reinforced composites

A particulate reinforced composite is a composite whose reinforcement is a particle with all the dimensions roughly equal. According to Richardson (1987), particulate fillers are employed to improve high temperature performance, reduce friction, increase wear resistance and to reduce shrinkage. The particles will also share the load with the matrix, but to a lesser extent than a fibre. A particulate reinforcement will therefore improve stiffness but will not generally strengthen.

ii. Fibre reinforced composites

A fibre reinforced composite is a composite that contains reinforcement having lengths higher than cross sectional dimension. Fibrous reinforcement represents physical rather than a chemical means of changing a material to suit various engineering applications (Warner *et al*; 1995). In fibre reinforced composite, the reinforcing fibre in a single layer composite may be short or long based on its overall dimensions. Composites with long fibres are called continuous fibre reinforcement and composite in which short or staple fibres are embedded in the matrix are called discontinuous fibre reinforcement (short fibre composites). In continuous fibre composites, fibres are oriented in one direction to produce enhanced strength properties. In short fibre composites, the length of short fibre is neither too high to allow individual fibres to entangle with each other nor too small for the fibres to loss their fibrous nature. The reinforcement is uniform in the case of composites containing well dispersed short fibres. There is a clear distinction between the behavior of short and long fibre composites.

iii. Hybrid composites

Hybrid composites are composites incorporated with two or more different types of fillers especially fibres in a single matrix. Hybridisation is commonly used for improving the properties and for lowering the cost of conventional composites. According to Mallick (1988), there are different types of hybrid composites classified according to the way in which the component materials are incorporated. Hybrids are designated as

- i) sandwich type
- ii) interplay

- iii) intraply and
- iv) intimately mixed

In sandwich hybrids, one material is sandwiched between layers of another, whereas in interply, alternate layers of two or more materials are stacked in regular manner. Rows of two or more constituents are arranged in a regular or random manner in intraply hybrids while in intimately mixed type, these constituents are mixed as much as possible so that no concentration of either type is present in the composite material.

iv. Laminates

Laminar composites are composites that are made from the two dimensional sheets or panels that have a preferred high strength (Rajput, 2013). A laminate is fabricated by stacking a number of laminae in the thickness direction. Generally three layers are arranged alternatively for better bonding between reinforcement and the polymer matrix, for example plywood and paper. These laminates can have unidirectional or bi-directional orientation of the fibre reinforcement according to the end use of the composite. A hybrid laminate can also be fabricated by the use of different constituent materials or of the same material with different reinforcing pattern. In most of the applications of laminated composite, man-made fibres are used due to their good combination of physical, mechanical and thermal behavior.

2.2 Metal matrix composite

In the recent years, there has been steady increase in the use of ceramic particle – reinforced metal matrix composite because of their advantages such as isotropic properties and the possibility of secondary processing facilitating fabrication of secondary components (Rao *et*

al; 2010). Metal matrix composites consist of a non-metallic reinforcement incorporated into a metallic matrix which can provide advantageous properties such as improved thermal conductivity, abrasion resistance, creep resistance, dimensional stability, exceptionally good stiffness-to-weight and strength to weight ratios over base metal alloys (Babalola *et al;* 2014).

According to Bobic *et al.* (2010), metal matrix composites are now replacing conventional materials in household appliances, computers, audio and video equipment, as well as in sport appliances.

2.2.1 Aluminium metal matrix composite

Aluminium Matrix Composite is developed by reinforcing aluminium matrix with ceramic particles and this provides aluminium matrix composite with better mechanical properties than unreinforced aluminium alloys (Babalola *et al;* 2014). The reinforcements used in metal matrix composite should be stable in the given working temperature and non-reactive too (Ramnath *et al;* 2013).

According to Saravanan *et al.* (2015), the ceramic reinforcements used in aluminum matrix composites (AMCs) are generally oxides, carbides or borides such as Al_2O_3 , TiB_2 , TiO_2 , SiC , TiC , B_4C , etc. The microstructure, physical properties, tribological properties and other desirable properties of the composite are influenced by the processes route of manufacturing, shape, size, and chemical affinity of reinforcement materials with matrix material. The formation of strong chemical bonds at the interface is favourable for the wetting of reinforcement by molten metal, which is considered as an important aspect in metal matrix composite synthesis. The lower wettability adversely affects the properties of the composite (Saravanan *et al;* 2015). The main cause of non-wetting of the reinforcement with the molten metal is primarily caused by

the presence of oxide films on the surface of molten metal and the adsorbed contaminant on the reinforcement. Metallic coatings on the reinforcements, addition of reactive elements, such as magnesium, calcium or titanium, to the molten metal and heat treatment of particles before addition are some of the techniques to improve metal–reinforcement wettability (Gowri and Jayashree, 2013).

Most of the commercial work on metal matrix composites has focused on aluminum as the matrix metal because of the combination of its light weight, environmental resistance, and favourable mechanical properties (Davis, 1990).

2.2.2 Application of aluminium metal matrix composite

There is abundance of literature concerned with the processing, material characterization, properties and manufacturing of metal matrix composites because recently, there is increase in the use of metal matrix composites in aerospace, automotive and bio-medical industries (Dandekar and Shin, 2011). Though many desirable mechanical properties are generally obtained with fibre reinforcement, these composites exhibit anisotropic behaviour and are not easily producible by conventional techniques. Nevertheless, MMCs reinforced with particles tend to offer modest enhancement of properties, but are more isotropic and can be processed by conventional routes (Mahadevan *et al.*; 2005).

Various applications of aluminium metal matrix composites

1. Aluminum composites and alloys are used for many aeronautical and automobile applications due to their low weight to strength ratio (Nikhil, 2015).
2. Aluminium matrix composite materials are widely used engineering structures, marine application, automotive bumpers and sporting goods (Devi, 2012).

3. Aluminium matrix composites are superior in nature for elevated temperature application when reinforced with ceramic particle (Saravanan *et al*; 2015).
4. Silicon Carbide reinforced Al MMCs are suitable materials for brake drums as they have high wear resistance but cannot be used in brake linings as it will damage the brake drum (Ramnath *et al*; 2013).

2.2.3 Aluminium alloys used for aluminium metal matrix composite with different reinforcement materials

Foo *et al.* (1994), presented a paper on electron microscope examination of the interface of silicon carbide particulate reinforced 6061 aluminium composite produced by a powder metallurgy process is presented. Energy dispersive X-ray spectroscopy (EDX) and electron energy loss spectroscopy (EELS) were used to identify the intermetallic compounds such as Mg_2Si and $FeSiAl_5$, some of which are found at the silicon carbide/matrix interface. Initial results suggest that these intermetallic particles have a detrimental effect as they cause early debonding at the interface.

Composite aluminium alloys (AA 2024 and AA 6061) reinforced with aluminium oxide particles (Al_2O_3p) have been produced by squeeze casting followed by hot extrusion and a precipitation hardening treatment (Babalola *et al*; 2014). Senthilkumar and Omprakash, (2011), reinforced aluminium alloy matrix with varying percentages of titanium carbide particle (TiC) using powder metallurgy route. Pathak *et al*; 2006, reinforced hypereutectic aluminium-silicon alloy-based composite with silicon carbide particles using centrifugal casting method. Chaudhury *et al*; 2004, used the convention vortex method to prepare Al-2Mg-11TiO₂ composite and magnesium was added to improve the wettability of the dispersoid (TiO₂).

A356-CNT composites have been produced by a special *compocasting* method using A356 aluminium alloy as the matrix and multi-walled carbon nano-tubes (MWCNTs) as the reinforcement. This was reported by (Abbasipour *et al*; 2010).

2.2.4 Limitation of Aluminium Metal Matrix Composite

Limitation of aluminium based composite may be contradictory due to the fact that a variety of aluminium alloy matrices and reinforcement type combinations may exhibit completely different behaviour. However;

1. aluminium metal matrix composite reinforced with graphite, silicon carbide, alumina, boron and mica in the form of particles, fibres, whiskers or monofilaments are more susceptible to corrosion attack than the matrix alloy (Bobić *et al*; 2010).

2.2.5 Some Recent Works on Aluminium Metal Matrix Composite

Ravichandran and Dineshkumar, (2014) carried out a research work by fabricating aluminium metal matrix composites through liquid powder metallurgy route. The aluminium matrix composite containing TiO₂ reinforcement particle was produced to study the mechanical properties such as tensile strength and hardness. The characterization studies are also carried out to show the phases present in the composite and the results are discussed for the reinforcement addition with the mechanical properties. Results show that, the addition of 5 weight percentage of TiO₂ to the pure aluminium improves the mechanical properties (Ravichandran and Dineshkumar, 2014).

