INFLUENCE OF SHORT HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF REINFORCED CNT THIXOFORMED ALLOY A319

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This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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DECLARATION

I hereby, declared this report entitled "Influence Of Short Heat Treatment On Microstructure And Mechanical Properties Of Reinforced CNT Thixoformed Alloy A319" is the result of my own research except as cited in references.

Signature : ……………………………………

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Date : 2nd SEP. 2021

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

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ABSTRAK

Kertas kajian ini akan membincangkan evolusi struktur mikro dan pengembangan sifat mekanik dalam Aluminium Alloy A319 yang diperkuat dengan jumlah serbuk karbon nanotube, CNT yang berbeza. Aloi A319 yang diperkuatkan ini akan menjalani proses thixoforming dan rawatan haba T6 sebelum diuji sifat mekaniknya dan juga struktur mikro setelah dirawat dengan rawatan haba pendek. Sehingga hari ini hanya terdapat kajian terhad mengenai CNT serbuk yang digunakan sebagai elemen penguat dalam bahan kejuruteraan terutamanya pada Al 319. Dalam kajian ini, Kaedah Taguchi yang merupakan eksperimen berdasarkan DOE telah dipraktikkan dengan dua parameter yang kami gunakan seperti masa pengadukan dan peratus berat Kandungan CNT. Terdapat 0% berat, 0,25% berat, dan 0,50% berat CNT serbuk yang digunakan sepanjang eksperimen ini dan sampel akan diimbas dengan menggunakan DSC untuk memperkirakan suhu optimum. Data kemudian dapat dikemukakan. Hasil daripada sembilan sampel yang diperolehi daripada eksperimen DOE hanya satu sampel yang terbaik akan menjalani ujian untuk mengkaji sifat mekanik dan struktur mikro. Proses cerun pendinginan digunakan untuk menghasilkan bahan baku sementara evolusi struktur mikro sampel juga dianalisis dengan menggunakan Mikrograf Optik dan Analisis Gambar. Eksperimen kemudian akan dilanjutkan dengan proses thixoforming yang selanjutnya akan dirawat dengan pelindapkejutan dalam proses air dan diikuti dengan proses penuaan buatan. Dengan menggunakan dua kaedah ujian mekanikal, termasuk ujian kekerasan dan ujian tegangan, semua sampel akan diuji. Menurut ASTEM: E8M standard spesimen dipotong dengan menggunakan Wire-cut EDM dan diuji dengan Universal Testing Machine sehingga patah tulang berlaku. Sementara untuk ujian kekerasan, sampel akan diukur, dan semua data yang dikumpulkan rata-rata minimum 10 bacaan dicatat untuk nilai kekerasan Vickers dan kemudian ditunjukkan dalam nilai HRB. Oleh itu, pada akhir eksperimen kami menantikan peningkatan positif dalam aloi komposit CNTs-Al319 dalam evolusi struktur mikro serta sifat mekaniknya kerana kami dapat menyimpulkan bahawa pada akhir eksperimen ini sifat mekanik rawatan haba pendek di Thixoformed Al319 Alloy telah meningkat sebanyak 12 HV dan 28 HV dalam ujian kekerasan untuk bahan As-cast dan Thixoforming masing-masing berbanding dengan rawatan haba pendek iaitu 108 HV.

ABSTRACT

This paper will discuss the evolution of microstructure and development of mechanical properties in Aluminium Alloy A319 reinforced with different amount of carbon nanotube, CNT powder. This reinforced A319 alloy will undergo thixoforming process and T6 heat treatment before being tested for its mechanical properties as well as its microstructure after being treated with short heat treatment. Until today there is only limited study on powdered CNTs used as reinforcing elements in engineering materials especially on Al 319. In this study, Taguchi Method which is DOE based experiment has been practiced with two parameters that we used such as stirring time and weight percent of CNTs contents. There are 0 wt.%, 0.25 wt.%, and 0.50 wt.% of powdered CNTs being used throughout this experiment and the sample will be scanned by using DSC in order to estimate optimum temperature. Data can then be presented. From nine samples resulted from DOE experiment, only one of the best samples will undergo mechanical test and microstructure test. Cooling slope process was used to produce feedstock while the evolution of microstructure of samples was also being analyzed by using Optical Micrographs and Image Analysis. The experiment then will be continued with thixoforming process that will be further treated with quenching in the water process and followed by the artificial aging process. By using two mechanical testing methods, including hardness test and tensile test, all the samples will be tested. According to ASTEM: E8M standards the specimens were cut by using Wire-cut EDM and being tested with Universal Testing Machine until fracture occurs. While for hardness test, the sample will be measured, and all average collected data minimum 10 readings were recorded for Vickers hardness values and then represented in value of HRB. Hence, at the end of our experiments we are looking forward to positive improvement in CNTs-Al319 composite alloy in microstructure evolution as well as their mechanical properties as we could conclude that at the end of this experiment the mechanical properties of short heat treatment in Thixoformed Al319 Alloy have increased by 12 HV and 28 HV in hardness test for As-cast and Thixoforming materials respectively as compared to short heat treatment which is 108 HV.

DEDICATION

I wholeheartedly dedicated my research work to my most beloved parents, who have been giving me positive and undeniable support. Thank you to them and also to my two sisters for being my source of inspiration and strength when I thought of giving up and continually provide their time in order to give me moral, spiritual, emotion and the most important is financial support. Thank you to my parent and my sisters for always be there in my journey.

I also grateful to Almighty Allah SWT, for giving me the power of mine, protection, skill, and strength as today world is really challenging with Pandemic Covid-19.

This work also specifically being dedicated to my supervisor, Assoc. Prof. Ir. Ts. Dr. Mohd Shukor B Salleh, for providing relevance solution for any arise issues throughout this experiment being conducted by me. Also, special gratitude to my senior, Senoir Raudah, a master student of FKP for always shows me how to run the experiment in our workshop correctly and sharing your knowledge with me. To our JK PSM and panels thank you for planning any possible solution in order for us to complete our Project Sarjana Muda on time as well to help us completing our study.

And lastly to my family, supervisor, Assoc. Prof. Ir. Ts. Dr. Mohd Shukor B Salleh, seniors, friends and JK PSM, panels for providing me with useful tips and references. I also thank them for their words in term of encourage and advice to finish this research.

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This research would be impossible to complete without participation and assistance of so many people whose names may not all be clearly stated but I am truly grateful for them.

TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing And Materials
Al-MMCs	-	Aluminium Metal Matrix Composites
Gr	-	Graphene
ANSI	-	American National Standards Institute
CNC	-	Computed Numerical Control
CNTs	-	Carbon Nanotubes
Cu	–	Copper
C	-	Carbon
CS	-	Cooling Slope Casting
CO_2	-	Carbon Dioxide Gas
DC	-	Direct Current
DSC	-	Differential Scanning Calorimetry
EDX	-	Energy Dispersive X-ray
EDM	-	Electrical Discharge Machine
Fe	-	Iron
HIP	-	Hot Isostatic Press
HT	-	Heat Treatments
HRTEM	-	High-Resolution Transmission Electron Microscopy
MWCNTs	-	Multi- Walled Carbon Nanotubes

MMCs	-	Metal Matrix Composites
Mg	-	Magnesium
OM	-	Optical Microscopy
PM	-	Powder Metallurgy
PMCs	-	Polymer Matrix Composites
Si	-	Silicon
SHT	-	Short Heat Treatment
SEM-EDX	-	Scanning Electron Microscopy with Energy Dispersive X-ray
SIMA	-	Strain Induced Melted Activation
SSM	-	Semi Solid Metal Processing
SWCNTS	–	Single Walled Carbon Nanotubes
UTM Universal	-	Testing Machine
UTS	-	Ultimate Tensile Strength
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescence
YS	-	Yield Strength

LIST OF SYMBOLS

%	-	Percentage
µm	-	Micrometer
Bal.	-	Balanced
°C	-	Degree Celsius
°C/min	-	Degree Celsius Per Minute
cm	-	Centimeter
g/cm^3	-	Gram Per Cubic Centimeter
g	-	Gram
GPa	-	Giga Pascal
HV	-	Vickers Hardness
kHz	-	Kilohertz
km	-	Kilometer
kN	-	Kilo Newton
kW	-	Kilowatt
L/min	-	Liter Per Minute
m/s	-	Meter Per Second
mm	-	Millimeter

MPa	-	Mega Pascal
Nm	–	Nanometer
rpm	-	Rotation Per Minute
TPa	-	Tera Pascal
wt. %	-	Weight Percentage

CHAPTER 1 INTRODUCTION

1.1 Background of Study

Aluminium is a non-molecular element in Group 13 of Periodic Table Elements with a lightweight silvery white metal. This element contributes a few of its electrons valence to metallic lattice resulting in metallic bonding which soon give outstanding properties to this white element such as malleable, a good heat and electric conductor due to their abundant free electrons, ductility and the most important is their lightweight that make it an outstanding raw material chosen by variety of fields and industries. In order to increase specific properties certain elements with controlled composition are added such as addition of Silicon, Si can increase in castability as Si will help in reducing solidification shrinkage while increase the fluidity same as when added some copper elements to Al-Si alloy that help in formation of Al_2Cu phase and some intermetallic compounds. These compounded elements will increase mechanical strength such as hardness by undergo heat treatment (Magno et al., 2017).

Carbon nanotubes, CNTs is a new element that is excellent to become reinforcing elements due to their extremely and lightweight besides their physical properties that pointed out is their opportunity to develop ultra-high strength in low – weight materials that in a same time will conduct electrical and thermal properties excellently as CNTs was made up of rolled – up sheets of single- layer carbon atoms (graphene) that build up to form a cylindrical molecules.

According to previous study semi-solid metal (SSM) processing was used in order to exploit all beneficial characteristics and properties of metallic alloys as the structure will changes gradually from dendritic to spheroidal. Thixoforming process involved solidifying billet and preheating prior to billet. From this study, strength, and hardness of an alloys (A319) can be represented as the formation of Mg_2Si a compound in globular microstructure. The structure was a result from shearing force on CS plate plus influence from the cooling rate in mould during the entire process. Intermetallic compounds such as Al_5FeSi, Mg_2Si, Al_2Cu and $Al_5(Fe, Mn)_3Si_2$ also a part of CS product that showed globularization of solid nuclei. The percentage increment in hardness by CS casting process was 12.2% compared to as-cast A319. Based on Vickers reading obtained by Salleh et al. for the thixoforming sample, there are 104 ± 2.47 HV while for as- cast sample, the reading showed was approximately of 89.7± 4.4 HV (Aziz et al., 2016).

The evolution of microstructure of A319 aluminium alloys by using CS casting process can be summarized and explained by using Kirkwood schematic illustration that consist of four parts/ zone which are impact zone, top zone, middle zone, and bottom zone. Each zone has different schematics diagram such as in impact zone is where we called as dendritic growth stage as nucleation of α – Al occurs actively. Top zone, dendritic structure starts to evolve into rosettelike shape, (rosette growth stage). While, in middle zone of CS plate, structures of alloys slowly change from rosette stage into some nearly spheroidal structure as the alloy enters the ripened rosette stage resulting from their microstructural evolution as well as for the bottom zone of CS plate the structure of alloy was gradually changed, where only few ripened rosette microstructures still present but mainly consist of nearly spheroidal microstructures. Ruckenstein et al. (2015) state that nucleation is the emergence of a new phase nuclei in first phase transition. By using CS plate, it may be acts as nucleating agent in promoting α- Al as well as enable the contact with shearing force for fragmentation of α – Al.

To prevent catastrophic failure in T6 heat treatment as well as their consuming process steps, short heat treatment that is much more economic in term of money and time can be applied. According to Hashim et al., 2021, short heat treatment can be done in 1 hour solution treatment, water quenching and another 2 hours of artificial aging can improve the mechanical properties of alloying and composite elements more effective compared to basic T6 heat treatment that most industries used today.

Redesign the experiment to examine microstructure of thixoformed billet with different filling contents. To the best of our knowledge, limited studies about carbon nanotubes reinforced metal matrix nanocomposites have been reported so far especially in Al 319 as the metal element. Due to lack of information and limited methods, we can say that bimetallic matric with CNTs reinforced composites have been hardly being study by researchers and engineers (Md. Hasan Ali and Robiul Islam Rubel, 2020). This study is about to investigate an appropriate technique in producing aluminium billet composite and to increase mechanical properties of thixoformed A319 Alloy that can save more time and more economic- friendly.

1.2 Problem Statement

In thixoforming process it was important to get homogeneous consistency throughout the whole process, alloy decrease in viscosity if it is sheared but will thicken again if it was allowed to. Hence, there must be only specified volume of solid fraction as the relationship of apparent viscosity and shear rate can be explained based on Poiseuille's equation. To put it simple, graphs on variation in apparent viscosity versus isothermal holding time at a constant shear rate and temperature shows purely relationship to the various solid fraction. Therefore, it is important to understand about each substance properties especially raw material for thixoforming process in order to get the right amount of material fraction to produce products with maximum tensile strength and elongation fracture.

Besides, as widely known alloy especially A319 can be classified as the most important engineering materials as it was applicable in many fields. But aluminium alloys development required a lot of research study in order to avoid scarifying the ductility of aluminium alloys while mechanical properties will be improving. However, the agglomeration and the dispersion of reinforcing elements of CNTs in AL 319 are still at improvement state if we use powder metallurgy process such as ball milling as the process can destroyed the structure of the reinforced elements which is CNTs (Hanizam et al., 2019) As a result, we are trying to mix CNTs as a raw material to produce aluminium billet composite using basically thixoforming process.

Carbon nanotubes, CNTs was selected because of its outstanding reinforcing elements in various engineering material that produce several categories of matrix such as metallic, polymer and ceramic that can be classified as majorly used. With high tensile strength that are approximately 100 GPA, large elasticity (~ 18% of elongation before fracture) as well as bending ability without damage, the ability to conduct electrical and high thermal. (Michael Berger, 2018).

However, previously there are still limited case study and research done related to the behavior of aluminium A319 Alloys reinforced with CNTs especially when treated with artificial aging by using short heat treatment. Many industries used conventional T6 heat treatment that can take roughly of 2 to 7 to 8 hours of solution heat treatment at temperature of 530 °C, another few hours for water quenching at 20 °C and proceed to 4 hours precipitation hardened process at 160 °C to improves mechanical properties (Rahman et al., 2020). This process will need longer time to produce the composite as well as it may consume more money.

1.3 Objectives

1. To investigate an appropriate technique with low cost and short time processing to produce aluminium billet composite of CNTs reinforced A 319 Alloy.
2. To examine the microstructure of the thixoformed CNT-reinforced billet with different CNTs contents.
3. To study the effect of short heat treatment on the mechanical properties of thixoformed CNTs reinforced Al 319 Alloy.

1.4 Scopes

This study will be conducted in research laboratory environment. Hence, some limitation must be highlighted to minimize unwanted results. It will be quite challenging to control air humidity in open space. There also very limited study of Carbon Nanotubes, CNTs content as reinforcement phase in aluminium A319 alloys in order to increase mechanical properties. From there, we find out that it difficult to produce metal matrix composite reinforced by well dispersed additional material/ substance such as Mg and Zr via conventional metallurgical process due to high temperature environment and density different as stated above.

Semisolid metal processing (SSM) or thixoforming process will be used to carry out this experiment. This process will be produced minimum casting defect while exploits metal rheological behaviors in solidus and liquidus state with uniform heating temperature needed.

With outstanding advantages that aluminium alloy can provide to user, aluminium alloys were significant material and has being used optimally in fields like aeronautical, aerospace industries like electronic industries and the mostly widely used in automotive field.

This study also will be involved short heat treatment process in order to produce Thixoformed Al 319 alloy reinforced by different CNT contents. The collected data will then analyze using SEM- ERD for microstructure investigation and will conduct hardness test and tensile tests for analyzing the mechanical properties.

1.5 Significant of study

After completing this study, we believe that there are some potential benefits that can be enjoyed by various fields especially automobile field. By using graphene content in thixoformed alloy A319, we could see an excellent reinforced effect on mechanical strength without compromise the ductility of an Al alloy matrix. There will be about 50% increasement in yield strength while tensile strength will increase from 373MPa to 400MPa until 467 MPa.

At the end we are able to produce a product that helps industries to fulfill their needs in producing an aluminium alloy billet composite with high mechanical properties. We also can develop a brand-new solution especially for raw material selection in SSM technique by the means of cut any unnecessary cost. Thixoforming technique will based on forming alloys in semisolid state into near net- shaped products that will increase hardness and reduce time as minimum casting defects such as macrosegration can be avoided and no second finishing process needed.

1.6 Organization of report

In chapter 1, background of the study was being discussed in a brief way in order to give to some idea to readers about what will this study is about. Some problems are identified through articles and journal reading related to title selection and why we need to carry this study. It may also based on citation of journal that currently need a study. Next, objectives to be achieved were drawn based on problem that we want to solve at the end of the study. This is followed by scope that state our research or study limitation and what method we are using in order to solve the problem while narrowing area of study. The impact of this study especially to automobile industry was revealed. Last but not least, summary was provided at the end of this chapter where all important things and issues will be summarized to wrap up the study.

1.7 Summary.

This work typically investigates the influence of solution/ ageing treatment on microstructure and mechanical properties of A319 Alloy in different CNTs content by using short heat treatment, involving T6. Composites will be produced by thixoforming process and their microstructural characterization analysis will be carried out related to mechanical behavior in different conditions: as thixoforming product and after solution / ageing treatment. We may also compare the different of microstructure and mechanical properties of CNTs- Al 319 alloy after being treated with conventional T6 Heat Treatment with T6 Short Heat Treatments.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will provide some survey of scholarly sources related to Thixoformed Carbon Nanotubes- reinforced A319 aluminium billet alloy. This study will investigate the effect of different CNTs powdered content on the mechanical properties of A319 aluminium alloy after being treated with short heat treatment specifically thixoforming process. In order to enhance reinforcement efficiency of CNTs in metal matrices, semi-solid metal (SSM) processing has been used. In this paper, metal matrix composite (MMCs) is a mixture of carbon nanotubes, CNTs powder and A319 aluminium alloy. Carbon nanotubes, CNTs act as reinforcement agent while A319 aluminum alloy acts as metal matrix producing a new development of CNTs - reinforced metal matrix composites (CNT-MMCs). In thixoformig process, the billet will be solidifying and preheating prior to forming the billet before a series of mechanical testing and material behavior of the thixoformed CNTs - reinforced A319 alloy be conducted.

2.2 Composites

Composite is a composition of materials from two or more-part materials with different properties, chemically or physically that will be combined to increase end product properties especially mechanical properties. Amalgamation of a base material and filler material is a results of combination process for composite without lose their individual identities, while synergistically contribute their special traits to improve mechanical properties such as high strength and stiffness with low thermal expansion (Rajak et al., 2019). In this experiment, CNTs - reinforced A319 aluminium alloy properties have been carried out.