Jenix Rina *et al*. (2013) compared the properties of Al6063 MMC reinforced with Zircon Sand and Alumina with four different volume fractions of Zircon sand and Alumina with varying volume fractions of (0+8)%, (2+6)%, (4+4)%, (6+2)% and (8+0)%. The hardness and the tensile

strength of the composites are higher for (4+4)%. In this combination, the particle dispersion is uniform and the pores are less where inter-metallic particles are formed.

According to Vijaya *et al.* (2013), the trimodal aluminium metal matrix composites and the factors affecting its strength had been investigated. The test result shows that the attributes like nano-scale dispersoids of Al_2O_3 , crystalline and amorphous AlN and Al_4C_3 , high dislocation densities in both NC Al and CG-Al domains, interfaces between different constituents, and nitrogen concentration and distribution leads to increase in strength.

Shi *et al.* (2011) studied the morphology and interfacial characteristics of aluminium matrix composites reinforced with the diamond fiber. The composite exhibit high thermal conductivity and low thermal expansion coefficient. Pressure-less metal infiltration process results in good bonding between the diamond fibers and the aluminium-matrix.

2.3 Periwinkle shell

Periwinkle shell is a waste product got from the consumption of small marine snail (periwinkle) which is housed in a v-shaped spiral shell and is found in many coastal communities in Nigeria (Abdullahi and Sylvester, 2015). It is also available in many coastal areas worldwide and is very strong, hard and brittle material. Stretching from the Niger Delta between Calabar in the East and Badagry in the Western part of Nigeria, the people in these areas take the edible part as sea food and dispose of the shell as waste product. One of the ways to dispose the waste would be utilization of some of it into constructive building materials as this would help to prevent the depletion of natural resources such as granite and gravel and to maintain ecological balance (Ohimain, *et al.*; 2009). There are however, large amount of these shells being disposed

of as waste thus constituting environmental problems in places where use cannot be found for them.

2.3.1 Some uses of periwinkle shell

Though periwinkle shell is a waste product, there are still some places where periwinkle shells can still be used. According to Otunyo *et al.* (2014), periwinkle shells have been used by the people of the coastal states. For instance, in Rivers State of Nigeria for over 30 years it has been used as conglomerate in concrete reinforcement.

Otunyo *et al.*; 2014, also carried out a study on the properties of periwinkle-granite concrete. It was discovered that periwinkle shells can be used as a partial replacement for granite in normal construction works and that the strength development in periwinkle granite concrete is similar to those of conventional granite concrete.

2.3.2 Recent work with periwinkle shell in metallurgy

Ofen *et al.*; 2012 made studied on the effect of filler content on the mechanical properties of periwinkle shell reinforced CNSL (cashew nut shell liquid) resin composites with particle sizes (400, 600, 800 μ m) and filler loading (10, 20, 30, and 40 wt%) and reported that 30% Filler content gave the best properties, while the highest tensile and flexural strengths were recorded at 30% filler content and 400 μ m particle sizes, the highest tensile modulus and impact strength were recorded at 800 μ m particle sizes but 30% and 40% filler content respectively. At 40% filler content the properties tends to decrease indicating that the optimum properties can be achieved at 30% filler content.

Ofem *et al*; 2012 later investigated the mechanical properties of the hybrid composite made of periwinkle shells (PWS) and rice husk (RH) as reinforcing fillers using cashew nut shell liquid (CNSL) resin composite as the matrix. With PWS particle sizes (400, 600, 800 μ m) and filler loading(10, 20, 30 wt%), the results shows that the highest tensile and flexural strengths were obtained at 30% filler content and 400 μ m particle sizes, and the highest tensile modulus and impact strength were obtained at 800 μ m and 400 μ m particle sizes, respectively, for the same percentage of filler content. The flexural strength from the result converges at 30% filler content. It can be concluded that the optimum properties can be achieved at 30% filler content.

Olutoge *et al*; 2012 studied the suitability of periwinkle shell ash as partial replacement for ordinary Portland cement in concrete, analyzed the chemical content of periwinkle shell ash and the following constituents was gotten with their weight percentage above; ZnO, CuO, Fe₂O₃, MnO₂, MgO, SiO₂, Al₂O₃, K₂O, CaO, Na₂O.

Aku *et al*; 2012, conducted an XRF analysis on periwinkle shell ash and confirmed that SiO₂, CaO, MgO, Cr₂O₃, and Fe₂O₃ were found to be major constituents of the periwinkle ash. Silicon dioxide, iron oxide, Cr₂O₃ and CaO are known to be among the hardest substances. Some other oxides like K₂O, Na₂O, and MnO were also found to be present in traces. The presence of hard elements like SiO₂, CaO, Cr₂O₃, and Fe₂O₃ suggested that, the periwinkle shell particles can be used as reinforcing material.

2.4 Methods of Production of MMCs

2.4.1 Processing of AMC

A key challenge in the processing of composites is to homogeneously distribute the reinforcement phases to achieve a defect-free microstructure. The reinforcing phases in the composite can be either particles or fibers depending on the shape. Particulate-reinforced composite is preferred to the fiber-reinforced composite due to the low material cost and suitability for automatic processing. Primary processes for manufacturing of AMCs at industrial scale can be classified into two main groups.

a. Liquid State Processes:

According to Rajeshkumar and Parshuram (2013), liquid state processes include stir casting, compo casting and squeeze casting spray casting and in situ (reactive) processing, ultrasonic assisted casting.

Among the variety of manufacturing processes available for discontinuous metal matrix composites, stir casting is generally accepted as a particularly promising route, currently practiced commercially. Its advantages lie in its simplicity, flexibility and applicability to large quantity production. It is also attractive because, in principle, it allows a conventional metal processing route to be used, and hence minimizes the final cost of the product. This liquid metallurgy technique is the most economical of all the available routes for metal matrix composite production and allows very large sized components to be fabricated.

i. *Stir Casting:*

In a stir casting process, the reinforcing phases are distributed into molten matrix by mechanical stirring. Stir casting of metal matrix composites was initiated in 1968, when S. Ray introduced alumina particles into an aluminium melt by stirring molten aluminum alloys containing the ceramic powders. Mechanical stirring in the furnace is a key element of this process. The resultant molten alloy, with ceramic particles, can then be used for die casting, permanent mold casting, or sand casting. Stir casting is suitable for manufacturing composites with up to 30% volume fractions of reinforcement (Rajeshkumar and Parshuram, 2013).

The cast composites are sometimes further extruded to reduce porosity, refine the microstructure, and homogenize the distribution of the reinforcement. A major concern associated with the stir casting process is the segregation of reinforcing particles which is caused by the surfacing or settling of the reinforcement particles during the melting and casting processes. The final distribution of the particles in the solid depends on material properties and process parameters such as the wetting condition of the particles with the melt, strength of mixing, relative density, and rate of solidification. The distribution of the particles in the molten matrix depends on the geometry of the mechanical stirrer, stirring parameters, placement of the mechanical stirrer in the melt, melting temperature, and the characteristics of the particles added (Rajeshkumar and Parshuram, 2013).

An interesting recent development in stir casting is a two-step mixing process. In this process, the matrix material is heated to above its liquids temperature so that the metal is totally melted. The melt is then cooled down to a temperature between the liquids and solidus points and kept in a semi-solid state. At this stage, the preheated particles are added and mixed. The

slurry is again heated to a fully liquid state and mixed thoroughly. This two-step mixing process has been used in the fabrication of aluminum. Among all the well-established metal matrix composite fabrication methods, stir casting is the most economical. For that reason, stir casting is currently the most popular commercial method of producing aluminum based composites. Obtaining homogeneous distribution during processing of composites to the reinforcement phases to achieve a defect-free microstructure

b. Solid State Processes:

Solid state process include Powder blending followed by consolidation (PM processing), high energy ball milling, friction Stir Process, diffusion bonding and vapors deposition techniques. The selection of the processing route depends on many factors including type and level of reinforcement loading and the degree of micro structural integrity desired (Rajeshkumar and Parshuram, 2013).

2.5 Mechanical Properties

2.5.1 Hardness

Hardness can be defined as the resistance of a material to localized plastic deformation; it indicates wear resistance and resistance to scratching, abrasion and indentation (Askeland, 1994). Hardness testing can be done with Rockwell, Vickers, Brinell, durometer, rebound and barcol hardness tester.

2.5.2 Tensile Properties

According to Liu (1999), tensile properties indicate how materials will react to forces applied on tension. Tensile properties are determined by performing a tensile test. Tensile test is

a simple uniaxial test that consists of slowly pulling a sample of material in tension until it breaks. These properties can be found from a tensile test: modulus of elasticity, elongation at break, tensile strength, tensile stress and tensile strain.

2.5.2.1 Stress (MPa)

Tensile stress of the material is defined as the force per unit area as the material is stretched (Liu, 1999). The area used in finding tensile stress is the original under formed cross sectional area because the cross sectional area of a material may change if the material deforms on stretching.

$$\text{Tensile stress } (\delta) = \frac{\text{Load or force}}{\text{Original cross sectional area}} = \frac{P}{A} \quad (2.1)$$

2.5.2.2 Strain

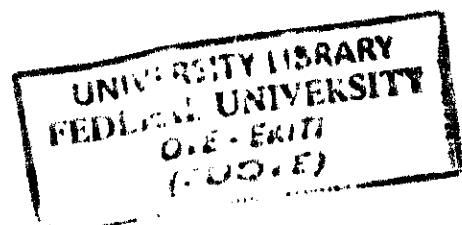
According to Onwuka (2001), strain is the non-dimensional measure of deformation of a material with respect to a given length dimension of that material. Tensile strain or engineering strain is the change in length of a sample of material divided by the original length or gauge length of the sample. Strain can be represented thus,

$$\text{Strain} = \frac{\text{Change in length}}{\text{Original length}} = \frac{\text{Change in length}}{\text{Gauge length}}$$

$$\frac{L_f - L_o}{L_o} = \frac{\Delta L}{L_o} \quad (2.2)$$

Where L_o = Original length

ΔL = Axial deformation or change in length, L_f = final length



2.5.2.3 Ultimate Tensile Strength (MPa)

According to Askeland (1994) Tensile strength or ultimate strength is the maximum amount of tensile stress that a material can absorb before breaking. It is the maximum tensile stress reached on a stress-strain diagram. Tensile strength of a material is affected by the preparation of the test specimen, the presence of surface defects (voids, porosity and inclusions), the temperature of the test environment and the nature of the material.

$$\text{Ultimate tensile strength} = \frac{\text{Maximum Tensile Force Applied}}{\text{Original cross sectional area}} \quad (2.3)$$

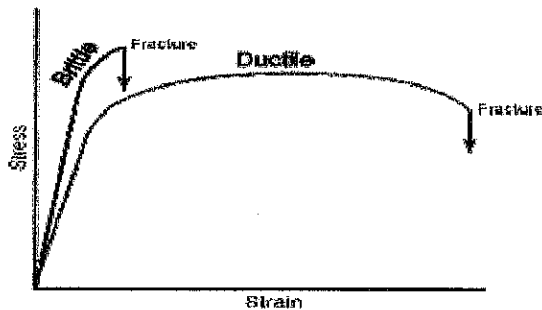


Figure 2.1: Stress – Strain curve

2.5.2.4 Young's Modulus (MPa)

Young's modulus or modulus of elasticity (E) is the slope of the stress - strain curve in the elastic region (Askeland, 1994). According to Hooke's law it is a measure of the stiffness of the material. Stiffness is the property of a material to resist deformation in the elastic range or within the proportional limit.

$$\text{Young's modulus (E)} = \frac{\text{Stress}}{\text{Strain}} \quad (2.4)$$

2.5.2.5 Elongation at Fracture (%)

Elongation at fracture is the strain on a sample when it fractures. It is usually expressed in percentage and is a measure of the ductility of the material. Elongation at fracture is the amount of uniaxial strain at fracture and is depicted as strain. Elongation at fracture is mostly calculated by removing fractured specimen from the grips, fitting the broken ends together and measuring the distance between gauge marks.

$$\% \text{ Elongation at fracture} = \frac{L_f - L_o}{L_o} \times 100 \quad \text{or} \quad \frac{\text{Extension}}{\text{Original length}} \times 100 \quad (2.5)$$

L_f = final length of tensile test specimen at rupture. L_o = initial length of test specimen.

2.6 Microstructure

Microstructure is defined as the structure of a prepared surface of thin foil of a material as revealed by a microscope above x25 magnification. According to Slouf (2010) microstructures are structures of dimension 1um to 1000um. The microstructure of a material can be analyzed with the optical microscope, scanning electron microscope, X-ray diffractometer and scanning transmission electron microscope.

According to the report from Lancaster State University handout (2010) microstructure is the microscopic description of the individual constituents of a material. Microstructure is also the study of the crystal structure of a material, its size, chemical composition, phases, defects, orientation and interaction and their effect on the macroscopic behaviour in terms of physical properties and mechanical properties such as strength, toughness, ductility, hardness, corrosion resistance, low and high temperature behaviour, wearability and so on. These properties govern

the application of materials in industry. The basis of materials science involves relating the desired properties to the structure of atoms and phases.

2.6.1 Optical Microscope

In optical microscope, the light microscope is used to study the microstructure; optical and illumination systems are its basic elements. The surface of materials that are opaque to visible light (all metals and many ceramics and polymers), is subject to observation, and the light microscope must be used in a reflecting mode (Callister, 2007). Contrasts in the image produced result from differences in reflectivity of the various regions of the microstructure. Investigations of this type are often termed *metallographic*, since metals were first examined using this technique.

Advantages of optical microscope

1. Direct imaging with no need of sample pre-treatment, the only microscopy for real color imaging.
2. Fast and adaptable to all kinds of sample systems, from gas, to liquid, and to solid sample systems, in any shapes or geometries.
3. Easy to be integrated with digital camera systems for data storage and analysis.

Disadvantages of optical microscope

1. Low resolution, usually down to only sub-micron or a few hundreds of nanometers, mainly due to the light diffraction limit.

2.6.2 Scanning Electron Microscopy

Scanning electron microscope is a microscope that uses electrons instead of light to form an image (Dunlap and Adaskaverg, 2000). Since scanning electron microscope has been

developed in the early 1950's, new areas of study in the medical and physical science communities have been developed. Scanning electron microscope has allowed researchers to examine much bigger variety of specimens because of the large depth of field which allows large particle of the sample to be in focus at one time (Boyde, 1994). The surface of a specimen to be examined is scanned with an electron beam, and the reflected (or back-scattered) beam of electrons is collected, then displayed at the same scanning rate on a cathode ray tube (similar to a CRT television screen) (Callister, 2007). Preparation of sample is easy in scanning electron microscopy and most scanning electron microscopes require the sample to be conductive. These properties have made the scanning electron microscope inevitable in the study of morphology of materials by researchers.

Nowosielski et al. (2006) characterized the microstructure of composite materials with powders of barium ferrite with x-ray diffractometer and scanning electron microscopy. The result of the scanning electron microscopy analysis made it possible to understand that the distribution of powders of barium ferrite in the polymer matrix was irregular and the powder particles had irregular shapes and dimensions.

Castejon (1998) used scanning electron microscope to study the cerebella golgi cells of mouse, telecast fish, primate and human species and the scanning electron microscopy analysis showed three dimensional neuronal geometry and smooth outer surfaces of the organisms. The University of Tennessee equally reported in 2000 that scanning electron microscope can be used in the following areas:

1. Analysis of the surface features of materials,
2. Obtaining the crystallographic information of the materials,

3. Analysis of the microstructural features of materials, that is, grain size, grain shapes, distribution of various phase and defects such as cracks and voids.
4. To determine the chemical composition of materials when used in conjunction with energy dispersive x-ray spectrophotometry.

2.6.2.1 Components of a Scanning Electron Microscope (SEM)

Figure 2.2 below shows the major components of a scanning electron microscope (Dunlap and Adaskaverg, and Adaskareg, 2000).