2.2.1 Classification of composite materials as according to constituents of types used.

Figure 2.1: Classification of composite.

2.2.2 Advantages and applicable properties of composite based material.

Sandwich-liked structure of composite material offered the biggest advantages as a light in weight but also strong and hard. Design flexibility in composite materials allow them to be molded into complex shapes. Kainer (2006) highlight some of the most applicable properties proposed by composite material such as:

- High wear resistance
- High Young’s modulus
- High corrosion resistance
- Low density
- High stiffness and high strength
- High fatigue strength, especially at elevated temperature
- High thermal and electricity conductivity

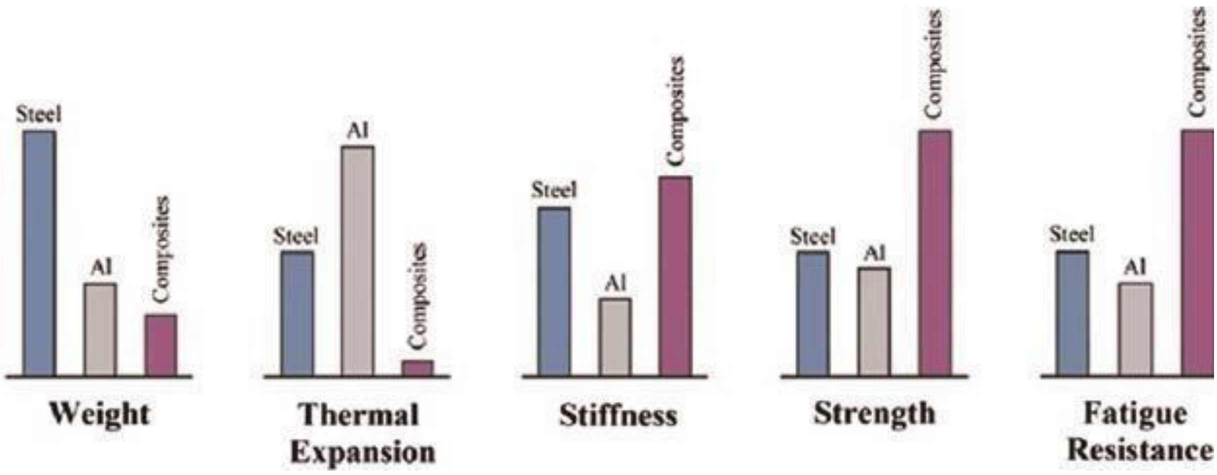


Figure 2.2 Properties of composite as compared to most commonly used alloys, steel and aluminium.

2.3 Metal Matrix Composites MMCs

The MMCs is a composition of continuous metallic matrix such as aluminium, magnesium, copper and titanium that was reinforced into a few phases either in dispersed ceramics or metallic phase to extract desirable properties of metallic materials and the reinforcement materials, second phase. MMCs development can improved specific stiffness and strength relative to unreinforced alloys. For example, reinforcing aluminium with 25% of particulate silicon carbide increases this to nearly MN m kg^{-1}. Some specific properties can be exploited such as weight savings and/or improvement of fatigue resistance, (Akhtar,2014).

Metal matrix composite gaining importance as this matrix composite can be mix together in solid, liquid or vapor states. Carbon fibers that will used for MMCs mostly in the form of continuous fibers, but short fiber is also applicable. Materials such as aluminium, magnesium, silver alloy, titanium, copper, nickel, and tin will act as matrices that will impose different applications. Because of low cost and good machinability, aluminium is the most widely used matrix metal. The mechanical properties of aluminium such as low density and relatively low temperature makes fabrication and joining process relatively convenient (Chung, et al., 2017).

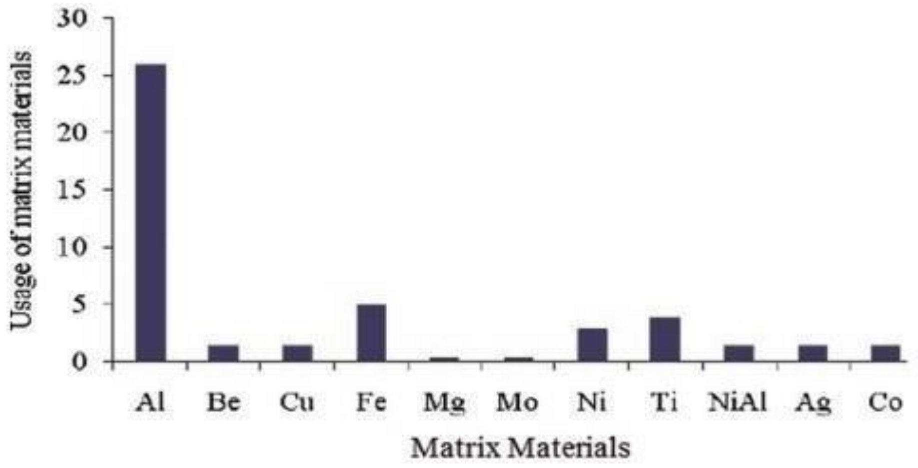


Figure 2.3: Usage of matric material in MMCs *(Adebisi et al. 2011).*

Since in the late of 1950s numerous combinations of matrices and reinforcements have being done. When MMCs compared to monolithic metals and polymer matrix composites, there are some disadvantages in MMCs such as limited-service experience, higher cost of some material system, and complex fabrication methods for fiber-reinforced system except for casting. Hence, development of this system is crucial as this system are still relatively immature technology and there are still a lot more to be explored to maximize the usage of this kind of material- reinforced.

2.3.1 Reinforcement and Matrixes

(Chung, et al.,2017) Reinforcement is a phase that is stronger and stiffer than matrix, also known as second phase(s). This second phase mainly responsible to carries the applied load in the composite. Matrices can be imagined as a soft phase that comes together with specific physical and mechanical properties such as ductility, formability, and thermal conductivity. Matrix is primary phase that holds reinforcement phase and shares applied load with the second phase. This both phases can dictate the performance of composite depending on:

- Physical, mechanical, and chemical properties of matrix and reinforcement.
- The constituents of morphology
- Interface between constituents
- Size and distribution of constituents

2.3.1.1 Reinforcement Forms

The structure and orientation of particles in composite material can be used to classified which category of reinforcements used in MMCs. Hence, the characteristics of reinforcement can generally distinguished metal-matrix composites, MMCs. Bahl, et al., (2020) said that MMCs can be grouped into four categories depending upon the form of reinforcement: a) fiber reinforcements either continuous or discontinuous. With diameter can be in the range of 0.10.5 µm, the length to diameter can go up to 200 for the reinforcement in whiskers form; (b) fiber reinforced MMCs in the formed of particulate that contain either particles or platelets from 0.5 to 100 micrometer(µm) in size varying; (c) particles of diameter less than 0.1 µm is an example of dispersion-strengthened MMCs; and (d) directionally solidified eutectic alloys is one of in situ MMCs.

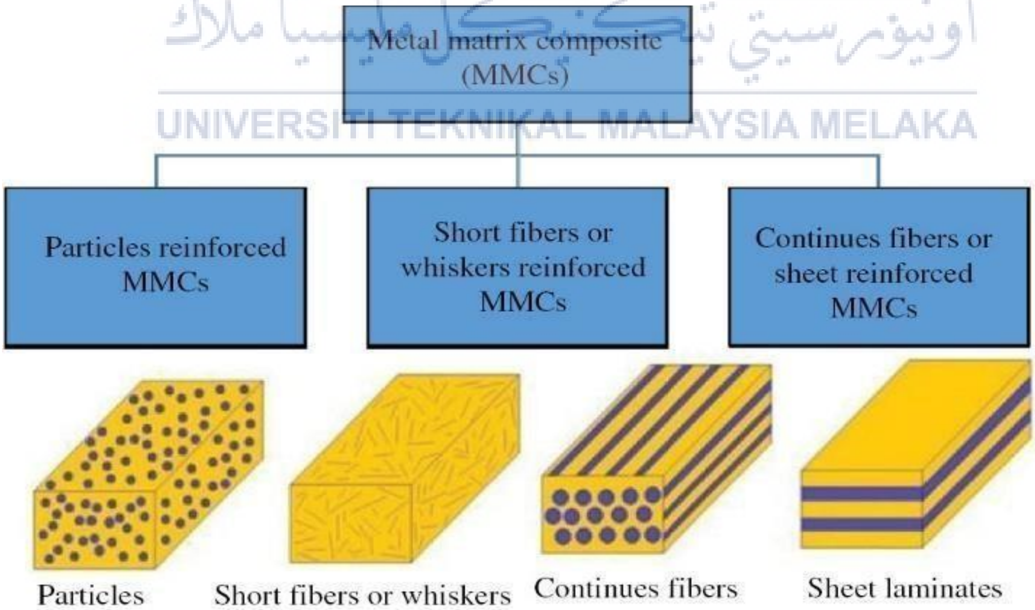


Figure 2.4: MMCs are classified on the basis of reinforcing elements (Science and Engineering of Composite Materials 25, 4. 2017)

D. Stefanescu state that discontinuous fiber reinforced composites and continuous fiber reinforced composites are the main types as they are typically circular with diameter below about 20 µm. The continuous fibers are used as the optimal load transfer takes place from the matrix to the fiber due to the end of the fiber do not provide full stress support and resulting in the end- effect. While, for the discontinuous fibers usually possess high aspect ratio that limiting the end-effect as it reduces it to a very small factor. Hence, the continuous fiber- reinforced MMCs provide the best mechanical properties and commercial potential.

Type	Aspect ratio	Examples of Reinforced Material	Diameter in µm
Continuous fiber	>1000	SiC, Al_2O_3, C, B, W, Nb + Ti, Nb_3Sn	3–150 µm
Nanotube	>1000	C	<100 nm
Short fiber (whisker)	10–10000	C, SiC, Al_2O_3, , SiO_2 + Al_2O_3	1–5 µm
Particle	1–4	SiC,Al_2O_3 , BN, WC	1–25 µm
Nanoparticle	1–4	C, Al_2O_3, , SiC	<100 nm

Source: Chawla, 2012

Table 2.1: Typical reinforcements used in metal–matrix composites

2.4 Fabrication of MMCs

Fabrication method can be divided into two major categories which are primary and secondary. Creating MMCs from its constituents can be considered as primary fabrication methods, while secondary fabrication is the resulting material that may be in a form close to desired final configuration and sometimes may require considerable additional processing such as machining, rolling, metallurgical bonding and forming. According to Alhawari et al., (2013) the most common used metal matrix is aluminium as aluminum is light in weight and low melting temperature.

Fabrication of MMCs can involve metals in solid, liquid or vapor state (Chung, et al., 2017). Solid-phase processes and liquid-phase processes are the most common methods used for fabrication of the MMCs at large-scale industrial level. well-establish methods in solid-state processes include blending of powder followed by isostatic pressing (powder metallurgy (PM) processing), spray deposition techniques, and diffusion bonding. Melts stir casting, melt infiltration, spray casting, and in situ (reactive) processing come from liquid-state processing (Bains, et al., 2016).

2.4.1 Liquid State Fabrication of Metal Composite

Involves incorporation of dispersed phase into a molten matrix metal, followed by solidification by itself. Good interfacial bonding also known as wetting between dispersed phase and liquid matrix should be obtained in order to provide high level of mechanical properties of the composite (Chandra Kandpal, et al., 2018). Stir casting, Infiltration like gas pressure infiltration, Squeeze casting infiltration or Pressure die infiltration are the methods of liquid state fabrication of Metal Matrix Composite.

2.4.1.1 Stir Casting

Due to the simplicity, proven process, lower cost production and mass production capability stir casting is the most suitable processing to produce MMCs. A bottom tapping stir casting furnace with electromagnetic and ultrasonic stirrer is recommended for the production of MMCs (Chandra Kandpal et al., 2018). Stir casting is a method where reinforced particulate is blended homogenously into aluminium matrix by mechanically stirring at high rotational speeds after that the molten metal is directly transferred to shape mould for solidification (Sharma et al., 2017).

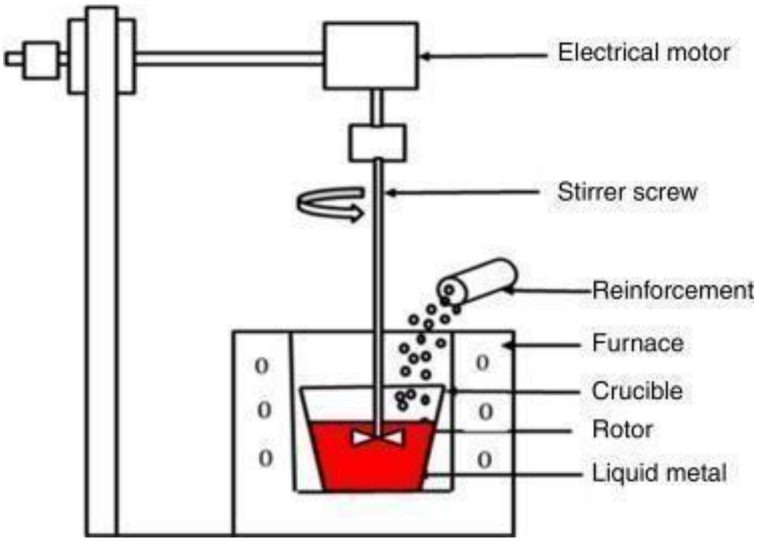


Figure 2.5: Stir Casting Method (Thandalam et al., 2015).

Mechanical properties evaluated by carrying the tensile and hardness testing of standard sample as per the ASTME E8/ M-08. From the microstructural investigation, clustering of particulates is occurring in the 8% SiC and gradually scattering in 9% and 10% SiC (Mishra D., et al., 2020).

2.4.2 Solid State Fabrication of Metal Composite

Solid state fabrication of MMCs is a process of bonding matrix material with reinforcements due to mutual diffusion arising between them in solid states at a higher temperature and under pressure. Panwar et al., (2018) state that the there are two main process recovered under solid state that such as friction stir and powder metallurgy.

2.4.2.1 Powder Metallurgy

Powder metallurgy can produce exactly net shaped components of complex shape with high accuracy (Sharma Pardeep et al., 2014). Ghosh state that the most common solid-phase process is based on powder metallurgy technique as they involve typically discontinuous reinforcements, due to ease of mixing and blending. When the ceramic and metal powder are mixed, isostatically undergoes secondary operation such as extrusion or forging. The ratio of reinforcement particle size to matrix particle size is crucial in achieving a homogeneous distribution of particles in the matrix.

Pm process got four basic steps which is blending of gas-atomized matrix and reinforcement, then compacting the homogeneous blend, next degassing preforms to remove volatile contaminants and lastly consolidation by vacuum hot pressing, as a result hot pressed billet can be extruded as shown in Figure 2.6 (Science and Engineering of Composite Materials, Volume 25, Issue 4, Pages 633–647, 2017)

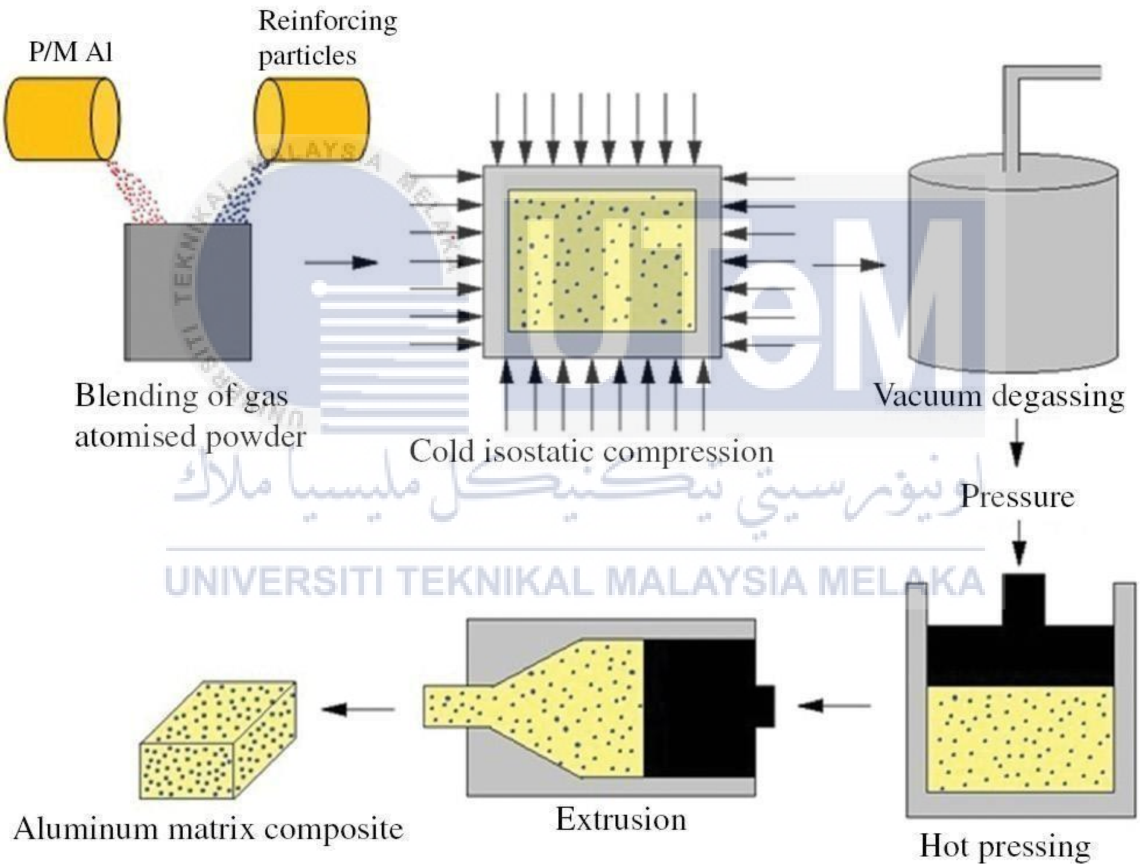


Figure 2.6: Powder Metallurgy Process

Science and Engineering of Composite Materials 25, 4; 10.1515/secm-2016-0278

Among all PM is the best method in producing MMCs due to good wettability between reinforcement and matrix, homogenous microstructure in the end product (fabricated MMC) and the ability to prevents the formation of any undesirable phases (Vani et al., 2018). Gomez L et al., (2009) state that because PM process use temperature that not as high as in fusion techniques, this method can guarantee a good desification and thus matrix/reinforcement contact while any possible reaction that could modify the nature of the materials as well can be hinder. Powder metallurgy is mostly used solid state process for fabrication as it provides uniform distribution of particle but costlier than stir casting Panwar et al., (2018). The drawback of powder metallurgy method is slightly expensive and complex for large quantity of powders, requires a long milling and consolidation time for uniform distribution of reinforcement particle make this process not suitable in mass production (Vasanthakumar et al., 2019).