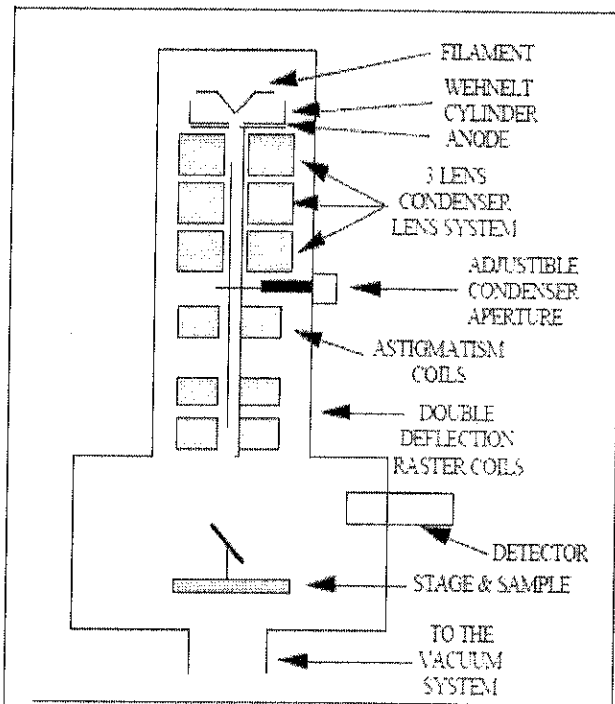


Figure 2.3: SEM column and specimen chamber (Dunlap and Adaskaverg, 2000)

Some of the components of SEM are the vacuum, beam generation, beam manipulation, beam interaction, detection, signal processing, and display and recording units. These systems function together to determine the results and qualities of a micrograph such as magnification,

resolution, depth of field, contrast and brightness. The functions are as follows: vacuum system: This system is required when using an electron beam because electrons will quickly disperse or scatter due to collision with other particles.

Electron beam generation system: This system is found at the top of the microscope column. It generates the illuminating beam of electrons known as primary electrons.

Electron beam manipulation system: This system consist of electromagnetic lenses and coils located in the microscope column and control the size, shape and position of the electron beam on the specimen surface.

Beam specimen interaction system: This system involves the interaction of the electron beam with the specimen and the types of signals that can be detected.

Detection System: This system can consist of several different detectors each sensitive to different energy/particle emission that occurs in the sample.

Signal processing system: This system is an electronic system that processes the signal generated by the detection system and allows additional electronic manipulation of the image.

Display and recording system: This system allows visualization of an electronic signal using cathode ray tube and permits recording of the results using photographic or magnetic media.

2.6.3 X-ray Fluorescence (XRF)

X-ray fluorescence (XRF) is the emission of characteristic “secondary” (or fluorescent) X-rays from a material that has been excited by bombarding with high energy X-rays or gamma rays. The phenomenon is widely for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials and for research in geochemistry,

forensic science and archaeology. XRF can also sometimes be used to determine the thickness and composition of layers and coatings and in most circumstances is non-destructive.

A typical X-ray fluorescence spectrometer is composed of excitation source (X-ray tube) and detection system. When materials are exposed to the radiation from X-ray tube, excitation of their component elements may take place and the material emits radiation which has energy characteristic or wavelength characteristic of the atoms present, the energy and the quantity will be measured by the detection system.

2.6.3.1 Principles of Operation of XRF

X-radiation is a form of electromagnetic radiation. X-rays have a wavelength in the range of 0.01 to 10 nanometres, characteristics of which are defined by energy (unit: keV) and wavelength (unit: nm). When a primary X-ray excitation source from an X-ray tube or a radioactive source strikes a sample, the X-ray can either be absorbed by the atom or scattered through the material. The process in which an X-ray is absorbed by the atom by transferring all of its energy to an innermost electron is called the "photoelectric effect." During this process, if the primary X-ray had sufficient energy, electrons are ejected from the inner shells, creating vacancies. These vacancies present an unstable condition for the atom. As the atom returns to its stable condition, electrons from the outer shells are transferred to the inner shells and in the process gives off a characteristic X-ray whose energy is the difference between the two binding energies of the corresponding shells. Because each element has a unique set of energy levels, each element produces X-rays at a unique set of energies, allowing one to non-destructively measure the elemental composition of a sample. The process of emissions of characteristic X-rays is called "X-ray Fluorescence," or XRF.

2.6.3.2 Features of the X-ray Fluorimeter (XRF)

- i. Large-volume sample chamber able to accept most different sized samples.
- ii. Inbuilt high-resolution camera sample viewing system provides easy sample alignment.
- iii. Vacuuming system for sample chamber enhances detection sensitivity of light elements (Na, Mg, Al, Si, etc)
- iv. Newest version of application software provides all the critical information on one screen. Display element spectrum, in-process results of elemental content, camera image, measurement time, etc
- v. Thermoelectric cooling of UHRD instead of liquid nitrogen refrigeration
- vi. Intelligent full-element software is compatible with hardware, easy to operate.
- vii. Built-in signal to noise enhancer improves the signal processing capability by 25times
- viii. Applicable in RoHS test (Restriction of Hazardous Substances Testing)
- ix. Automatic switch of collimator (a device that narrows a beam of particle or wave) and filter according to different samples

2.6.3.3 Areas of Applications of XRF Analysis

- i. Ecology and environmental management: measurement of heavy metals in soils, sediments, water and aerosols
- ii. Geology and mineralogy: qualitative and quantitative analysis of soils, minerals, rocks etc.
- iii. Metallurgy and chemical industry: quality control of raw materials, production processes and final products
- iv. Paint industry: analysis of lead-based paints
- v. Jewellery: measurement of precious metals concentrations

- vi. Fuel industry: monitoring the amount of contaminants in fuels
- vii. Food chemistry: determination of toxic metals in foodstuffs
- viii. Agriculture: trace metals analysis in soils and agricultural products

CHAPTER THREE

MATERIALS AND METHOD

3.1 Materials and Equipment

The major materials required for this work are aluminium 6063 alloy and PPSs. The aluminium 6063 with chemical composition shown in table 3.1 was purchased from Nigerian Aluminium Extrusion Limited (NIGALEX), 31-37 Apapa Oshodi Expressway, Lagos. The periwinkle shell was obtained as waste from periwinkles and sourced from the local market at Otueke, Bayelsa State, Nigeria. Nitric acid as Etchant and epoxy for mounting of the samples for metallography were supplied by the Engineering Materials Development Institute (EMDI) Akure, Nigeria. Table 3.1 shows the purchased aluminium ingot while plate 3.1 shows the particulate periwinkle shells.

The equipment used in this study are: Metal mould, bench vice, hammer, crucible, gas fired furnace, cupola furnace, ball mill, a set of British Standard sieves, sieve shaker, electronic weighing balance, measuring cylinder, optical microscopy, Intron universal testing machine, microhardness tester, lathe machine, grinding machine, polishing machine, mounting press, X-ray fluorescence (XRF) machine.

Table 3.1: Composition of the aluminium ingot

Element	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr
Average content	98.18	0.5953	0.4635	0.0117	0.0244	0.3459	<0.002	0.0107
Element	Ni	Ti	Sr	Zr	V	Ca	Be	
Average content	0.0347	0.0566	<0.000	0.0772	0.0114	>0.070	<0.000	

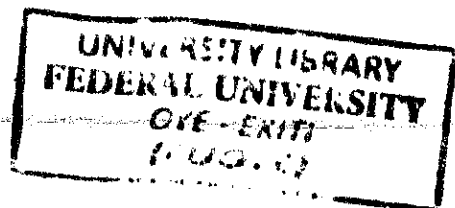




Plate 3.1: Periwinkle shells

3.2 Materials Preparation

3.2.1 Preparation of the Reinforcement

The reinforcement material used in this study is periwinkle shells. The periwinkle shells were boiled in water; soaked for 2 hours in the water and then washed thoroughly so as to remove the impurities like dust and fine sand particles. After washing, the periwinkle shells were sundried for 14 days so as to remove moisture from it. The dried periwinkle shells were reduced to smaller sizes in a metallic mortar and pestle before being taken to the mineral processing laboratory in Federal University of Technology Akure where ball mill was used to reduce the broken periwinkle shell to particles. Plate 3.2 shows the ball mill used for the milling of the periwinkle shells.



Plate 3.2: Ball mill

After milling, a mechanical sieve shaker with a set of sieves was used to sieve the milled periwinkle shells to obtain the required particle sizes of 75 μ m and 150 μ m. Plate 3.3 shows PPS

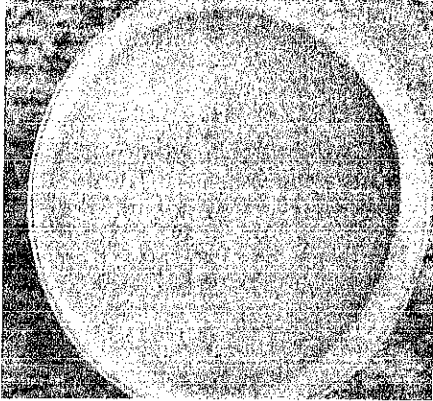


Plate 3.3: Particulate periwinkle shell

3.2.2 Preparation of the Matrix

The matrix material used in this study was melted aluminium 6063 ingot. The purchased aluminium ingot was melted with cupola furnace and cast on sand into small sizes for easy handling.

3.2.3 Preparation of the Composite

The composites were produced using facilities at the foundry workshop of Federal University of Technology, Akure, Ondo State. The matrix materials as prepared in section 3.2.2 were weighed according to mass required for each composition. A 250g of aluminium 6063 alloy was weighed with a digital weighing balance and it was melted at 670 °C with a crucible inside the gas fired lift out crucible furnace and poured inside the metal mould without any filler to produce the control sample. The composite were prepared using 1wt%, 5wt%, 10wt% and 15wt% weight fraction of PPSs in the matrix. Two-step casting technique was used as described by Rajeshkumar and Parshuram (2013) to ensure the dispersion of the filler in the matrix. For

each specimen, the weighed aluminium alloy was melted and heated to 670°C. The weighed filler was poured into the melted metal and stirred with steel rod. The mixture was allowed to cool below the melting temperature of aluminium to be in a semi-solid state (paste). It was then stirred vigorously to enhance the dispersion of the filler in the matrix. Thereafter, the mixture was reheated in the furnace above the melting temperature, stirred, the surface films were removed and the composite was cast using a metal mould. Table 3.2 shows the details of all the samples of composites produced.

Table 3.2: Various specimens of the particulate aluminium-periwinkle shell composite produced

Specimen	Particulate size of reinforcement (μm)	Weight percent of reinforcement (wt%)	Volume fraction of reinforcement	Actual weight of reinforcement (g)	Weight percent of matrix (wt%)	Volume fraction of matrix	Actual weight of matrix (g)
1	Nil (control)	00	00	00	100	1	250.00
2	75	1	0.01	2.50	99	0.99	247.50
3	75	5	0.06	12.50	95	0.94	237.50
4	75	10	0.11	25.00	90	0.89	225.00
5	75	15	0.17	37.50	85	0.83	212.50
6	150	1	0.01	2.50	99	0.99	247.50
7	150	5	0.06	12.50	95	0.94	237.50
8	150	10	0.11	25.00	90	0.89	225.00
9	150	15	0.17	32.50	85	0.83	212.50

3.3 Characterization of Reinforcements and Composite Materials

3.3.1 Determination of the Chemical Composition of Periwinkle Shell

The elemental composition of the milled periwinkle shell was determined with X-ray Fluorescence Spectrometer at the EMDI Akure, Nigeria.

3.4 Density and Porosity of Composite Materials

Density of each specimen was determined using Archimedes principle. In this process, each machined specimen was weighed so as to get the mass. Afterwards, the specimen was immersed in water contained in a measuring cylinder and the displacement of the water inside the measuring cylinder gives the volume of the specimen. The known volume and mass of each specimen was now used to calculate the density.

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} = \frac{m(g)}{v(\text{cm}^3)} \quad (3.1)$$

Also, theoretical density of each composite was calculated based on the rule of mixtures as described by Kumar *et al*; 2011.

The theoretical density is calculated from equation 3.2.

$$\rho_c = V_m\rho_m + V_r\rho_r \quad (3.2)$$

Where ρ_c is the density of the composite,

ρ_m is the density of the matrix

ρ_r is the density of the reinforcement

$$V_m = \text{Volume fraction of the matrix} = \frac{\text{Volume of matrix}}{\text{Volume of matrix} + \text{Volume of reinforcement}}$$

$$V_r = \text{Volume fraction of reinforcement} = \frac{\text{Volume of reinforcement}}{\text{Volume of matrix} + \text{Volume of reinforcement}}$$

The degrees of porosity or void in the composites were calculated from equation (3.3) as explained by Kamaal *et al*; 2015.

$$\text{Void fraction} = \frac{\text{theoretical density } (\rho_t) - \text{experimental density } (\rho_e)}{\text{theoretical density } (\rho_t)} \quad (3.3)$$

3.5 Tensile Testing

Tensile testing of the samples was done at the Strength of Materials Laboratory, EMDI. The machining of the samples to the dimension of the tensile testing machine was done at the Mechanical workshop of the Federal University of Technology, Akure (FUTA). Figure 3.1 below shows the dimensions of the tensile piece.

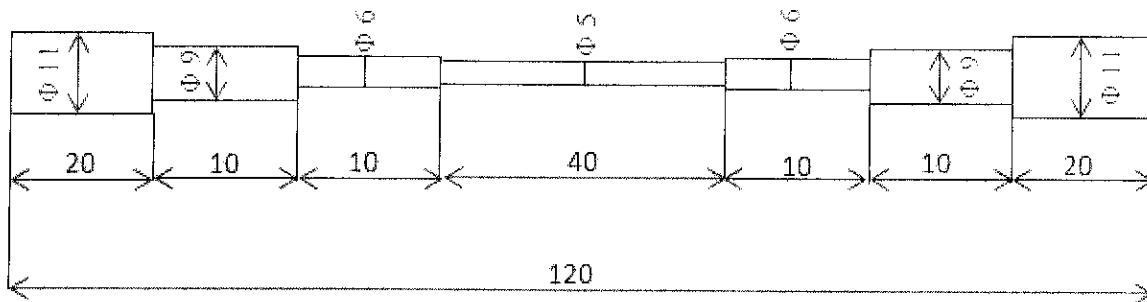


Figure 3.1a: Circular tensile test piece standard 2 step grip with all dimensions in mm.

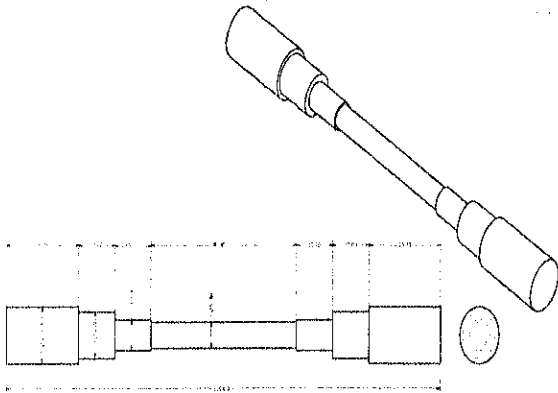


Figure 3.1b: 3D diagram of circular tensile test piece

After machining, the machined samples were taken to Strength of Materials Laboratory, Engineering Materials and Development Institute, Akure, Ondo State where they were subjected to tensile load with the aids of Instron testing machine. The Instron Testing Machine is shown in plate 3.4. The gauge length and diameter of each sample was correctly imputed. Each sample was loaded gradually at a strain rate of 10mm/s. During loading, the sample was stretched with a gradual increase in gauge length but reduction in cross sectional area until the sample breaks after necking. The superimposed stress-strain graph for each sample was automatically plotted by the machine. Other tensile properties like elongation at fracture, tensile strength and young modulus were determined from this stress- strain curve.

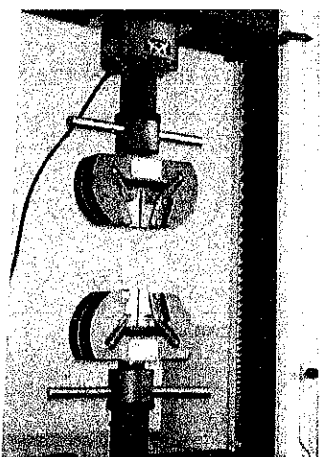


Plate 3.4: Instron Universal Testing Machine at EMDI

3.6 Hardness Test

The hardness test was done at the EMDI in accordance with ASTM E384 (Standard test method for microhardness of materials) using Vickers Micro-hardness Tester of model LM700AT at the applied force of 980mN and dwell time of 10s. Samples for hardness test were machined from the cast of aluminium/PPS composites. Each sample was polished and loaded vertically and indented in a compressive manner by square based pyramid indenter as shown in Plate 3.5 b to measure the hardness values of the control and aluminium/ PPS composites and the results were reported as Vicker hardness numbers. For each specimen, the indentation was done in three places and the average computed to ensure correctness.