2.4.3 Semi-Solid Metal Processing (SSM)

Semi solid metal processing is the most useful process in exploiting attributes of the unique properties for the metallic microstructures. Spencer et al., (1972) identified some essential thixotropic properties, while Vasanthakumar et al., (2019) state that SSM is a new technology that offers advantages to both liquid processing and solid processing. By using this process, semisolid behavior as well as reducing macrosegragation, porosity, and forming forces during shaping process can be control. After SSM processing, metallic alloy microstructure becomes spheroidal rather than dendritic, it is important to enable easy move in α-Al when the external load is exerted.

Semi-solid metal processing has two main routes to produce globular microstructure in alloys which is thixoforming and rheocasting. In thixoforming process there are two additional steps compared to rheocasting: solidifying the billet and pre-heating prior to forming the billet. By using SSM processing manufacturer and researcher are able to provide high integrity components, which are heat treatable, provide improved mechanical properties of the end product, produce components/end product with complex shapes and tight dimensional control more easily but based on the current situation, future efforts should be directed in development of new processes, understanding the mechanism for formation of globular structure, development of new alloy for SSM processing, rheological behavior of SSM slurries and microstructure-property relationship in SSM formed materials (Fan, Z. 2002).

2.4.3.1 Rheocasting

Rheocasting is semi-solid slurry that generated directly from liquid, introduced in late 1990s. Midson et al., (2014) state that there are two separate markets evolved for rheocastings namely, the production of castings of exceptionally high quality and the production of improved quality die casting.

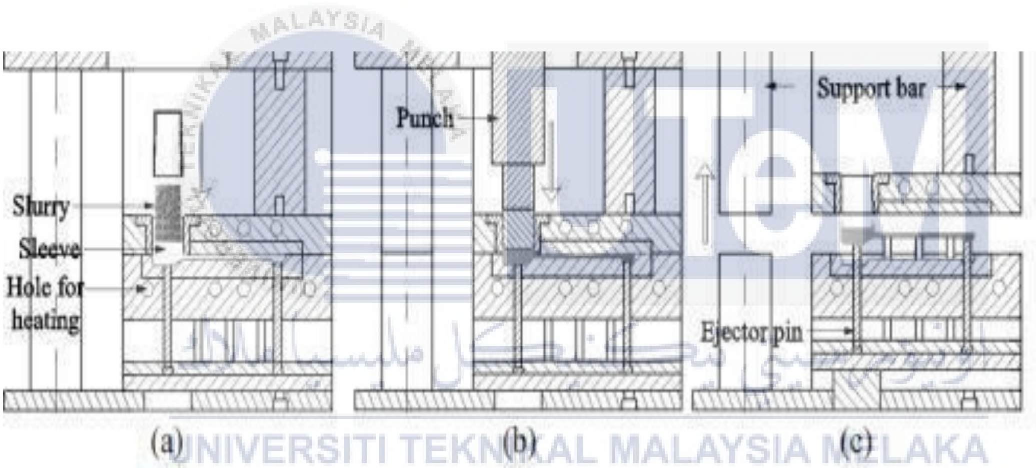


Figure 2.7 : Indirect rheocasting process: (a) input semi slurry, (b) forming and (c) ejecting (Jin et al., 2015).

2.4.3.2 Thixoforming

Flemings and his students were the first to discover thixotropic behavior in the early of 1970s during performing continuous hot tearing test of solidifying Sn-15%Pb. In thixotropic condition, an alloy decrease in viscosity if it is sheared but it will thicken again if it is allowed to stand (Omar, et al., 2011). Thixoforming basically consist of three phases; producing a material with a globular microstructure, heating the material to the forming temperature, and forming the material in die-casting press (Salleh et al., 2013).

Thixoforming is a process which exploits metal rheological behavior in solidus and liquidus in a range of temperature. This process requires uniform heating and partial remelting of alloy slug in order to get homogeneous consistency throughout. Furthermore, this process can produce less casting defect component such as macrosegration, porosity and shrinkage. Many researchers start to focus on new material used to produce superior mechanical properties as a product of thixoforming process (Husain et al., 2017).

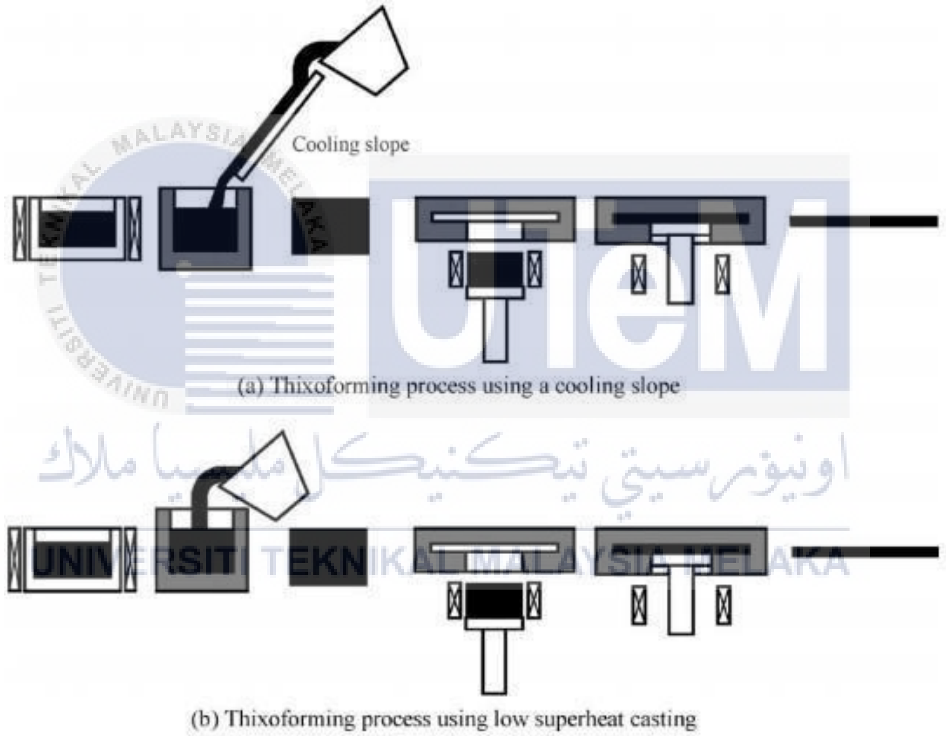


Figure 2.8: Example of two kinds of billet less thixoforming process (Haga et al.,2002).

2.4.3.3 Heat Treatment

Heat treatment involved numbers of simple steps in producing desired mechanical properties in controlled way to exploit the outstanding behaviors of metal such as to make them stronger, increase malleable, increase ductility and increase the resistant to abrasion. Heat treatment helps modify alloys' microstructures without compromise their ability and their structure of the entity that involving solution treatment, quenching and artificial aging as many past research proved that right after five minutes of solution of heat treatment, silicon particles in aluminium, Al 319 start to develop and change into more globular microstructure (Rahman et al., 2020). However, in 2001 and 2016, researchers such as Jerry et al., and Costa et al., proposed methodology that involved four steps to perform T6 heat treatment with expected time and controlled temperature (Table 2.2). This research is then continued by Magno et al., 2017 stated that the aimed of solution treatment is to dissolve Al_2Cu which is in intermetallic states in Al- rich matrix (α-Al)and get the equilibrium state, while in quenching step the formation of Al_2Cu intermetallic metastable phase can eventually spread in α-Al solution by the help of warm water to produce superheated solid solution that will precipitated lastly in aging step (Figure2.9). Conventional T6 heat treatment will take a longer time to complete the whole processes (three main processes), according to Hashim et al., 2021, as the minimum duration of 9 hours needed to complete the processes, solution treatment at 50°C required 6 – 12 hours and for artificial aging another 3 to 5 hours will be spend to complete the process at 155°C.

Process/ Stages	Expected Time, Hours	Controlled Temperatures, in °C
Solution Heat Treatment	5 h	490°C ± 2°C
Quenching in warm water		60°C ± 2°C
Immediate Aging	3 h	155°C ± 2°C
Air Cooling		

Table 2.2: Methodology Proposed by Jerry et al.,2001 and Costa et al., 2016

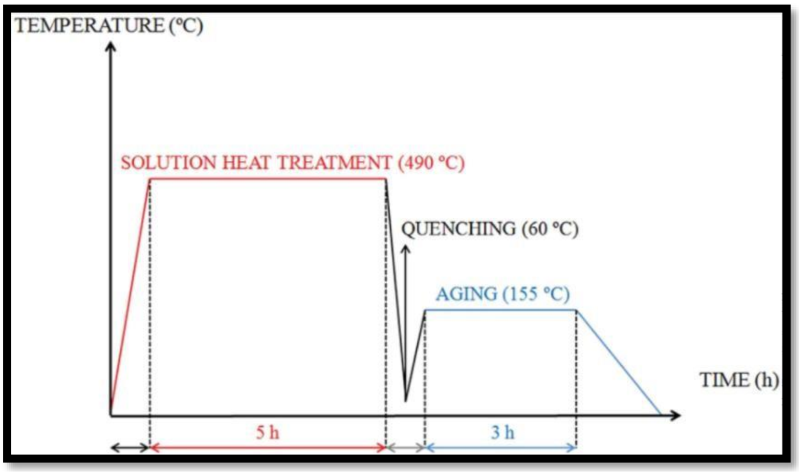


Figure 2.9: Stages of T6 heat treatment applied represented schematically (Magno et al., 2017).

2.4.3.3.1 Short Heat Treatment

A new T6 heat treatment with solution times less than 30 minutes showed better properties in mechanical properties compared to old and standard conditions of heat treatment that needed minimum of 9 hours to complete 3 stages involved in producing SSM composites . According to previous research, researchers stated that time of 50 mins with 540°C in solution treatment is sufficient enough to produce α – aluminium dendritic microstructure with homogeneous distributed silicon and Mg while only 30 minutes is needed for solution treatment when casting A356 alloy at 540°C for low pressure die casting. Thixoformed samples will be treated with shorter solution treatment at 540°C for 1 hour, followed by quenching in water at 26°C and 27°C, room temperature before being aging artificially for 2 hours at 180°C using Nabertherm 30°C to 30000 °C furnace (as showed in Figure 2.10) in short T6 heat treatments (Hanizam et al., 2019) Significantly, reducing solution treatment time when applied in SSM has big impacts on productivity because the cooling rate is faster compared to HPDC, High Pressure Die Casting generating inter-metallic compounds with more small in size and more homogeneous structured components that near to eutectic microconstituent in α – phase globules as well as the economical issues (Menarguesr et al., 2015).

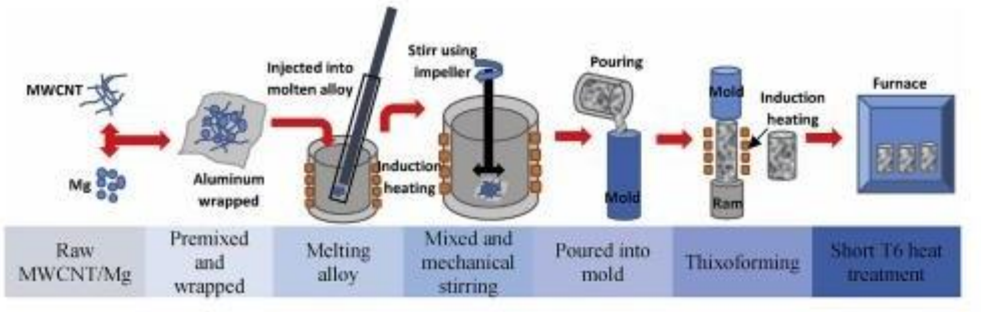


Figure 2.10: Mixing Process involved Short T6 Heat Treatments (Salleh et al., 2019)

While, Chinese researcher such as Lu et al., in 2018 showed in graphical approaches that UTS and elongation percentage of Al - composite alloy were improved 31% and 23 % respectively compared to as- cast alloy and nearly same mechanical properties to conventional T6 heat treatments. Chen et al., 2018 said that their result revealed new heat treatment method or this short heat treatments process are able to minimizing the growth rate of eutectic silicon and allow the formation of fully spheroidization of eutectic silicon.

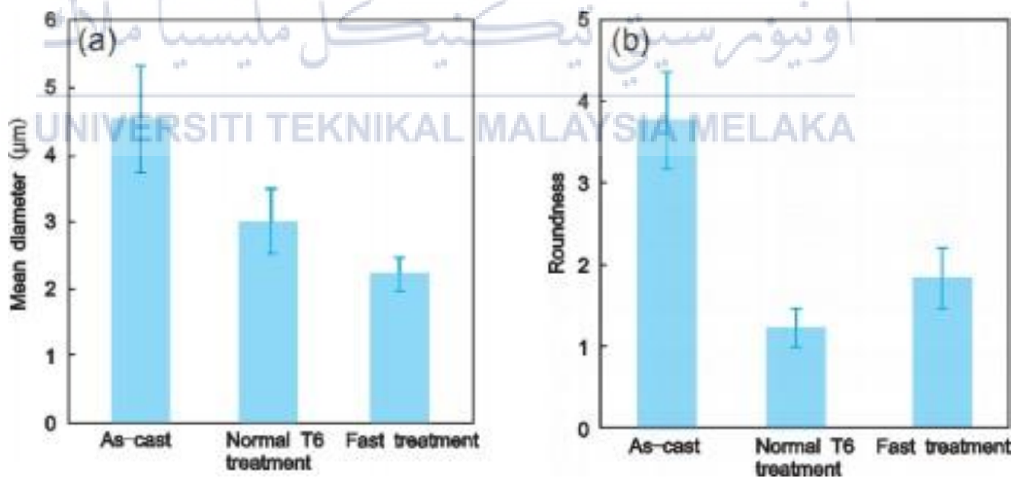


Figure 2.11: Comparison of average values of (a) mean diameters and (b) roughness for As-cast and T6 Treatments (Lu et al., 2018).

2.5 Aluminium and A319 Aluminium Alloy

2.5.1 Aluminium

Aluminium is soft, lightweight, fire-proof and heat-resistant, able to conduct electricity and easy to work into new shapes. It reflects light and heat very effectively and does not rust. The characteristics properties of aluminium, high strength stiffness to weight ratio, good formability, good corrosion resistance and recycling potential make it the idea candidate to replace heavier materials such as steel and cooper. Aluminum has a density of 2.7% g/cm^3, which is about one-third mass of steel, copper, and brass. Hence, aluminium usage especially in automotive applications has grown more than 80% in the past 5 years. Aluminium penetration has been limited up to now due to few factors such as raw material cost, manufacturing cost, industrial structure, recycling, and regulation (Miller et al., 2000).

2.5.2 A319 Aluminium Alloy

In this paper, A319 aluminium alloy will act as matrix to produce MMCs that later will be reinforced by using powdered graphene. Alloy A319s with the nominal composition of Al- 60%Si-3.0%Cu-0.35 %Mg. is a primary version of low-cost foundry alloy A319. The mechanical properties of semi-solid cast 319 -T6 are better than permanent mold cast as the T6 alloy exhibits mechanical properties that cannot be achieved by another foundry alloy. Alloy 319 may provide niche for semi-solid casting (Midson et al., 2014).

Process	Alloy	Temper	0.2% Yield Strength (MPa)	UTS (MPa)	Elongation
Rheocast	319s	T6	340	400	8
Permanent Mold	319	T6	165	250	2

Table 2.3 (a): Mechanical property data for alloy 319 and 319s

According to the, the distinctive alloying element in A319 alloy is silicon, copper, magnesium, manganese, zinc, and silicon with different weight percentage (wt.%) as compared to A319s stated above.

Si	Cu	Fe	Mg	Zn	Mn	Cr	Al
6.15	2.12	0.72	0.44	0.4	0.13	0.05	Remaining

Table 2.4 (b): Chemical composition of aluminium alloy A319 (Salleh et al., 2014).

2.5.3 Mechanical Properties

We can determine mechanical properties of material by doing some testing such as hardness testing. Mechanical properties are crucial role in determining any related processes such as fabrication process as well as their application in specific industries. In this study alloy A319 is chosen as the matrix to produce reinforced billet as it was low-cost production and lab- friendly. A319 also commonly used in automotive industry due to their good fluidity and mechanical strength.

Alloy hardness of A319	Improved from 104.2 ± 2.7 to 124.2 ± 3.2
Ultimate tensile strength	241 ± 3.4 MPa to 298 ± 3.0 MPa
Yield strength	176 ± 3.3 MPa to 201 ± 2.6 MPa
Elongation to fracture	(3.2 ± 0.5) % (4.5±0.3) %

Table 2.5: Summary of Mechanical Properties of A319 Alloy (Salleh et al., 2014).

2.5. 4 Comparison of Al319 with Al356

Al-Si-Cu alloy or 8.1wt% in A319 that contain higher Si element than in Al- Si alloy or 7.15 to 7. wt.% in 356 or A356, will increase the dendrite growing phase because the amount of solute that interferes with dendrite also increase help in producing finer microstructure in the former alloy. Otherwise, A356 mix with 0.5 Cu will produce finer secondary dendrites compared to all three Al-Si alloys as the additional in A356 is possible to have stronger refining effect amongst all Al – Si alloys (Roy et al., 2017).

Alloy-Condition	Si	Cu	Fe	Mn	Mg	Zn	Ti	Al (Balance)
206	0.17	5	0.15	0.25	0.32	0.005	0.021	93.88
319	8.3	3.17	0.68	0.39	0.34	0.31	0.1	86.62
356	7.21	0.14	0.39	0.25	0.37	0.17	0.19	91.28
A356	7.32	0.01	0.18	0.06	0.4	0.005	0.16	91.87
A356+0.5Cu	7.46	0.44	0.14	0.08	0.34	0.01	0.16	91.37

Table 2.6 : Detailed composition in wt% of various alloys majored in Si and Cu elements (Roy et al., 2017)

While, the A319 containing Mg element with same concentration in A356 alloy showed higher hardness values than in 356 alloys regardless their modification condition at all aging times and temperatures (Tash et al., 2007). This is due to Al_2Cu and Mg_2Si cooperative precipitation that increased due to increasing Mg contents than only Mg_2Si precipitation in case of A356 alloys that do not associate to hardness profile with Mg contents.