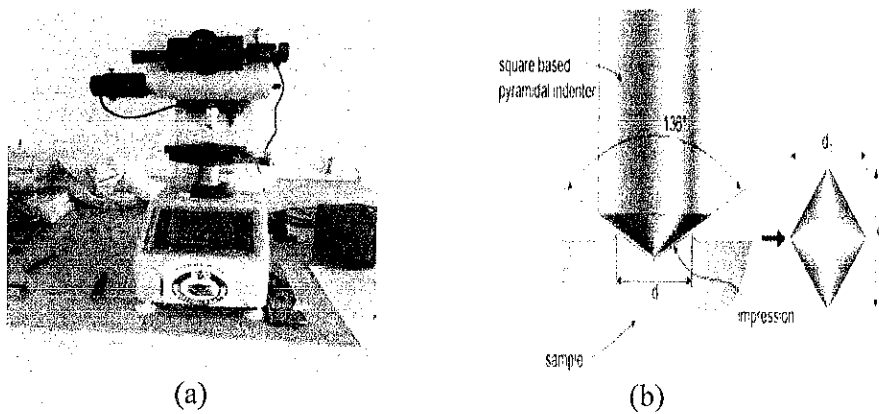


Plate 3.5: Vicker Hardness tester (a) Vicker Hardness Tester in Operation; (b) Vicker's Indentation and Impression Diagonal Measurement (Midwal Materials Testing)

3.7 Metallography

Cubic samples each of side 10mm was cut from casts of aluminium alloys and aluminium alloys/PPS composites of 1, 5, 10 and 15 %weight of PPS particle additions. Each of the cubic samples was mounted using epoxy resin and hardener which form a polymeric compound around the sample for proper handling during grinding and polishing processes. Surface of each

specimen was prepared on emery papers of 220, 320 and 400 grades mounted on the grinding/polishing machine. The ground surface of the sample was cleaned with soap solution washed in distilled water. The washed surface was finished using Selvyet polishing cloth covered with magnesia powder. The finished surface was thoroughly washed and etched in an aqueous solution of 0.5 cm³ nitric acid. The etched surface was dried in boiling ethanol and then examined with the aids of optical microscope at x50, x100 and x200 magnifications.

3.7.1 XRF Analysis

The elemental composition of the PPS was determined using Optical X-ray Fluorimeter machine at Engineering Material Development Institute, Akure.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter presents the results of the research carried out and their discussions.

4.1 Densities and Porosity:

The density of the aluminium 6063 alloy, the theoretical and experimental densities of the aluminium-PPS composites with different particle sizes and weight fractions of reinforcement are shown in the figure 4.1.

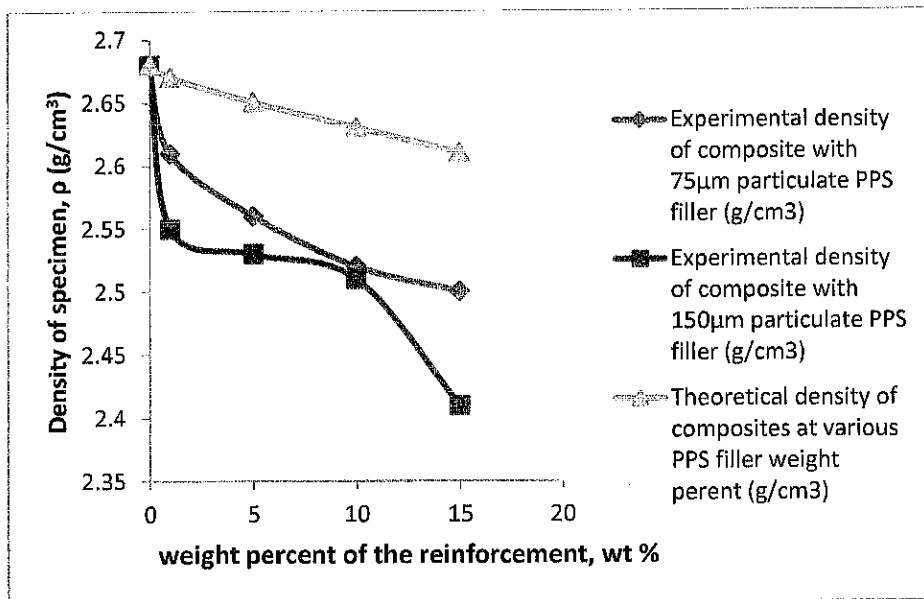


Figure 4.1: Density of the specimen

From figure 4.1, it is observed that the composite has lower density than the alloy. The density decreases gradually as the weight fraction of the filler increases. There is more significant decrease in density of composites using a filler of higher particle size. The decreased density with addition of the particulate periwinkle filler is a result of the lower density of the filler

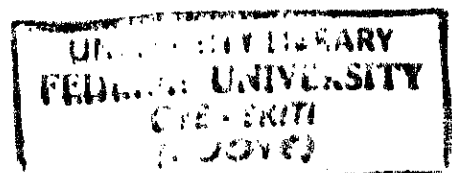
compared to the matrix. This is a positive development because it will further maximize the utilization of aluminium alloy where lighter weight is desired and properties such as specific strength and stiffness will be higher in the composite compared to the aluminium alloy.

The porosities of the various composite are shown in table 4.1

Table 4.1: Porosities of the various composites

Specimen	Particle size of filler (μm)	Weight percentage of filler in composite (Wt %)	Void fraction	Void percentage (% porosity)
2	75	1	0.025	2.5
3	75	5	0.037	3.7
4	75	10	0.045	4.5
5	75	15	0.044	4.4
6	150	1	0.048	4.8
7	150	5	0.048	4.8
8	150	10	0.049	4.9
9	150	15	0.078	7.8

The difference between the theoretical densities of the composites and the experimental densities is as a result of porosity or void in the composite materials. The porosity increases with increase in filler content. The degree of porosity is higher using filler of higher particle size due to low surface area of the filler. Porosity is as a result of trapped air or poor wettability of the reinforcement. High porosity results to poor strength and other mechanical properties.



Generally, the porosity of the composite materials is low due to the two-step casting method adopted and the ability of the alloy to wet the PPS. Figure 4.2 shows the variation of porosity in the various composite samples.

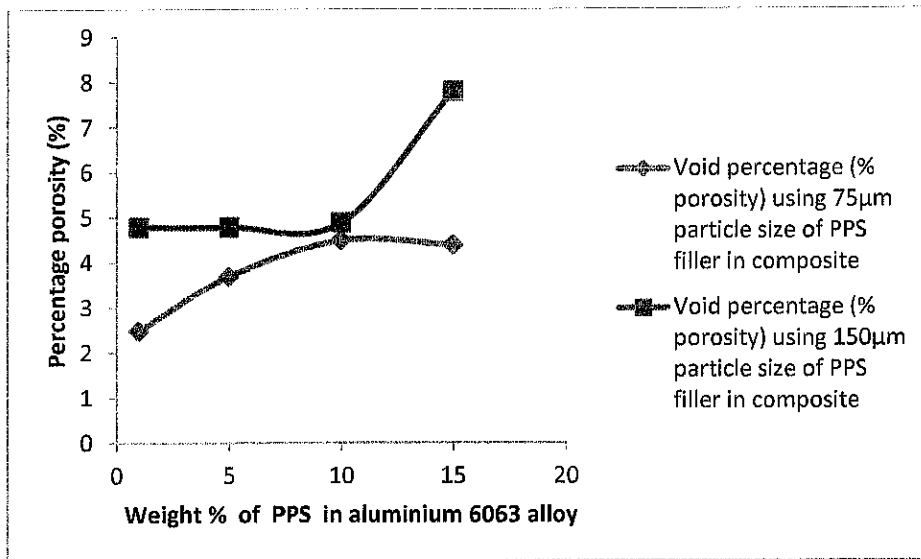


Figure 4.2: Variation of porosity in the various composite samples

More research could be carried out in the areas of improving matrix-filler wettability in the composite by techniques such as filler coating, heating of filler and by the modification of the casting technique such as stirring time and pouring temperature or use of powder metallurgy to further reduce porosity.

4.2 Microstructure

Results of optical microscopy are presented in figures 4.31 – 4.39

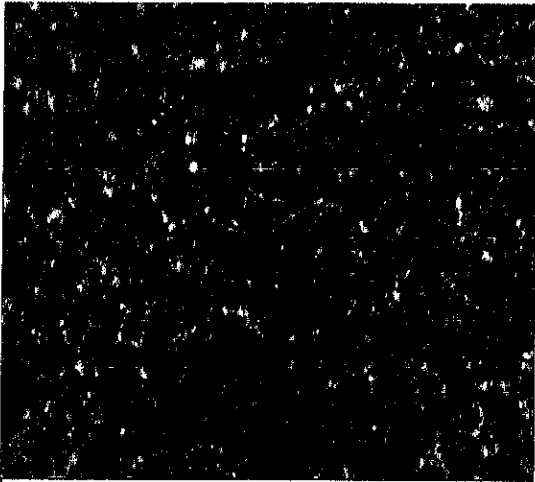


Figure 4.31: Micrograph of specimen 1 (x50)

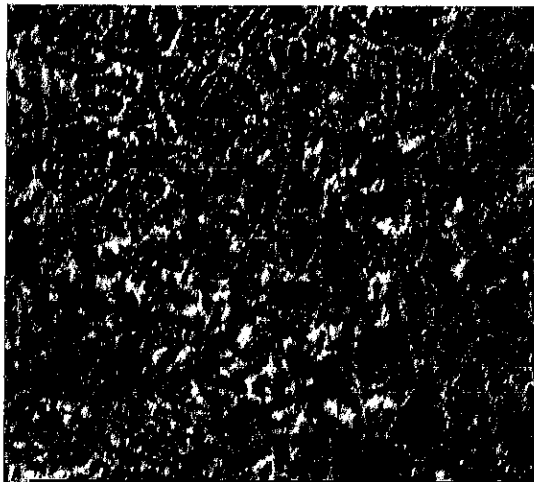


Figure 4.32: Micrograph of specimen 2 (x50)

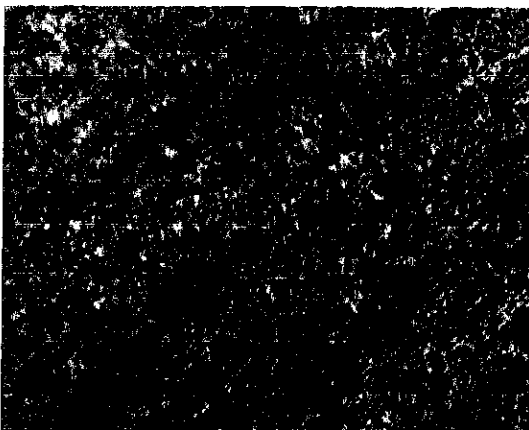


Figure 4.33: Micrograph of specimen 3 (x50)

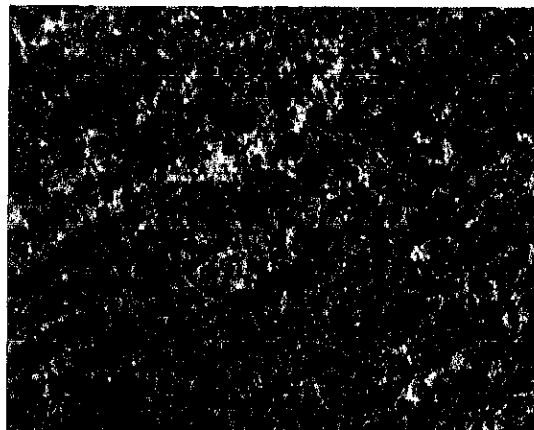


Figure 4.34: Micrograph of specimen 4 (x50)

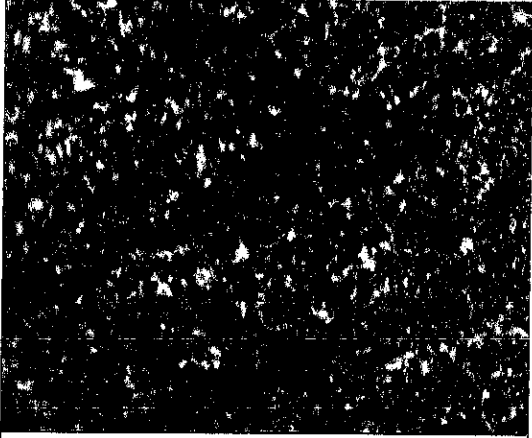


Figure 4.35: Micrograph of specimen 5 (x50)

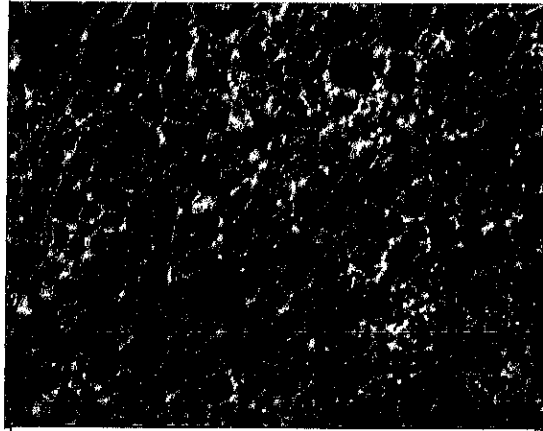


Figure 4.36: Micrograph of specimen 6 (x50)

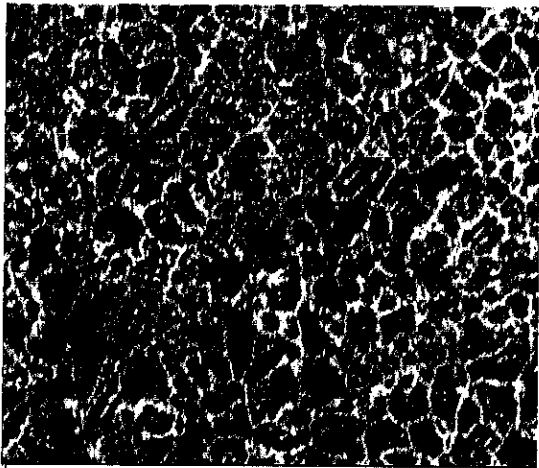


Figure 4.37: Micrograph of specimen 7 (x50)



Figure 4.38: Micrograph of specimen 8 (x50)

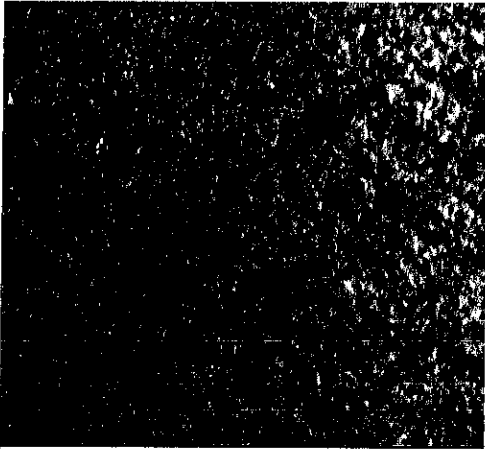


Figure 4.39: Micrograph of specimen 9 (x50)

4.3 Chemical Composition

Table 4.2: Major elemental composition of PPS

Element	Ca	Fe	Si	Mo	Al	P	S	Sn	Sb	Others Elements
Content	70.3350	0.5066	0.0724	0.2372	0.1938	0.2746	0.3987	0.4561	0.4511	27.0745