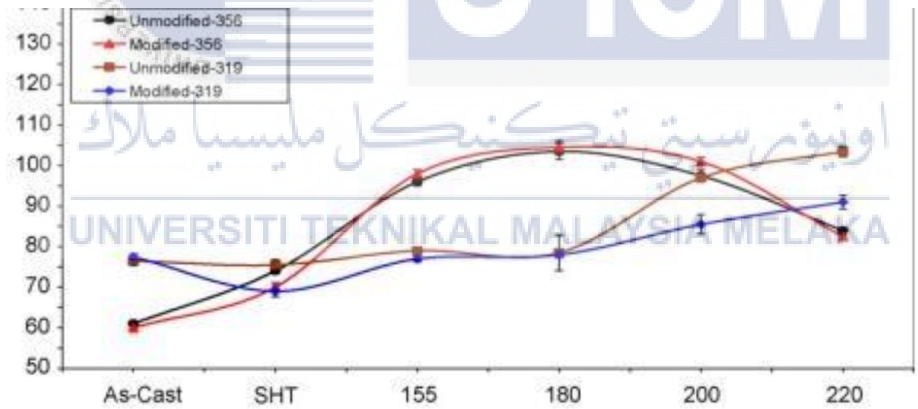


Figure 2.12: Graph of Hardness, HB Against Aging Temperature C (Tash et al., 2007)

2.6 Carbon Nanotubes

Author of books from Royal Society of Chemistry, Michael Berger said that CNTs made up of single -layer of graphene which are carbon atoms. Historically, CNTs were discovered by Sumio Iijima in 1991 when he finds out hollow and nanometer – size tubes of graphitic carbon either one of allotropes of carbon referred as single - wall carbon nanotubes (SWCNTs) Or bound weakly together by Van Der Waals interactions formed shaped in tree ring – like structure referred as multi – wall carbon nanotubes (MWCNTs). CNTs can be considered as flexible element that suitable to use as an additive material and they also can be said to be among the stiffest axial fibers that existed today due to their axial properties proposed by rolled – up graphenes with the in – plane properties (Kinloch et al., 2018).

2.6.1 Nature and Types of CNTs

CNTs can be build by coiling up covalent graphene building units with two-dimensional allotrope of carbon bonded by sp^2 orbital into hexagonal two- dimensional, 2D crystal lattice resulting in three - dimensional, 3D CNTs structure from geometric perspective (Sheshmani et al., 2013).

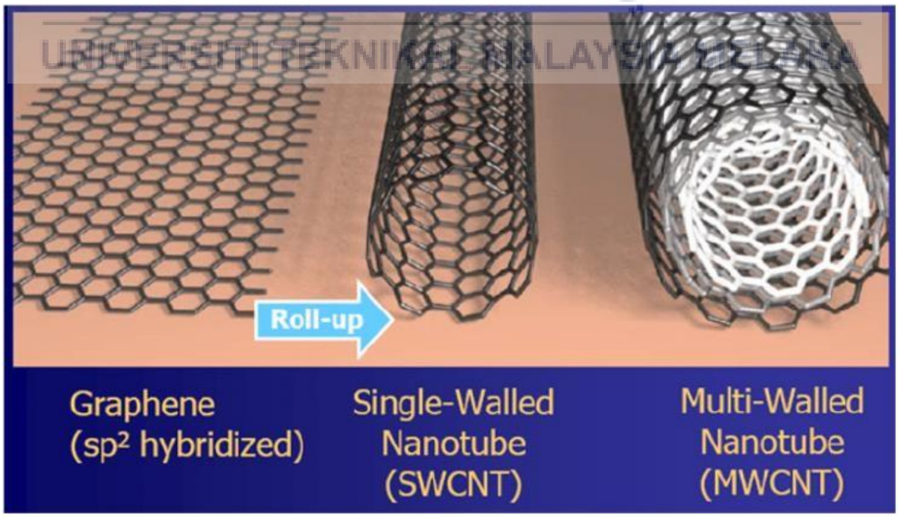


Figure 2.13: Schematic Diagram How Cnts Can Be Produce By Graphene Ideally (Hammond Et Al., 2016).

2.6.1.1 Single Walled CNTs

As mentioned, hexagonal shaped planar carbon atoms can start from any angels producing different Single Walled CNTs (Figure 2.14) with different forms or chiralities like Armchair, Chiral and Zig -Zag types (Shi et al., 2015). With diameter ranges from 0.4 to 3 nm with simple geometry the structured SWCNTs can be determined by chiral vector denoted n and m, armchair involved n=m, Zig Zag can have m = 0 and interestingly chiral have not specific chiral notation (Rafique et al., 2015).

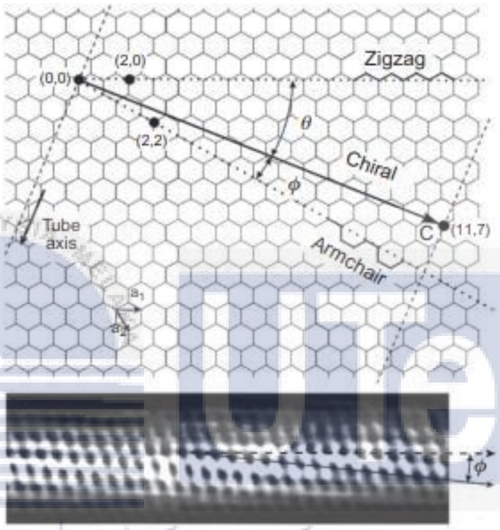


Figure 2.14: Curling Diagram of CNTs (Shi et al., 2015).

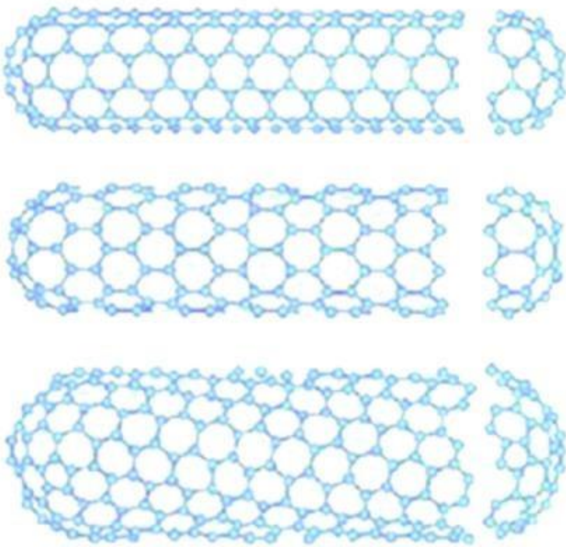


Figure 2.15: CNTs Types of Forms (N. Saifuddin et al., 2013)

2.6.1.2 Double Walled CNTs

DWCNT is made up of two concentric cylinders typically from double graphene rolled together known as special type of MWCNTs (Rafique et al., 2015). DWCNTs show several properties from both SWCNTs and MWCNTs making small gap between this two CNTs. Outer wall of DWCNTs can be optimized without effecting any properties mechanically or electrochemically when covalently functionalized to increase mechanical stability that will higher than SWCNTs but still small in diameter, length and the bundles ability that same as SWCNTs (Maryam Khan and Qayyum Husain , 2020). Due to their formation that have being synthetically blend together with MWCNTs and SWCNTs, these CNTs showed superior electrical and superior thermal stability and flexibility that widely used in sensors equipments, dielectric, as well as field – emission display and nanocomposite materials (Bhatt et al., 2016).

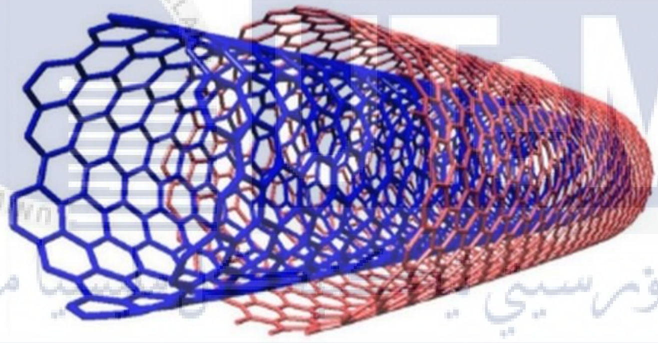


Figure 2.16: Double Walled Carbon Nanotubes (https://nanografi.com/blog/double-walled-carbon-nanotubes-dwcnts/.)

2.6.1.3 Multi Walled Carbon Nanotubes

Bhatt et al., 2016 claim that MWCNT is a tube structured elements that made up of several layers of graphite wrapped around one after another simply as rolled up newspaper from single sheet of graphite. Systematically the sizes and shape of MWCNTs can be altered as well as the modification of their surfaces proposing three types of MWCNTs which is cylindrical, polygonal and spiral ganglion (Aneta Ostróżka-Cieślik and Beata Sarecka – Hujar, 2017).

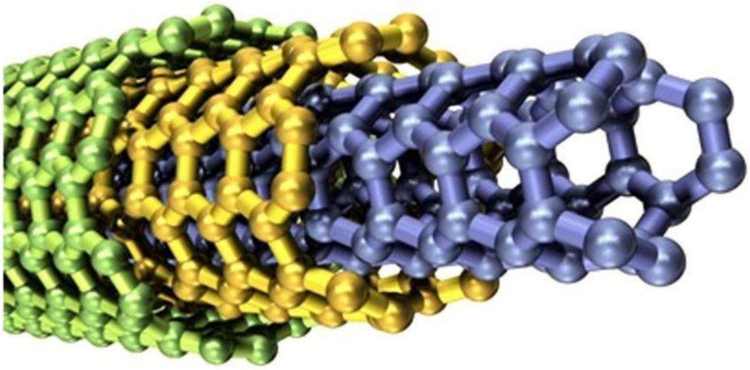


Figure 2.17: Schematic illustration of MWCNTs (Aman et al., 2015).

2.6.2 Processing Techniques for CNTs

The right processing technique will effectively be achieved outstanding application of CNTs and overcoming four main synthesis challenges such as mass- production scale, selection of right scale to control defects and their properties, organization level that control the location and orientation of CNTs towards produced nanotubes on specific substrates and lastly mechanism level showed all growing phases of nanotubes in synthesis processes (Ikram et al., 2020.). There are three main synthesis method available for CNTs such as Arc Discharge Method, Laser Ablation Method and Chemical Vapor Deposition Method while other methods such as ball milling, silane solution method, flame synthesis method and bottom-up organic approach. Table shows summary of three main methods of synthesis of carbon nanotubes.

Method	**Chemical vapor deposition**	**Arc discharge**	**Laser ablation**
Condition	Low pressure inter gas (argon)	Argon or nitrogen gas at 500 Torr	High temperatures about 500–1000 °C at high energy laser beam
Yield	High (60–90%)	Low (20–100%)	Low (up to 70%)
Purity	Medium to high	Medium	Low
Temperature	500–1200°C	~4000°C	25–1000°C
Product	SWCNTs: long tubes with diameters ranging from 0.6 to 4 nm	SWCNTs: short tubes with diameters of 0.6–1.4 nm	SWCNTs: long bundles of tubes (5–20 μm) with individual diameter from 1 to 2 nm
	MWCNTs: long tubes with diameter ranging from 10 to 240 nm	MWCNTs: short tubes with inner diameter of 1–3 nm and outer diameter of approximately 10 nm	MWCNTs: not very much interest in this technique

Table 2.7 : Summary and comparison of CVD, Arc Discharge and Laser Ablation Methods (Wang et al., 2019).

2.6.2.1 Arc Discharge Method

The most popular and commonly used method in producing CNTs is Arc Discharge Method consist of an anode and cathode of graphite inside a steel chamber, containing inert gas at high pressure around 3000°C – 4000°C that will vaporizes forming MWCNTs, carbon and some graphite sheets (Slabei et al., 2017). They also said post synthesis treatment is important for purification of CNTs making this method more costly that required two graphite electrodes and inert gases that must have high purity.

2.6.2.2 Chemical Vapor Deposition Method

The most effective and easy method to synthesis CNTs is CVD as it was so simple in preparing the equipments use, simple operation and economic friendly. This method only needs low pressure of argon (inter) gas with a temperature range between 500 °C to 1200 °C. In term of yields and purity of product, CVD process can selectively remove amorphous carbon without damaging the nanotubes while improving the life and activity of the catalyst by added water vapor, oxygen in the air and an ethanol (Wang et al., 2019).

2.6.2.3 Laser Ablation Method

This method can be said to be as same as Arc Discharge Method due to their principles and same mechanism with high demand of power supply as well as laser cost. A shaped like mat of few ropes each ropes contained of numbers of single- walled nanotubes with diameter of 10 – 220 nm and 100micro meter or more in length (Cheap Tubes Inc., 2019). This process required temperature 1200°C by using high power laser with controlled argon gas used to incorporate with catalysts (Ikram et al., 2020).

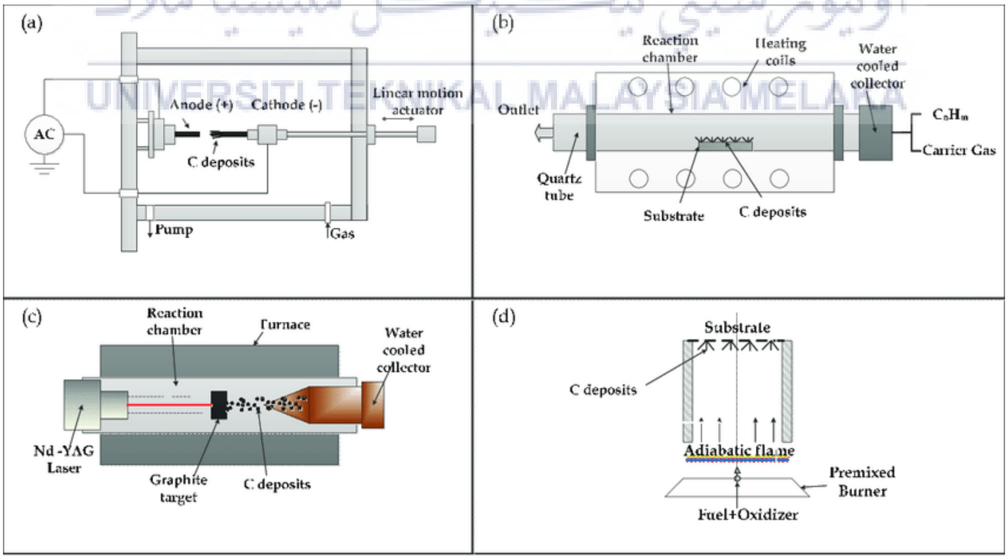


Figure 2.18 : Schematic Diagram of (a) Arc Discharge; (b) Chemical Vapor Deposition ; (c) Laser Ablation and (d) Hydro Carbon Flames (Jay P. Gore and Anup Sane 2011).

2.6.3 Properties of CNTs

2.6.3.1 Thermal Properties

Due to their unique size and structure, thermal properties of CNTs are large at room temperature even when the SWNTs were aligned in bulky samples. Bhatt et al., 2016 said that CNTs exceed thermal conductivity of the best thermal conductor such as diamond. Many research done showed that CNTs is the best thermal reinforcing materials for polymers due to their extremely high thermal conductivity,3000 W/mK when using Nanocyl 3100 MWCNTs because CNT acts as a bridge which phonons pass through the resin without losing energy (Trakakis et al., 2020).

2.6.3.2 Electrical Properties

Pure CNTs have high range of electrical conductivity as 10^6 to 10^7 due to their microstructure of CNTs that all atoms are arranged in unchanged honeycomb grid sheet of carbon atoms. CNT can be the best choice of reinforcements materials for nanocomposites to increase electrical conductivity at very low filler loading with high property contrast and extreme aspect ratio. Actually, free electron that is mobile at an outer layer provide an ability for electric conduction (Wang et al., 2017). Other research stated that the chirality that denoted by n and m can conduct electricity and known as metallic. Bhat et al., 2016 emphasis that metallic is 1000 times better than copper in term of conductivity, hence we can say that CNTs with armchair structured type is better conductor compared to other metallic CNTs because of their complex structured proposed by MWCNTs.

2.6.4 Mechanical Properties of CNTs

2.6.4.1 Tensile Strength

Theoretically, Akira Takakura et al., 2019 found that chiral angel and diameter of nanotubes have clear relation on the strength of the elements by considering directions of carbon- carbon bonds against the direction of stress concentration and tensile loads. Predicted by many researches CNTs are the strongest material with tensile strength of 11 to 63 GPa due to their sp^2 covalent bonds bonded between single carbon atom. Besides that, CNTs re- aggregation, incomplete CNTs wetting as they are poorly dispersion and viscosity building issues make tensile strength differ from modulus as tensile strength does not increase monotonically (Khan et al., 2016).

With 100 GPa of ultimate tensile strength, cylindrical rolled graphene sheets of Single Walled Carbon Nanotubes have being predicted as game changing structural materials. The highest stress that measured before fractured indicate the ultimate tensile strengths is 50 GPa (fracture strain of nanotubes) suggesting the extrinsic factors are dominant reasons of the fracture (Akira Takakura et al., 2019).

2.6.4.2 Young Modulus

Previous studies either by using theoretical studies or using molecule dynamics, MD showed that the elasticity of CNTs can be influenced by the radius size of the CNTs. Young's Modulus is a function of tube radius and helicity and not a simple material property as they can varies according to their geometries resulting in torsional strain that increase significantly with decreasing tube diameter but somehow increase when the tube helicity decreases. The fact can be supported by Ghasempour et al., 2018, when they stated that due to coaxial intertube coupling or Van Der Waals forces in MWCNT, MWCNT showed highest Young's Modulus with 1.1 to 1.3 TPa compared to SWCNTs that were nearly to 1 TPa with diameters between 1 and 2 nm. Table shows different mechanical properties in SWCNT and MWCNT. With the Young's Modulus greater than 1 TPa the element was considered approximately 5 times higher that steel (stainless steel) (Ikram et al., 2020)

Materials	Young's Modulus (GPa)	Tensile Strength (GPa)	Density (g/cm^3)
MWCNT	1200	~150	2.6
SWCNT	1054	~150	1.3
Graphite (in-plain)	350	2.5	2.6
Steel	208	0.4	7.8
Wood	16	0.08	0.6

Table 2.8: Mechanical Properties of Different Engineering Materials (Ghasempour, R., & Narei, H.,2018).

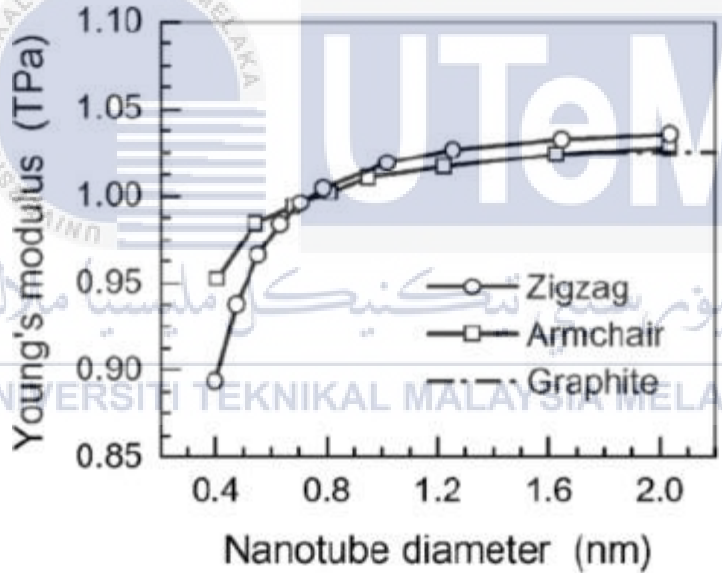


Figure 2.19 : Young's Modulus of SWCNTs With Different Diameters (Lei et al., 2021)

2.7 Microstructural Morphology of Rheocast A319 Aluminium alloy.

After semi-solid metal processing, spheroidal microstructure enables easy movement in primary phase of a- Al when an external load is exerted compared to dendritic microstructure. This is due to microstructure characterized by interlocking between the grains, this resulting in material resistant to movement (Koeune et al., 2011). SSM processing can change microstructure of metallic alloy to becomes spheroidal rather than dendritic.

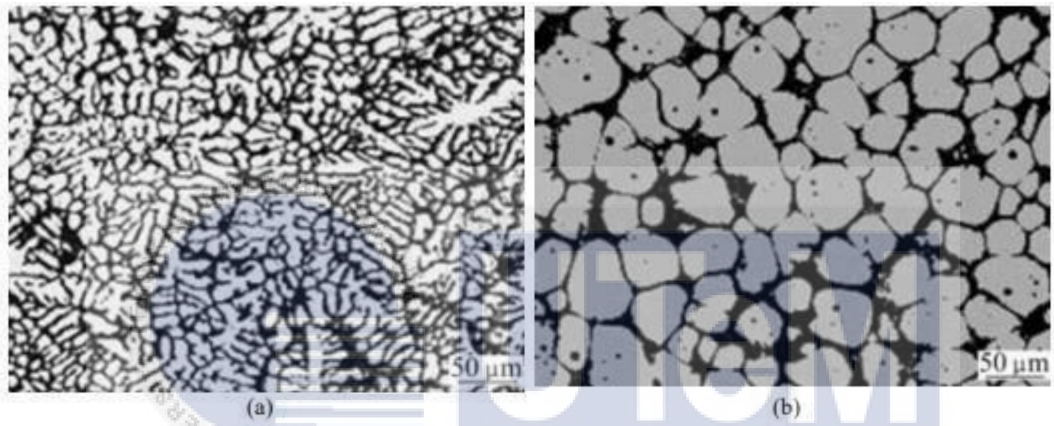


Figure 2.20: Differences of (a) Dendritic microstructure and (b) globular microstructure (Salleh et al., 2016)

2.7.1 The rheology of aluminium alloy in cooling slope processing

Salleh et al., (2014) pointed out three parameters which is angle (°), pouring temperature(°C) and length (mm) that were based on the results of optimization study conducted.

Aziz et al., (2016) state that there are four impact zone which different microstructure of A319 on CS plate. They divide the zone into four stages which is dendritic growth stage, rosette, ripened rosette and nearly globular. This phenomenon of microstructural evolution is affected by shearing process of molten alloy that occurs along CS plate. Evolution of the microstructure can be explained better in schematic illustration provided by Kirkwood (1994).

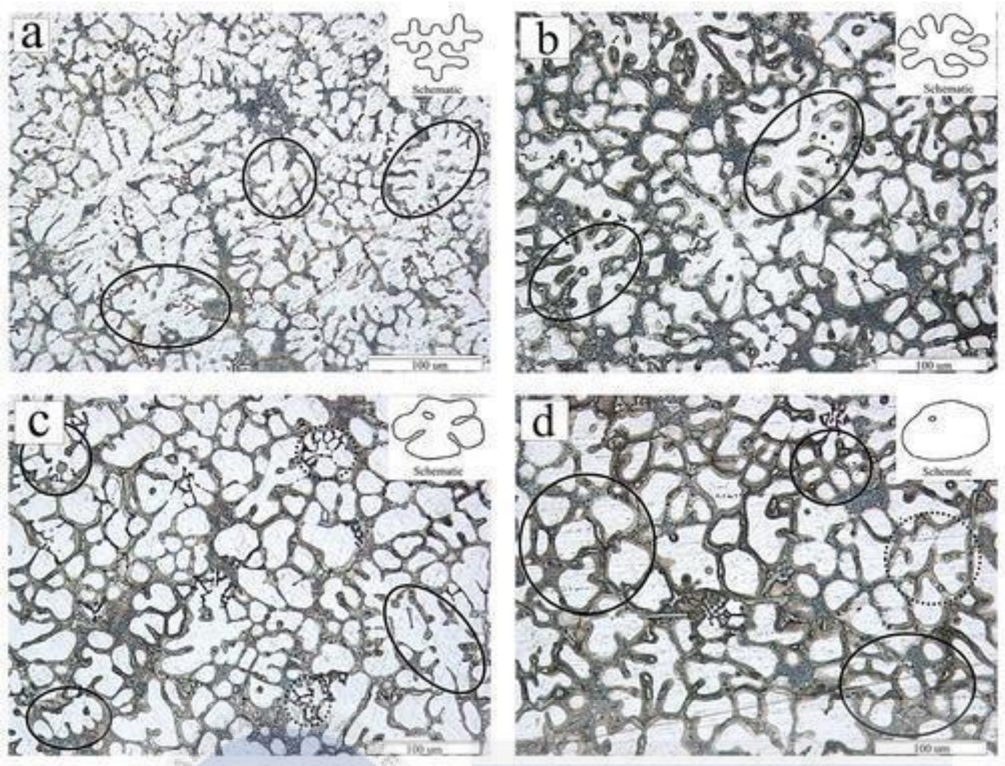


Figure 2.21: (a) impact zone, (b) top zone, (c) middle zone and (d) bottom zone for microstructure of A319 (Aziz et al., 2016)

Changing in the microstructure of alloy from impact zone to top zone to middle zone and until bottom zone (dendritic growth stage to rosette-like shapes to ripened rosette stage until nearly spheroidal microstructure) is the result of shear force acting between molten alloy and the CS plate. Besides that, microstructure of A319 alloy produced in CS mold contribute to the evolution of spheroidal microstructure (Robert et al., 2007). Salleh et al., 2014 said that there is slight improvement in globularity of microstructure in A319 alluminium alloy as compared to microstructure produced in mold with bottom zone of CS plate as shown in the Figure 2.22.

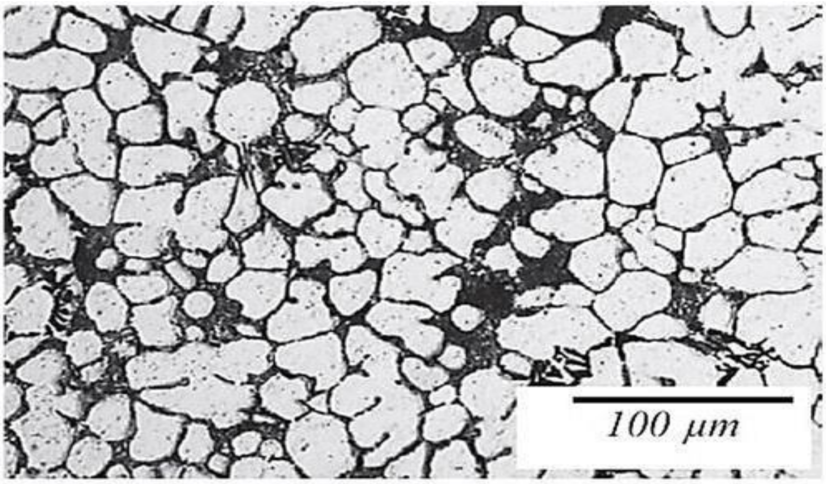


Figure 2.22: Microstructure of A319 aluminium alloy after CS casting process (Salleh, 2014).

2.7.2 Mechanical Properties

Burapa et al., (2010) said that mechanical properties can be affected by the grain size and shape factor of the primary a-Al. The hardness of material of microstructure of a-Al increased progressively and becoming more spheroidal compared to as- cast sample as well as the increment in hardness where 12.2 % increment occurred in CS casting process as compared to as-cast A319. Salleh et al., (2014) summarized the range of hardness for A319 aluminium alloy in four positions as stated above with as- cast hardness as shown in Figure 2. 23.

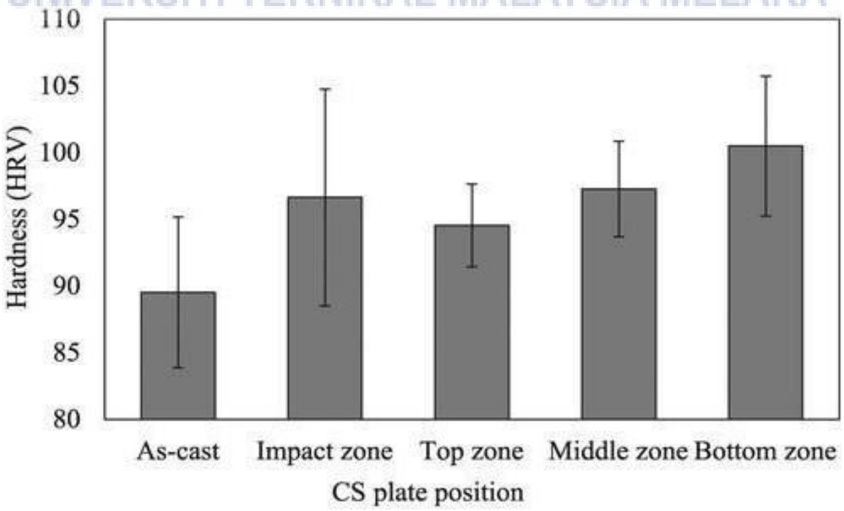


Figure 2.23: Summary of as- cast hardness with hardness of A319 aluminium alloy in four positions in CS plate (Aziz et al., 2016).

2.8 Aluminium – CNTs Reinforced Alloy.

2.8.1 Effect of CNTs in aluminium alloy.

There are a lot of advantages of CNTs/Al alloy nanocomposite as CNTs can be used widely as a reinforcement agent without compromising the characteristics of the metallic elements. As we can see, the addition of CNTs in bimetallic Al-Mg yield better tensile strength with maximum increase of 129% when 5 vol% of CNTs being addded and enhanced the mechanical properties such as elastic modulus, UTS and the failure strain as compared to pure bimetallic (Md. Hasan Ali and Robiul Islam Rubel, 2020).

2.8.2 Thixoformed A319- MWCNTs Composites

In T6 heat treatment highest hardness value obtained by the A319 composite alloy with 0.75wt.% CNT is 94.6HV and for tensile strength the increasing wt.% in CNTs will increase the tensile strength significantly which is optimumly at 0.75 wt.% CNTs. This can be explained by using the evolution of microstructure. Due to lower porosity and formation of new spheroidize shapes in thixoformed samples the grain size become smaller that effected the mechanical properties to increase the tensile and hardness values (Rahman et al., 2020).

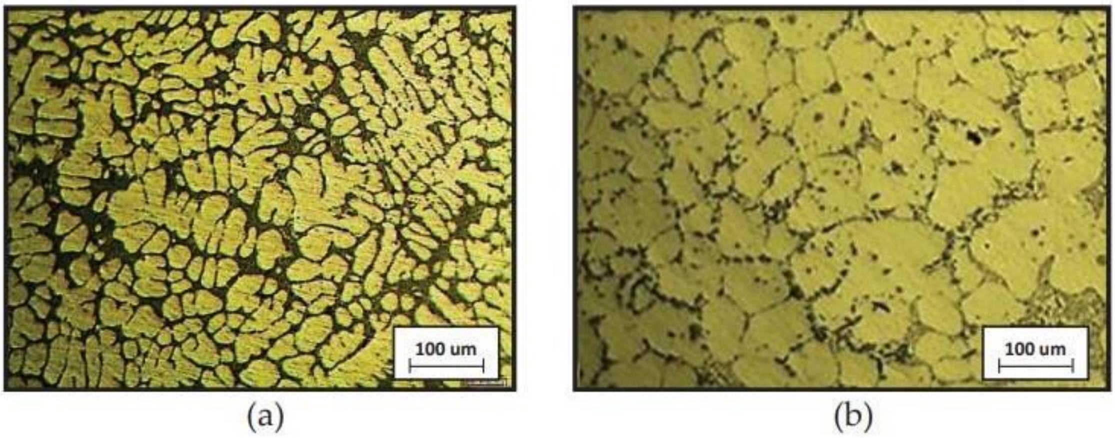


Figure 2.24: Microstructure of (a) as- cast of A319 aluminium alloy and (b) Thixoformed A319- MWCNTs Composite Alloy (rahman et al., 2020).

While the aluminium A319 alloy with Al, 5.5 wt. % Si and 3wt.% Cu showed 36% increase in hardness after subjected to T6 heat treatment due to precipitation of metastable of Al_2Cu in aging process compared to in as-cast (Magno et al., 2017).

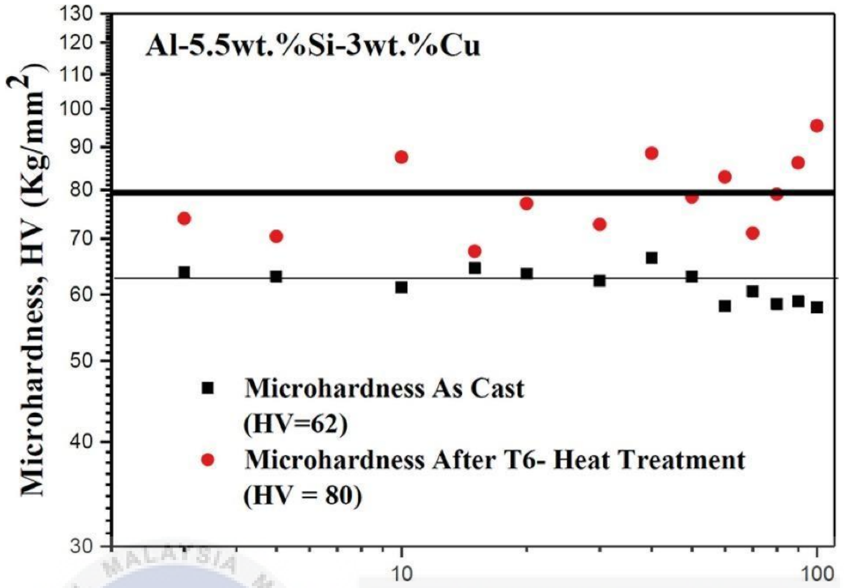


Figure 2.25: Microhardness after as- cast and after T6 Heat Treatment (Magno et al., 2017).

2.8.3 Al-MWCNTs In Short Heat Treatment

The graph provided by Tillová et al., 2018, shows the effect of solution treatment on Si-particle size in conventional heat treatment. We can conclude that at early first 2 hours in solution treatment at 530° C the average diameter of silicon particles increased swiftly but slowly increased after 3 hours.

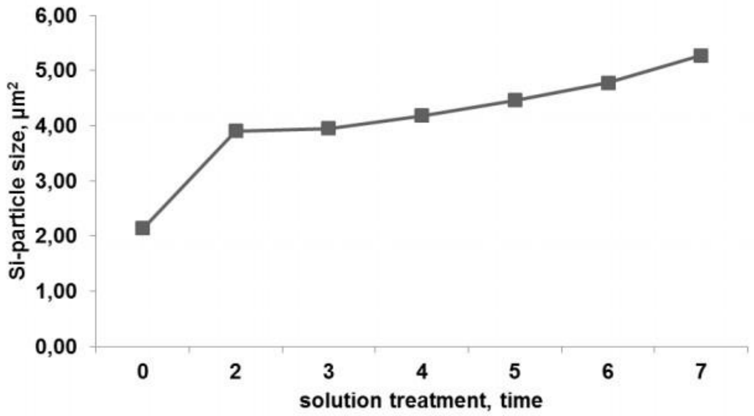


Figure 2.26: Graph of the effect of solution treatment in times on Si particle size (Tillová et al., 2018).

On the other perspective, the inter-diffusion of Si-Al particles need only 150 kcal/mol which is minimum activation energy. As compared to the dissolution of Si into Al matrix, the evolution and growth of Si particles is more sensitive and faster as the diffusion of Si-Si particles required activation energy only around 80 kcal/mol. A356 alloy at 500 °C after 3 minutes will dissolve the Si particles and the short period to homogenize at grain boundaries of the metal matrix was insufficient because the distribution of phases, composition, size and morphology after solidification can influence the solution time needed. Mg dissolution and transformation of π-AlSiMgFe phase into β-AlFeSi phase can be obtained in semisolid A356 alloy with only 30 minutes solution time at 540 °C -550 °C and considered as safe solution time , but there are still some plate-like morphology of Si particles. Hence, by adding MWCNTs in the al- matrix, the duration of 1 hour of solid solution treatment is enough to create integration between reinforced particles and soluble phase (Hanizam et al., 2019). Microstructure image of A356 after the 1 hour solution treatment in short T6 heat treatment indicate that eutectic Si particles evolved into spherical shape to strengthen and prevent micro-fracture in the composite alloy (Hashim et al., 2021).

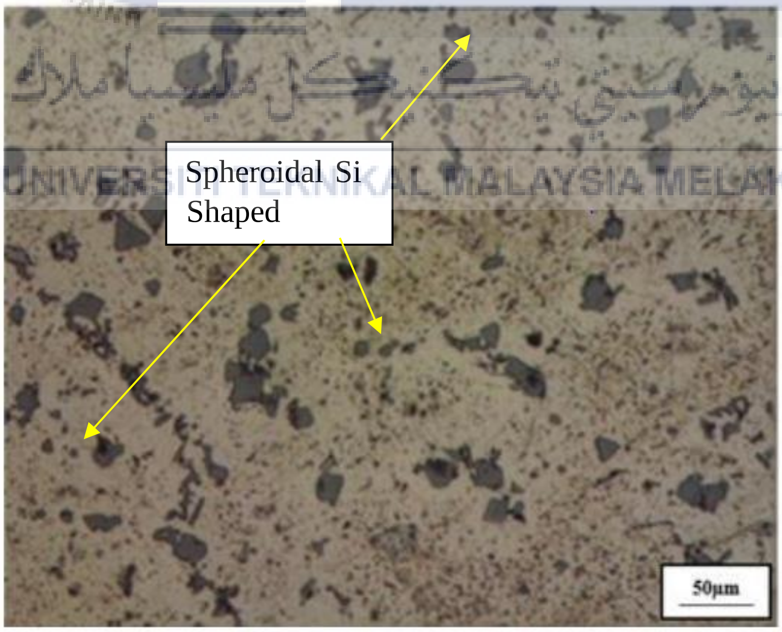


Figure 2.27: Microstructure of Thixoformed A356 – MWCNTs Composite after Short T6 Heat Treatment (Hashim et al., 2021).

Chapter 3

Methodology

Research methodology is a systematic way to solve problem. The procedures by which researchers go about their work of describing, explaining and predicting to give plan research. In this chapter we will cover how this project develop from preparing A319 aluminium composite reinforced by graphene. This chapter also will help us to get brief explanation on how we can achieve our objectives as stated in Chapter 1 and also obtain the desired result from our experiment.