4.4 Mechanical Properties

4.4.1 Tensile Properties

4.4.1a Tensile Strength

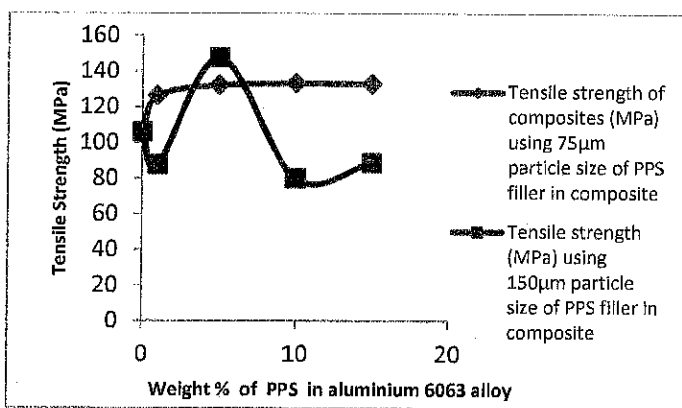


Figure 4.4.1a: Tensile strength

4.4.1b Elastic Modulus

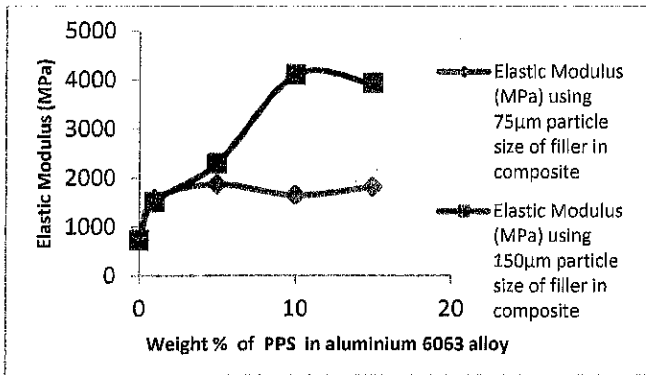


Figure 4.4.1b: Elastic Modulus

4.4.1c Percentage Elongation

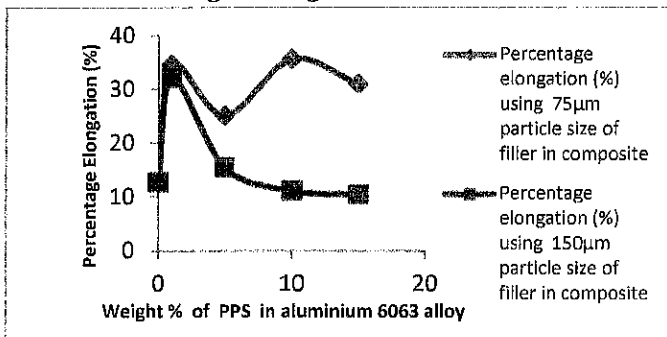


Figure 4.4.1c: Percentage Elongation

4.4.1d Fracture Strength

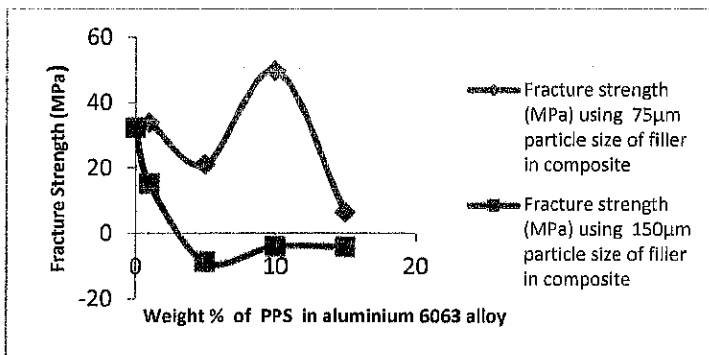


Figure 4.4.1d: Fracture Strength

4.4.1e Energy at break

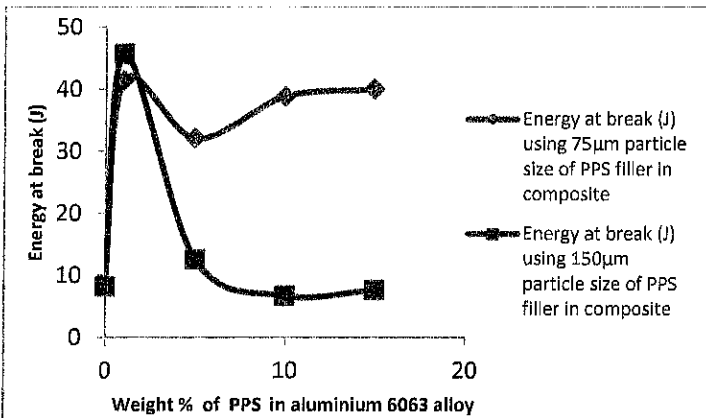


Figure 4.4.1e: Energy at break

4.4.2 Hardness

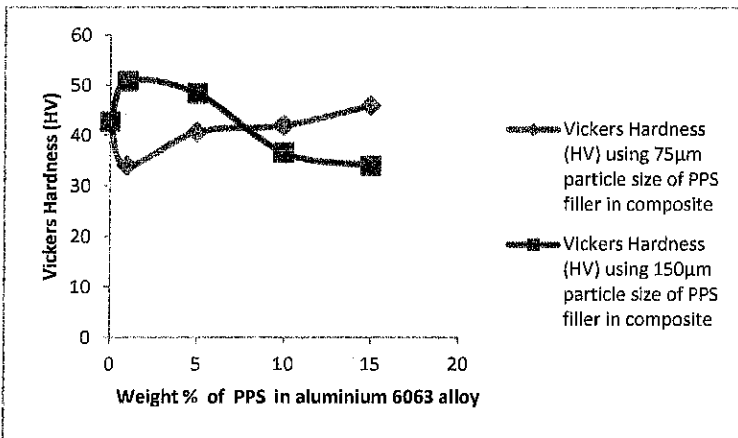


Figure 4.4.2: Hardness

4.5 Discussion of results

4.5.1 Mechanical Properties

The mechanical properties of the composites improve with addition of filler with lower particle sizes.

4.5.1.1 Tensile properties

The tensile properties showed how the aluminium alloy and aluminium-PPS composites reacted to tensile forces. Figure 4.4.1a - 4.4.1e showed the graph of tensile properties versus weight percent of PPS in aluminium 6063 alloy. From these graphs it can be seen that the materials were ductile. The reason for the ductility is because the composites sustain large deformations before fracture. From figure 4.4.1a shows the tensile strengths of the composites. At lower particle sizes, the tensile strengths of the composites were improved with increase in weight fraction; this is attributed to the fact that the composites at lower particle sizes possess high surface area therefore resulting to good wettability and less porosity but at higher particle sizes, the tensile strength of the composites were reduced with increase in weight fraction and this is attributed to the fact that the composite at higher particle sizes possess low surface area which results to high porosity and poor wettability. From figure 4.4.1b, the increase in elastic modulus is as a result of using harder material (PPS) to reinforce softer material (Aluminium alloy) while from figure 4.4.1c, the percentage elongation (ductility) which was meant to decrease was increased due to the presence of calcium that refined aluminium grains from coarse grains to fine grains.

4.5.1.2 Hardness

Figure 4.4.2 shows that at lower particle sizes, the hardness of the composites increased with increase weight fraction of the filler. This is because PPS possesses higher strength than aluminium alloy due to presence of iron which is a harder material than aluminium in the filler. Also, high surface area of PPS with lower particle sizes improves wettability of the filler by the

matrix. But at higher particle sizes, the hardness of the composites is decreased due to high porosity and low wettability.

4.5.2 Microstructure

Optical microscopy analysis was done to observe the distribution and interaction of PPS fillers in the aluminium matrix. Figure 4.31 – 4.39 shows the micrograph of aluminium and aluminium-PPS filler. It was observed that the PPS was well dispersed in the matrix; this is due to the fact that there is good wettability from the matrix on the PPS. Figure 4.32 – 4.35 shows that the PPS was well dispersed and there is low porosity as a result of two step casting techniques used and also as a result of high surface area of the PPS particles. Figure 4.36 – 4.39 shows that PPS was well dispersed but there are high porosity due to low surface area PPS particles. In figure 4.31 – 4.39, it was also observed that the presence of calcium in the PPS refined the grains from being coarse to fine.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The study showed from the optical microscopy microstructural analysis that the PPS is well dispersed in the interface of the aluminium matrix. The dispersion is due to the fact that the matrix was able to wet the PPS filler. Also, it showed that the grains of the aluminium alloy were refined from coarse grains to fine grains due to the presence of calcium which refine aluminium grains in the filler.

It was found from the study that density of the composites reduces with increase in weight percentage and this is due to the fact that PPS has lower density than aluminium alloy. The composites at small particle sizes also possess low porosity due to the fact that the PPS with small particle sizes possess high surface area and also due to the two steps casting methods used. But the composites at high particle sizes possess high porosity due to the fact that PPS possess low surface area which in the process reduces wettability of the PPS by the matrix.

Also, it was found that the tensile strength and hardness of the composites were improved with increase in weight percent at small particles sizes and this is also due to good wettability by the matrix on the PPS and also due to high strength of the PPS.

Hence, aluminium-PPS metal matrix composites at small particle size of fillers could be used in engineering applications such as aeronautical and automobile applications due to their low weight to strength ratio. Also, it could be used in engineering structures, marine application, automotive bumpers and sporting goods due to the high strength and hardness. Other applications of aluminium-PPS metal matrix composites include compressor bodies, vanes and

rotors, piston sleeves and inserts, connecting rods, drive shafts, fan blades, and shrouds, springs, bumpers, interior panels, tires, brake shoes, clutch plates.

5.2 Recommendation

1. Effect should be made to commercialise the production of Aluminium-PPS metal matrix composites since the data from the results makes it attractive for several applications.
2. The government should assist engineering research in universities through the provision of some good quality equipment for materials characterizations such as X-ray Diffractometer, Scanning Electron Microscope, Instron Universal Testing Machine and Optical Emission Spectrometer
3. The use of smaller particle sizes of PPS for composite production should be further investigated.
4. Coating of the surface of PPS with big particle sizes with coupling agents should be done to further improve interfacial adhesion.
5. Further research could be carried out in the areas of utilization of PPS as nanoparticles in composites.
6. The effect of process parameters such as stirring time, stirring speed, casting temperature etc can further be investigated in the composite production.
7. Further characterisation could be done in the areas of wear properties, electrical properties, thermal properties, effect of variation of casting parameters and effect of heat treatment on Aluminium-PPS metal matrix composites.

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