3.1 Gantt Chart

Gantt chart is one of the most useful project management tools that illustrate a project timeline and progress in a visualized bar chart. By using Gantt Chart, we can ensure, or project will be performed within prescribe time and help us to keep the project on track. Basically, I create Gantt Chart based on start date and end date of my task as well as the duration on how long the task will take. Hence, for Final Year Project 1 and 2, there are several activities prepared and conducted for final year students including our report that include Chapter 1 until Chapter 5. All activities listed out by JK PSM have duration time in order to make students more organized and the purpose of PSM can be achieved in the set time for Final Year Project 1. In Final Year Project 2, there were basically based on practical and laboratory works such as preparing samples and analysis data provided by experiment that took place through the semester two. After that, a brief explanation will be presented in form of report and being evaluate by responsible lectures.

3.2 Design of Experiment.

Design of experiment or DOE is a branch of applied statistics that deals with planning, conducting and analyzing as well as interpreting-controlled test. In this experiment DOE is the best tool evaluate factors that control values of a parameter or a group of parameters. DOE is a powerful data collection and analysis tool that people often used in a variety of experimental situation. In DOE, we can choose methods that suitable for our experiment such as Taguchi method/ fractional method, full factorial method, or response surface methodology.

3.2.1 Using Taguchi Method To Identify Parameters.

For this experiment, we used Taguchi Method since in this experiment there were two parameters that will help us to conduct this experiment and achieved desired goals while reducing variation also known as Robust Design. Parameters involved in this experiment include CNTs contents, wt.% and stirring time (minutes). These two parameters have three different values that make run order to have nine readings samples.

Levels / Factors	Low/ Pure	Medium	High
Amount of CNTs contents, %	0%	0.25%	0.5%
Stirring Time, Min	5	10	15
Constant Heat Treatment	T6	T6	T6

Table 3.1: Three levels with two factors and constant T6 heat treatment.

Run Order	CNTs Contents, (wt.%)	Stirring Time (Minutes)	Heat Treatment	Hardness (HV)	Tensile Strength
1	0	5	T6		
2	0.25	5	T6		
3	0.50	5	T6		
4	0	10	T6		
5	0.25	10	T6		
6	0.50	10	T6		
7	0	15	T6		
8	0.25	15	T6		
9	0.50	15	T6		

Table 3.2: DOE, Taguchi Method with nine run samples (3 x 3).

3.3 Summary of experiment in Flow Chart

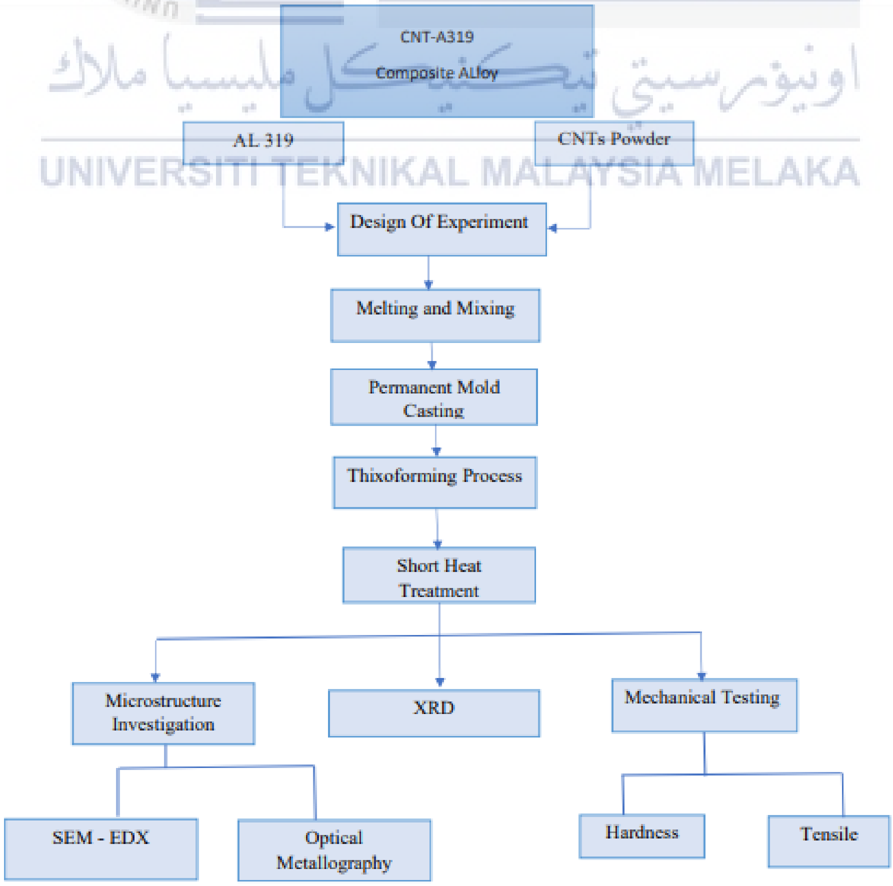


Figure 3.1: Flow Chart of Experimental Works

3.4 Experimental Procedures

3.4.1 CNTs Synthesis

There is small amount of material needed from plenty resources that available today which can be used to produce CNTs. Synthesis of carbon nanotubes, CNTs is important as we desired only a few wt.% of CNTs contents.

CNTs acts as reinforcement agent can be synthesized by Hummer's method and chemically reduced by using hydrazine hydrate at a temperature of 95 °C for 24H.

3.4.2 Metal Matrix Preparation

In this experiment aluminium A319 will be used as metal matrix and was provided by FKP that soon will be reinforced with CNTs powders. By using X-ray fluorescence (XRF) technique to determine chemical composition of A319. Table listed chemical composition of A319 alloy data generated by XRF. The sample will then be used in differential scanning calorimetry (DSC) to estimate pouring temperature, solidus and liquidus temperature as well as liquid fraction profile within semisolid transition range.

By using bandsaw machine that was available in FKP laboratory (Figure 3.2), A319 aluminium alloy ingots is cut into thin pieces with thickness of 10mm or approximately of 30 mg. Small pieces of A319 alloy is used to increase melting availability in the furnace, as the sample was pre-heated in nitrogen to prevent oxidation.

Element	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al
Wt.%	6.26	0.53	2.91	0.19	0.30	0.03	0.06	0.71	0.03	Remaining

Table 3.3: A319 Composition of A319

Figure 3.2: Bandsaw Machine from FKP Laboratory

3.4.3 Nanocomposite Preparation and Casting Process

The CNTs powders about 2 g were wrapped in an aluminium foil that contain small percent of magnesium, Mg to increase wettability and being mixed together with molten A319 alloy. At first, small pieces of A319 alloy with approximately 400g will be melted in an induction furnace in FKP Laboratory with controlled temperature among 700°C to 650°C until all the solid metal changes into liquid or fully melted. We used K-type Thermocouple to control temperature (Figure3.4). The temperatures were fix based on suitable temperature data collected by DSC test. At temperature constant of 650°C, wrapped MWCNTs will be injected into the bottom of crucible by using a plunger before being constantly stir with three blade impellers at 500 rpm which is medium speed to avoid air entrapment that may cause high porosity in the composite end products. The steps will be repeated for nine times to produces nine samples according to DOE table stated above with stirring time of 5 minutes, 10 minutes and 15 minutes as well as the different contents of CNTs of 0%, 0.25% and 0.50%. Lastly, the feedstock will be poured in cylindrical stainless-steel mold at preheated temperature of 150°C with diameter of 23 mm and 120 mm height (Figure 3.6). This size and shape are the optimum for next thixoforming process. Cooling slope casting (Figure 3.5) was used for this experiment as it can control atmosphere by the present of argon gas. The angle of cooling slope is set to 60° through the experiment to reduce adhesion of solidified alloy. To produce less dendritic microstructure, the cooling slope plate was cooled under run water, increasing α- Al particles. Permanent mold casting or as- cast sample with temperature of 700°C was poured into the same mold preheated to 150 °C will produce accumulation of MWCNT on the top side of billet due to rolling effect in cooling slope.

Figure 3.3: Casting Process

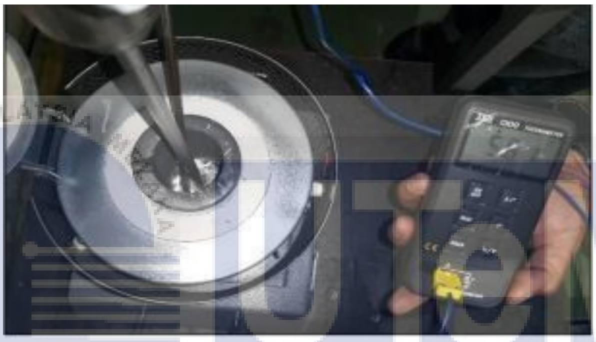


Figure 3.4: K- Type Thermocouple

Figure 3.5: Cooling Slope Casting Apparatus (Salleh et al., 2014)

Figure 3.6: Stainless Steel Mould

Figure 3.7: Feedstock Billets (Samples)

3.4.4 Thoxoforming Process

This process (Figure 3.8) is widely used for forming alloys in a semisolid state near net shaped end product. After the preparation of the feedstock, the feedstock must be re-heated to semisolid state to provide SSM slurry subsequently used for shaping. The obtained ingot from cooling slope casting process will then were sectioned into smaller size and re-heated to produce semisolid states before placed them into die (Figure 3.9). This is important as thixoforming technique requires fine and globular solid particles distributed in liquid matrix.

Figure 3.8: Thixoforming Machines at FTKMP

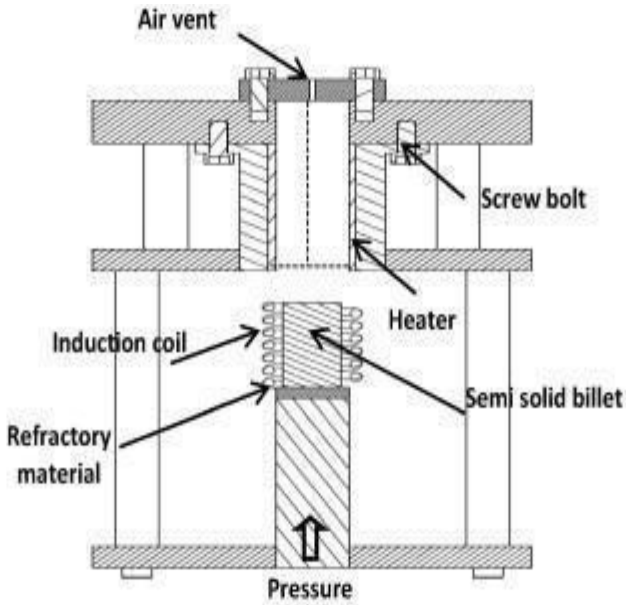


Figure 3.9: Die for Thixoforming (Salleh et al., 2014)

An induction coil with high frequency such as 30-80 kHz, 35 kW was used to heat the ingots into semisolid state in situ. By using K-type thermocouple, the heating temperature was controlled. The thermometer will be placed at 8 mm from the top of slug. Oxidation that can occurred in thixoforming process can be prevent by using 2.5 l/min Argon gas. Rapid heating at 130°C/min is applied to avoid undesirable grain growth but the ingot will be held at 571 °C for 5 minutes to produce spheroidization of the grains while pre-heated process at upper and lower dies were performed to 300 °C.

Thixoforming process was performed using hydraulic cylinder press after thermocouple was withdrawn from the sample. The hydraulic cylinder press provides forging load of 20 kN with maximum ram speed of 85 mm/s.

3.4.5 Short T6 Heat Treatment

Some thixoformed parts were allowed to short T6 heat treatment. This treatment involved short processing time as compared to conventional T6 heat treatment with shorter solution treatment that allows the samples to quench in warm water at 540 °C for approximately 1 hour, followed by artificial ageing for 2H at 180°C in a furnace that may withstand a high temperature between 30 °C to 3000°C. this furnace equipped with programmable temperature controller.

Samples from Thixoformed Short T6 heat treatment will lightly polish with silicon carbide paper and polishing cloths with diamond paste with properties of 3µm then followed by 1µm before being treated in etching process with Kellers solution for 10 seconds, 10s.

3.5 Hardness Test

After thixoformed and casting process, the samples were polished by using polishing cloth with diamond paste of 6µm, 3µm and 1µm. We are also grinded the samples using SiC grit paper with different size. Rockwell hardness tester is used to measure hardness by imposing

100 kg load with1/16’’ for 10 measurements. The average of at least 10 measurements were used to measure and record data for Vickers hardness values and will be represented in the value of HRB.

Figure 3.10: Rockwell Hardness Tester
from FKP Laboratory

3.6 Tensile Testing

Samples from casting process and thixoforming process were machined by using CNC Turning Machining to obtained dog-bone shape (as in Figure 3.11) tensile specimens with the size of 20mm in length according to ASTEM E8M standards. To obtained cylindrical sample according to ASTEM: E8M standard thixoformed and thixoformed T6 specimens were cut into small-size specimens of ratio 4:12.5 was used and were cut by using Wire Electrical Discharge Machining, Wire- cut EDM.

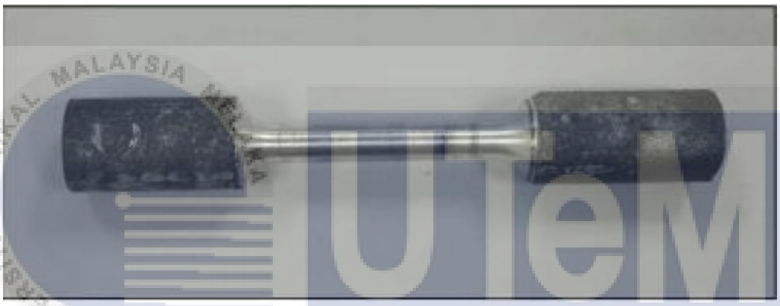
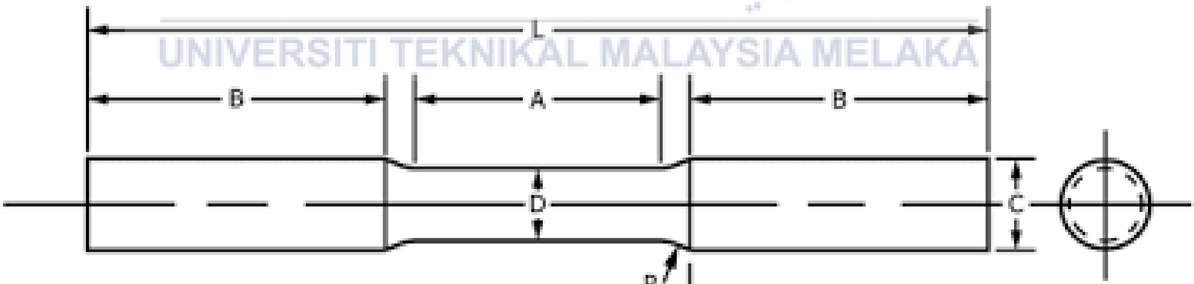


Figure 3.11: Dog-Bone Shape for Tensile Test



	Small-Size Specimens Proportional To Standard			
	9	6	4	2.5
G—Gage length	45.0± 0.1	30.0 ± 0.1	20.0± 0.1	12.5 ± 0.1
D—Diameter (Note 1)	9.0 ± 0.1	6.0 ± 0.1	4.0 ± 0.1	2.5 ± 0.1
R—Radius of fillet, min	8	6	4	2
A—Length of reduced section, min (Note 2)	54	36	24	20

Figure 3.12: ASTEM E8M standards

	Specimen 1	**Specimen 2**	**Specimen 3**	**Specimen 4**	**Specimen 5**
G-Gauge length, mm	62.5 ± 0.1	4.5 ± 0.1	30.0 ± 0.1	20.0 ± 0.1	12.5 ± 0.1
D-Diameter, mm	12.5 ± 0.2	9.0 ± 0.1	6.0 ± 0.1	4.0 ± 0.1	2.5 ± 0.1
R-Radius of fillet, min	10	8	6	4	2
A - Length of reduced section, min	75	54	36	24	20

Table 3.4: ASTM E8M Standard Tensile Test Specimen

Figure3.13: Wire Cut EDM Machine from FKP Laboratory

Figure 3.14: CNC Machine from FKP Laboratory

By using 100kN Universal Testing Machine (UTM) based on standard of ASTM E8M, a tensile test had been carried out in room temperature with constant crosshead speed of 5.0mm/min. total elongation values were measured until fracture as an extensometer was placed on the gauge length. After this test smaller section will be tested for microscopic observation.

Figure 3.15: Universal Tensile Machine

Figure 3.16: Tensile Test Sample
from FKP Laboratory.

3.7 Microstructure Structure

Samples from thixoforming method were cut into cubic samples with approximately 1.5cm^3 and uniformly distributed about the bar center for microstructural analysis. The samples can be prepared by successive polishing steps on manual wheel using silicon carbide papers, alumina powder, diamond suspension and colloidal silica. But in this experiment, we used simplest method such as grinded and polishing, at first the sample will grinded ascendingly by using silicon carbide grits number 240 up to 1200. Then the samples will then be polished by using polishing cloths together with abrasive diamond compound. This process will continue until the sample become mirror-like surface before immersed the samples into etchant. For final step, all the grinded and polished samples were etched in etchant usually Keller’s agent for 20s to eliminate impurity from the surface of the samples. This step will ensure microstructure investigation can be performed smoothly.

3.7.1 Optical Micrographs by using Optical Microscopy.

Optical microscopy was performed by using invented metallurgy microscope, insrumented with camera. By using Olympus optical microscope, optical micrographs were captured to analyze image by using commercially available Image-J software. Software that we used can performed calculation for shape factor (SF), and globule size (GS) of α-Al phase. Shape factor was defined by equation 3.1 below:

$$S= \frac{4\pi A}{p^2} \ldots \text{ Equation 3.1}$$

Where;

A= is area of the particle

S= Sphericity

P= is perimeter

Note: the shape factor of circle is equal to one.

While for average globule size of the primary particles can be determine by the equation 3.2 of:

$$\frac{\sum 2(Ai/\pi)^{1/2}}{N} \quad \text{… Equation 3.2}$$

Where;

A= Area of each particle

N= Total number of particles in each image performed

Note: The best shape factor and smallest globule size of -Al were selected for Thixoforming process.

3.7.2 X-Ray Diffraction Analysis (XRD)

XRD was performed to identify the phase evolution in this experiment. Before the sample can be examined, all the sample have to be polished as we are collecting data on crystal structure, phase, grain size and crystal defect of the samples. Hence, it is important to eliminate impurities.

This analysis gave data by measuring x-ray diffraction.

3.7.3 Scanning Electron Microscopy and Energy Dispersed X-ray (SEM EDX)

Phase microanalysis was conducted by using energy dispersed X- ray. Thixoform samples were identified by using Carl Zeiss (EvoMa10) scanning electron microscope (SEM) with energy dispersive X-ray (EDX) (d8 advance). By using EDX analysis we can reveal the composition of the globules. In this project, Backscatter SEM-EDX is used to investigate morphology and composition of the samples by investigate the variation of grey level produced by SEM Image. This testing method required gold sputter coating (coated using Mini sputter coater) and polishing step in providing the sample with an ultra-thin coating of electrically conducting metal. It is to improve the optical micrographs as well as secondary electron emission.

Figure 3.17: Mini Sputter Coater from FKP Laboratory

Figure 2.18: SEM- EDX Machine from FKP Laboratory

Chapter 4

Result and Discussion

In this chapter result will be presented and discusses focusing on microstructure evolution and mechanical properties on Thixoformed A319 alloy reinforced with CNTs after treated with T6 short heat treatment. By using DOE approached total of nine samples are prepared accordingly, but only a single sample with outstanding behaviors from each specific process will be used in testing to present a better and clear understanding of the finding in this experiment.

4.1 Microstructure Analysis

4.1.1 Optical Microstructure of Raw Material A319

Any were first being metallographically polishing by using 3μm properties of diamond paste on a cloth and using Keller's solution for etched process for 10 seconds. DIC mode is used to collect the microstructure of the A319. The figure shows that the microstructure of A319 consist of majorly α-Al dendritic arms (as marked below), Si- rich platelets, Al- Cu phases as well as intermetallic compounds in Fe. This primarily dendritic morphology, a characteristic tree like structure in A319 containing different intermetallic phases in the interdendritic regions showed homogeneous structure. As we can see the dendritic size in A319 is finer than in A356 which is ~46 to 47μm and ~60μm respectively.

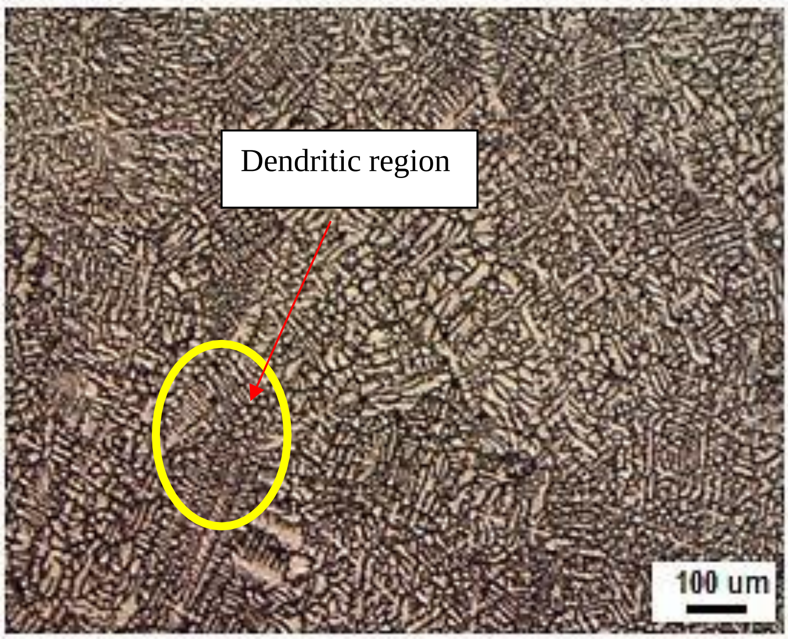


Figure 4.1: Microstructure of Raw material A319

4.1.2 Optical Microstructure of Feedstock Material After Cooling Slope Process

In cooling slope process, poured alloy coming into contact with cooling slope plate resulting rapid quenching that making the alloy solidified as it flows down the slope. The solidified sample was cutting into three main part namely as top zone, middle zone and bottom zone. Each zone will be shaped into three billet samples that were equal in size but in this experiment, we choose billet no.2 from each zone to be a sample as we could see the differences more clearly.

As for the top zone, we could see the dendritic structure as in figure start to change it shape into rosette growth stage. The change is due to the movement of the molten along the slope as well as temperature drops below liquidus temperature that encourage the evolution of a-Al to expand and nucleate their size producing a mixed globular microstructure that shaped like rosette. Due to the surface tension between the liquid and the matrix, a phenomenon called entrapment of liquid occur between dendrite arms resulting finer microstructure in interdendritic areas.

Next, as the molten flow down the slope, middle zone shows that some dendritic arms are broken because alloy disturbed the microstructure of rosette resulting in ripened rosette. The broken dendritic arms evolved into nearly spheroidal structures. Besides, by continuing shear time and by abrasion with other grains the rosette structure will face ripening phase before the ripening proceeds to spheroidal structure in further cooling.

Lastly, the bottom zone will appear as nearly spheroidal structure due to high shear and adequately slow cooling. However, some ripened rosette still occurred as the microstructure size obtained at the bottom zone were scattered randomly showing that the nucleation was continuously occurs during the formation of a- al. Figure © shows that the microstructure has equal dimension and dispersed equally in the alloy referred as equiaxed grains.

In conclusion, CS process can be used to develop spheroidal or globular structure in A319 alloy with two phases; α – Al phase and β – Al phase represented by $Al_{15}\ (Fe, Mn)_3Si_2$ and Al_5FeS respectively. These two iron metallic compounds showed that homogeneous and heterogeneous nucleations can be obtain by shearing force along the cooling slope.

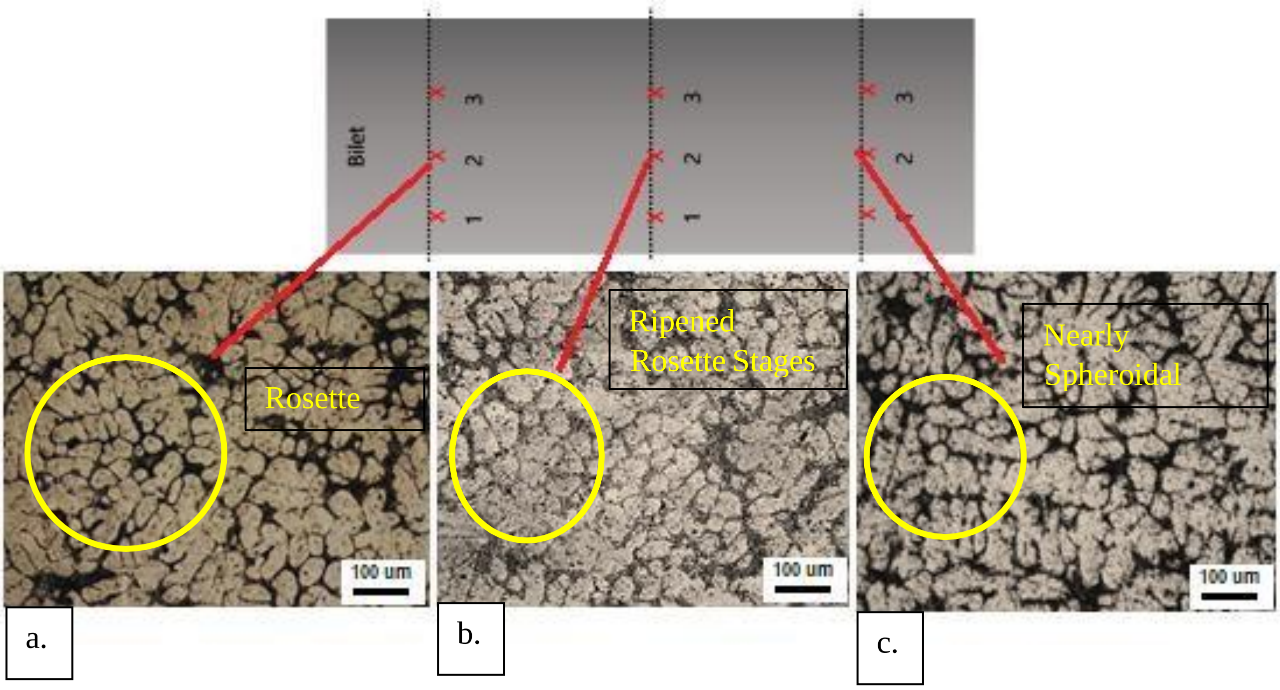


Figure 4.2: Microstructure Of Feedstock Material After Cooling Slope Process

4.1.3 Optical Microstructure of CNTs – A319 Composite After Thixoforming Process

Figure below shows the present of two nondendritic phases that evolved from dendritic structure that is mainly α- Al phase which is the yellowish region and Si – eutectic phase which is the darker area. The size of α – al grains are increases due to perfect heating rate in thixoforming process producing homogeneous globules size microstructure that provide flowing ability in billet during forming and equiaxed morphology. Based on the figure, we could see that the spheroidal α – Al helps to ger rid of micro- porosity by diffuse and coalesce with one another in form of slug that flow in laminar while keeping the β – Si unchanged.

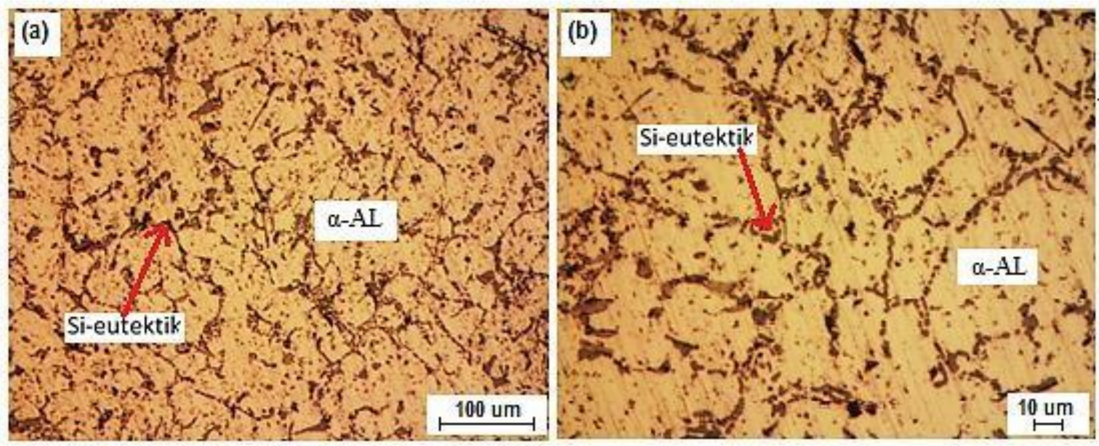


Figure 4.3: Microstructure after thixoforming of A319- CNTs Composite Alloy

4.1.4 Optical Microstructure of CNTs – A319 Composite After T6 Heat Treatment

After being treated with Short T6 heat treatment, the obvious change is the size of Si- eutectic and the Si – particles spheroidised like a perfect rounded shapes. A dark grey color indicated the present of intermetallic phase of Mg_2Si. After about 5 mins of solution heat treatment the Mg_2Si become smaller, the dissolution and diffusion of the size make Si crystal to grow and become more rounded. Due to fine dispersion of coherent precipitates, dissolution of Mg_2Si in aluminium α matrix is the main reason for age hardening. Another phase that found after T6 heat treatment for this sample is Al_2Cu. Al_2Cu occurred during alloy solidification and is more compact with blocky shape as compared to Mg_2Si. Formation of this Al_2Cu as the intermetallic compound can be relate to fracture in alloy that presented as brittle behaviour. Figure 4.4 shows that 1 hour solution in short T6 heat treatment is enough to dissolve the CNTs – A319 alloy composite and get homogenize sample as all silicon eutectic have evolved into more circular shape.

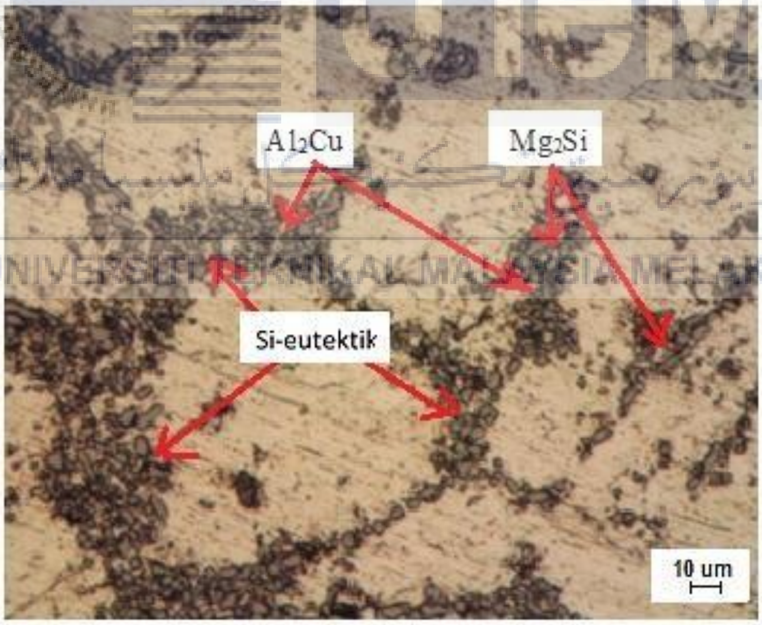


Figure 4.4: Microstructure of CNTs – A319 Composite After T6 Heat Treatment

4.1.5 SEM – EDS Analysis of MWCNTs – A319 Composite Alloy

Figure 4.5(a.) shows the elements presents in microstructure of A319 alloy modified with CNTs for SHT such as Al, Si, Cu and Mg with their chemical composition. In this figure we could see the present of Si rich eutectic and Al_2Cu phase with rounded structures. The microstructure also showed the Mg – bearing and Cu intermetallics phases with finer or coarser spheroidal morphology. Result shows that MWCNTs have great effect as a reinforcement agent on the grain refining of Al-Si alloy which is A319 alloy for this study. The evolution of the microstructure is affected by the stirring process during injection of the CNTs in the plunger as well as the heterogeneous nucleation in CNTs. The collision and squeezing help the distribution of CNTs in A319 molten alloy breaking the dendritic arms and prevent agglomeration to improve the microstructure which is more globular. As some tertiary element such as Mg that is added to increase the wettability that will not being able to be observed in some of the blocky or the spherical particles. Figure 4.5 (b.) indicate that carbon elements in CNTs can be found in MWCNTs – A319 composite alloy that help to increase the mechanical properties of the aluminium by reconstruct and improve the microstructure of the aluminium. While, in Figure 4.5 (c.) the spheroidization and the reduction in aspect ratio of Si eutectic during short heat treatment shows loss of interconnectivity in eutectic phase. The microstructure shows that the eutectic Si particles are nodular with size of 3~5 µm. last but not least, Figure (d.) shows the microstructure of the Al element that consist of interdendritic, α – Al dendritic and intermetallic region. The complex morphology'Chinese Script' intermetallic, α- $Al_8(Fe,\ Mn)_2Si\ precipetates$ at the eutectic region. While the Si particles embedded uniformly around α – Al globules may increase the mechanical properties of the aluminium alloys.

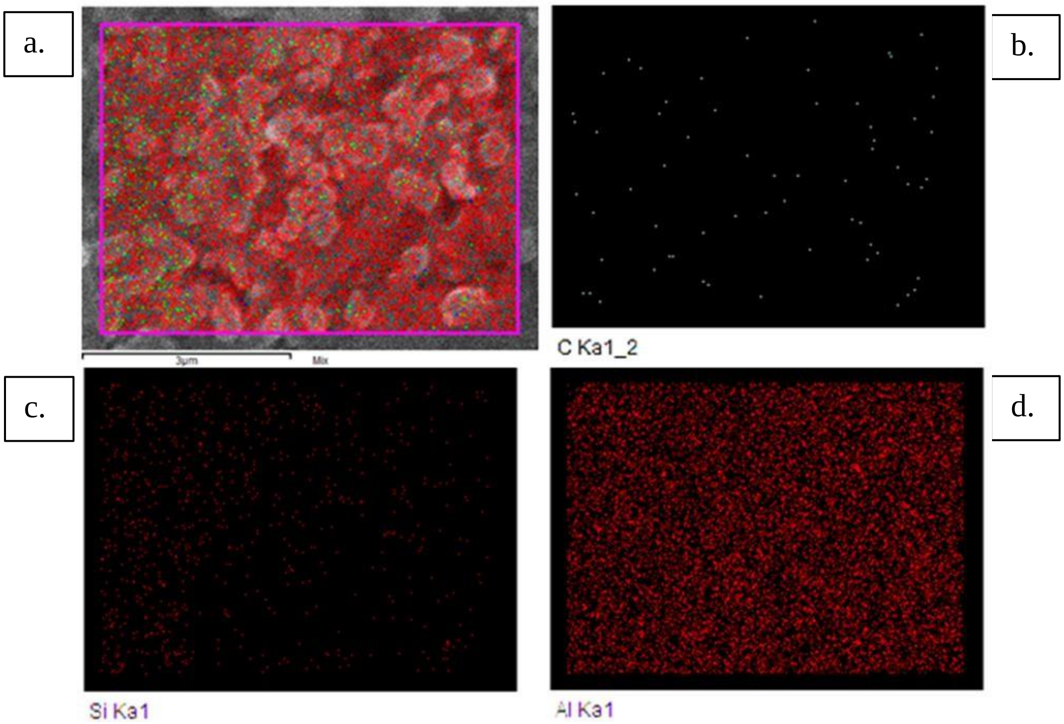


Figure 4.5: SEM – EDS Analysis of MWCNTs – A319 Composite Alloy

4.1.6 Elements in Thixoformed Short T6 Heat Treatment A319 Aluminium Alloy Reinforced CNTs by FESEM EDX.

Figure 4.6 shows that some elements such as Si, Mg and C with chemical composition corresponding to intermetallic phases after SHT. In eutectic region, precipitation of that dispersed equally at grain boundaries Mg_2 Si, $Al_8Mg_3FeSi_5$ and $Al_5Cu_2Mg_8Si_6$ in Thixoformed T6 composite can be further study. The present of C in the graph showed that MWCNTs reinforcement agent is well dispersed in A319 alloy modified with CNTs. The dissolution of Cu rich phase confirm that phases with CNTs present a high mechanical properties as well as the thermal properties besides the lightweight properties and the carbon remain unchanged after SHT showing the present of additional C element as the tertiary that fill the pore between A319 elements and give new microstructure that may longer the time to fracture as the particle arrange close to each other and the C- bond that hold them together. There are also elements such as Mg and Si that soon produce Mg_2Si in the composite consistently and some will left in the solid solution at quenching stage during short T6 HT. This is the reason why α- Al phase have excellent saturated Mg and Si phases while the remaining Mg react with Cu and Fe to produce intermetallic compounds .

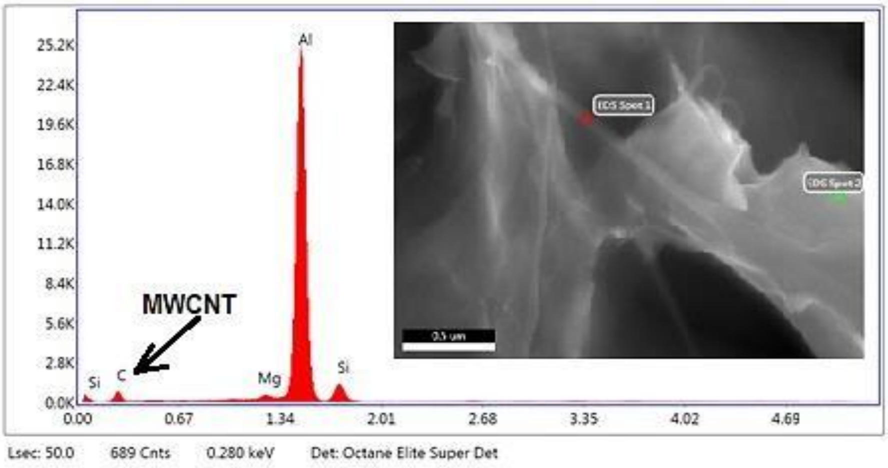


Figure 4.6: FESEM EDX Analysis shows elements in A319 – MWCNT Composite Alloy (The present of MWCNTs)

4.2 Mechanical Properties Analysis

4.2.1 Hardness Test

To calculate the hardness test result, a sample from each process will be tested using Rockwell Hardness Test (HRB) machine. The data will be repeated to 3 times measurement before the average was converted to Vickers Hardness in HV unit. Table below shows hardness values for as – cast, thixoforming and short T6 heat treatment. The sample with 0.5 wt.% of CNTs is select for each process and the result then presented and tabulated in a graph manner.

SSM Processing Type	Mechanical Properties – Hardness (HV)
As- Cast	80HV
Thixoforming	96HV
Short T6 Heat Treatment	108HV

Table 4.1: Hardness Test Result

Different SSM processing type will give different hardness values, the highest hardness value of A319-CNTs composite alloy is 108HV given by Short T6 HT with optimum parameters. Short T6 HT attained the highest value of HV when a sample from 0.5 wt.% CNTs with medium stirring rate for 15 minutes being tested showing that the CNTs content of reinforcement agent as well as the stirring time play important role to produce homogeneous sample with non-dendritic microstructure. While the lowest hardness value is from as- cast which is only 80 HV compared to 96 HV and 108HV for thixoforming and Short T6 HT respectively. From the graph below (Figure 4.7), we could see the increasement of 20% for thixoforming sample compared with as – cast sample and about 35% increasement in HV for Short T6 HT compared to the as – cast, while the hardness comparison between Short T6 HT and Thixoforming samples shows only 12.5% increasement. This can be explained by the microstructure of each sample, Short T6 HT consist of coarser microstructure as the present of Al_2Cu particles at the end of T6 HT can dissolve eventually and diffuse perfectly into the matrix region. In thixoforming sample, we could see the microstructure shows evolution from dendritic to spheroidal structure, the dendritic of the as- cast sample will evolve into globular shape when thixoforming take places. The evolution shows the decreasing porosity that increase hardness. The present of Mg will acts as wettability agent of CNTs in α- Al region.

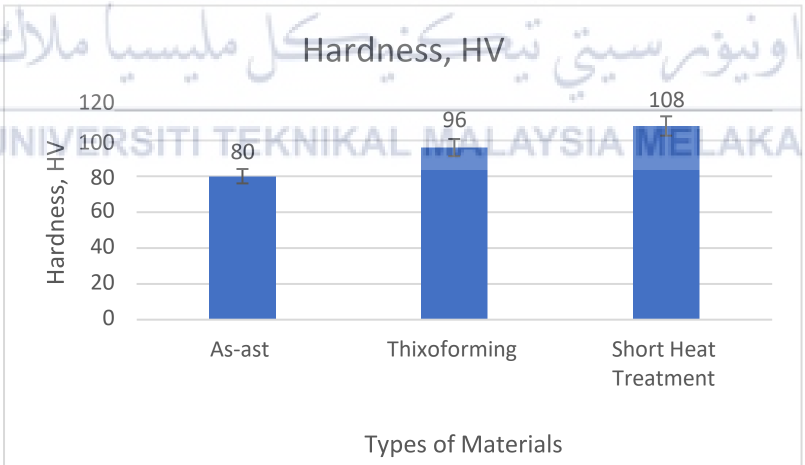


Figure 4.7: Graph of Hardness Test Result for A319 – MWCNTs Composite Alloy

4.2.2 Tensile Test

Tensile test will cover some test to determine the mechanical behaviour such as ultimate tensile strength (UTS), yield strength (YS) and the elongation of fracture in percentage.

SSM Processing Type	UTS (MPa)	YS (MPa)	Elongation to fracture (%)
As- Cast	190 MPa	186 MPa	3.2 %
Thixoforming	230MPa	213 MPa	5%
Short T6 Heat Treatment	280MPa	251 MPa	7.2%

Table 4.1 : Tensile Test Results

Positive improvement in tensile test can be shown by graph a (Figure 4.8) from as – cast to thixoforming and short T6 HT for A319 – CNTs composite alloy. Same as above, each sample from each SSM processing will be tested. Sample from 0.5 wt.% CNTs with 15 minutes stirring time at medium stir rate will be use for all three SSM processing, As – cast, Thixoforming and also Short T6 Heat Treatment. Short T6 HT shows outstanding tensile test result as compared to thixoforming and as- cast. The composite alloy shows the highest UTS which is 280MPa due to sizeable change and the addition of CNTs. The optimization of favorable condition in Short T6 HT which is much shorter processing time increase the ultimate tensile strength with 21.74 % MPa and 47.3 6 % MPa increment as compared to thixoforming (230 MPa) and as – cast (190 MPa) with respectively.

Graph b (Figure 4 .8) shows lowest yield strength for as – cast with 186 MPa but improve significantly to thixoformed composite alloy to Short T6 heat treatment, 213 MPa to 251 MPa respectively. The steady increasing MPa in as – cast to thixoformed, 14.5%, and thixoformed to short T6 heat treatment with 17.84% proved that the evolution of microstructure as well as thermal stress between the al- matrix and CNTs effect the formation of globular microstructure of silicon particles in short T6 HT that only need approximately of 5 minutes, as the sample must undergo thixoforming process prior to T6 HT. The sample use shorter overall solution treatment and reach maximum hardness for the formation of θ - phase as no more intermetallic phase was detected after 30 minutes.

Adding more CNTs above the optimum percent will make the composite alloy to become more brittle which due to agglomeration of nanoparticles and nanocomposites. The present of CNTs improve the ductility if not exceed the optimum value. The elongation to fracture decreases gradually from Short T6 heat treatment, 7.2 % to thixoforming, 5 % to As -cast, 3.2%. According to Salleh et al. in 2017, fracture in as- cast sample can be classified as brittle fracture and as – thixoformed sample showed ductile fracture. The different between the thixoformed samples are they is fine and well distributed dimple fracture as compared to as – cast sample with fracture on long Si particles.

The mechanical testing result for elongation to fracture, yield strength and ultimate tensile strength showed that outstanding mechanical properties are fulfill with the short T6 heat treatment in SSM processing. The improvement also can be said as a result from addition of CNTs into the alloy as the reinforcement agent helps to restrict the movement in matrix while reconstruct the microstructure of the composite alloy to become more spheroidal in short time in processing steps.

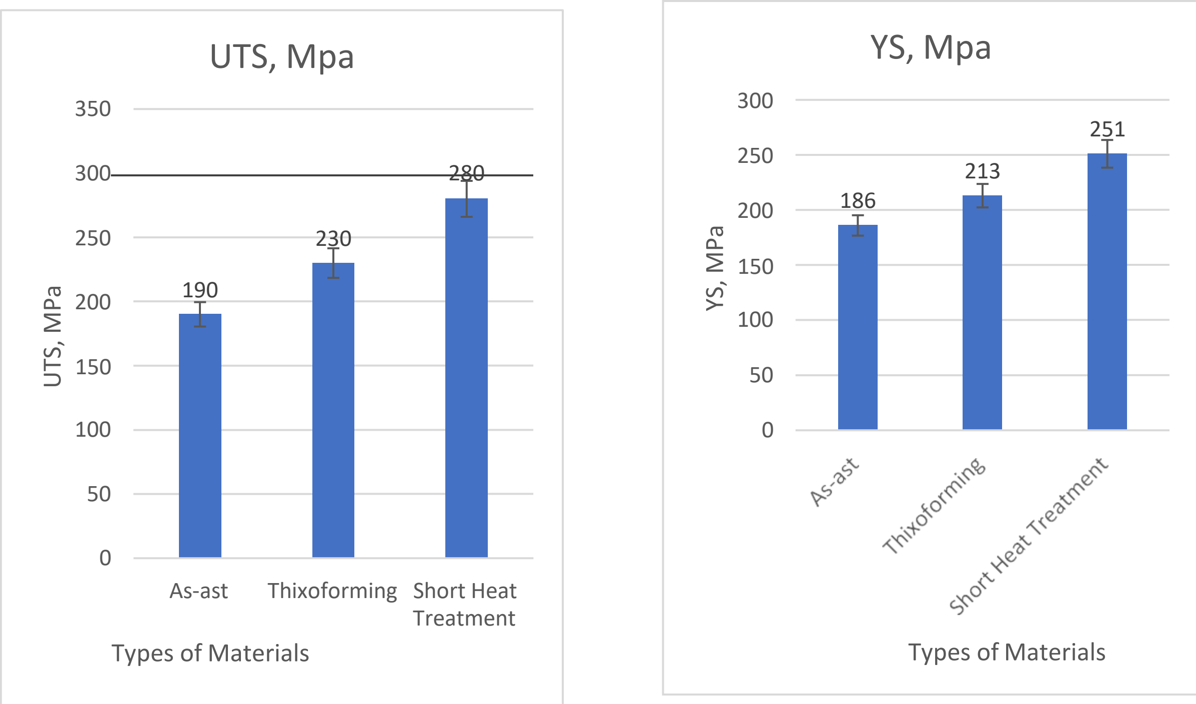


Figure 4.8: Graph of Tensile Test Result for A319 – MWCNTs Composite Alloy; a)UTS and b) YS

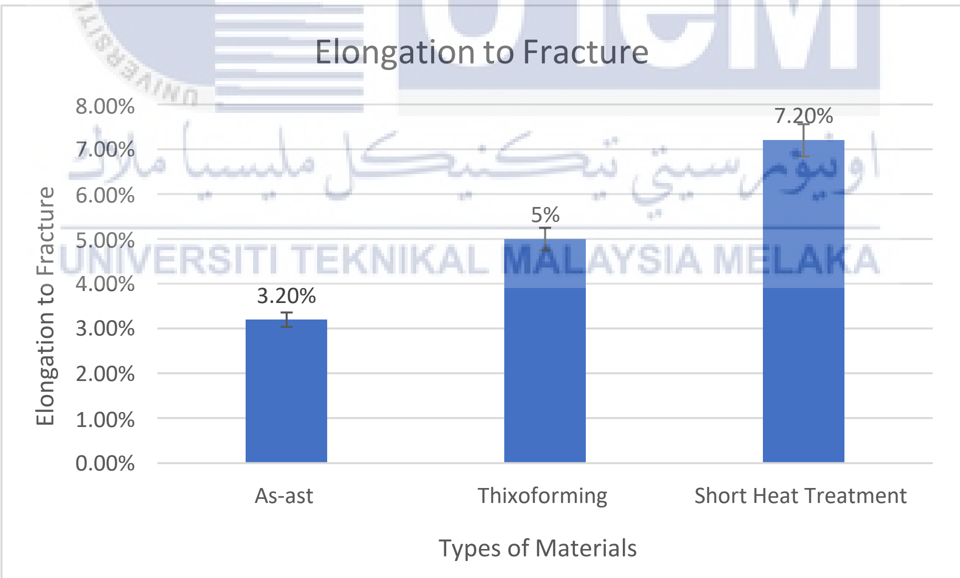


Figure 4.9: Graph of Elongation To Fracture
Tensile Test Result for A319 – MWCNTs Composite Alloy

Chapter 5

CONCLUSION

In this chapter, a conclusion can be drawn based on previous results and discussion in Chapter 4, a recommendation also can be suggested to provide better and new work solution to improve and to maximize solutions findings. This chapter will also provide the summary of this study either all the objectives were able to achieve or not. Any sustainability problems and issues also being presented in this chapter to solve any related issues.

5.1 Conclusion

Aluminium alloy is the most commonly used material in automotive and airbus industry due to their properties that is lightweight but as strong if not stronger than some steel. The extreme durability and the strength make aluminium suitable for everyday applications. While Al 319 consists of main elements such as Al, Si and Cu contain metastable strengthening precipitates such as θ – phase, make this alloy suitable to be fabricate into complex components with high mechanical properties, high castability but low production costs. But, A319 alloy consist of α- Al dendritic region that lower the mechanical properties of this alloy. Luckily, microstructure characterization of Al 319 alloy can be grouped by eutectic Si particles in interdendritic region with intermetallic phases and α- Al dendrites. To increase the amount of solute that effect the growth of dendritic structure to produce a finer microstructure, the higher Si content in Al – Si – Cu alloy can be select.

By using different content of MWCNTs as the reinforcement agent and stirred at medium rate, which are 0 wt.%, 0.25 wt.% and 0.5 wt. %, with 5 minutes, 10 minutes and 15 minutes the CNTs were reinforced into A319 aluminium alloy producing A319 – CNTs composite alloy. Besides the physical properties of CNTs such as high thermal and electrical conductivity, lightweight, small and fine structure as well as the plenty of resources to produce CNTs with only small amount of material make this reinforcing element as popular choice to be use in order to improve mechanical properties by re-construct the microstructure with no agglomeration.

Short T6 Heat Treatment is the new approach in heat treatment of SSM processes to produce aluminium casting alloy. Same as the conventional heat treatment, there are solution heat treatment, quenching process and artificial ageing. The different of short T6 heat treatment with the common heat treatment is the time taken for common heat treatment is minimum 9 hours to complete the whole process while for short T6 heat treatment, we only need 30 minutes in solution treatment, 2 H in artificial aging while the temperature must be fix to optimum C for each treatment. After, the A319 – CNTs composite alloy produce, the ingot will be cut into small pieces and the sample must undergo microstructure test as well as mechanical test to study the microstructure, hardness, ultimate tensile strength and Yield strength of A319 alloy reinforced with CNTs after being treated with short T6 heat treatment. Only single sample from total 9 samples will be tested to compare them with sample from as – cast and thixofoming processes.

Heat treatment prove they can modify microstructure and increase hardness as well as tensile strength of an aluminium alloy, while CNTs will provide homogeneous solution with the present of Mg as wetting agent to increase wettability and prevent agglomeration in the solution. From results and discussion of this study, there are several highlighted analysis findings can be drawn to summarise this study:

1. CNTs can be divided into SWCNTs and MWCNTs which can be use as a reinforcement agent to improve and increase the mechanical properties by dispersed homogeneously as their sizes are small in the metallic matrix, A319 alloy and fill in the boundaries to aligned with direction in the composite alloys because the CNTs restrict the dislocations movement in the matrix. This composite microstructure proposed and improved the mechanical properties such as hardness and tensile strength without effecting other properties such as thermal and electrical conductivity.
2. Microstructure of A319 – MWCNTs shows deterioration of the composite's properties due to the agglomeration. Hence, stirring casting process use in this experiment can help to formed homogeneous Al matrix with finer grain boundries.
3. In T6 heat treatment catastrophic failure can be prevent while increasing the mechanical strength but the processing time is longer with minimum 9 H. The production cost is also high with any unnecessaries processing time and steps. While, in short T6 HT 30 minutes to 1H in solution treatment with 540C, then being cooled down in 180C before being artificially aging for 2 hours. Globular microstructure of

silicon particles starts to evolve after 5 minutes, we could also see the formation of Al_2Cu particles that diffuse into the surrounding matrix. A319 – CNTs composite alloy tend to transform β - Al_5FeSi to α - $Al_8(Fe, Mn)_2\ Si$ after dissolution of Mg_2Si take places.

4. Hardness test was conducted using Rockwell Hardness Test and the value is presented in HV. While the lowest hardness value for A319 – CNTs composite alloy is from as- cast which is only 80 HV compared to 96 HV and 108HV for thixoforming and Short T6 HT respectively. Increasement of 20 % and 35 % in hardness for thixoforming sample and short T6 heat treatment sample as compared to as – cast showing that heat treatment will produce homogeneous matrix solution reinforced by CNTs.

5. In tensile test, UTS, YS and elongation to fracture in short T6 heat treatment showed highest value compared to as – cast and thixoforming with 280 MPa, 251 MPa and 7.2 % respectively. As – cast also showed the lowest UTS, YS and elongation to fracture due to the micropores and the distribution of CNTs reinforced agent in the Al – matrix in producing A319 – CNTs composite alloys.

5.2 Recommendation

From this study there are some suggestion and proposal can be made to improve any arise issues related to this study. The recommendation provided below can be used to achieve better and favorable results regarding production of A319- CNTs using Short T6 Heat Treatment.

1. Pre – oxide or do surface treatment of the reinforced phase.

For example, Mg elements can be use as wetting agent to increase wettability in the aluminium matrix reinforced with CNTs. The previous study reveals that augmentation of CNTs using powder metallurgy improved the strength of A319 -CNTs composite alloys.

2. Reduce the size of the casting die in thixoforming process.

The thixoformed ingot produce by thixoformed casting die is too big for tensile test. Hence, removal process like cutting using Electron Die Machine, EDM Wire Cut and CNC machine before being tested. These activities consume unnecessary time and the use of power energy can increase the cost in production of the tensile test samples. By minimizing the size of the die casting we can save more money and save time as well as save the material by reducing the scraps.

3. Use spark plasma sintering followed by hot extrusion and use ball milling in powder preparation.

A319 – CNTs composite alloy using ball milling in powder preparation and fabricated method involved spark plasma sintering followed by hot extrusion can produce homogeneous distribution of CNTs in the composite product with dimple fracture that help in dispersion strengthening and form bridge between al- matrix and oxides.

5.3 Sustainability of this study

Brundtland Commission famously defines sustainability as the ability to develop that meets the need of present without compromising the ability of future generations to meet their own needs. There are three pillar of sustainable interconnection which is Environment, Economic and Society. Production of metal matrix composites (MMCs) become an important part of mostly fabricated components in term of material selection to increase the mechanical properties without compromising their own properties. Hence, today many people around the world especially researcher and engineer claimed that aluminium alloy as a sustainable material because some alluminium alloy can be recycled.

Production of A319 -CNTs composite alloy using short T6 heat treatment can be considered as sustainable process as the production cost is economic and the time taken to produce the composite alloy also short. We do think that harmful gases can also be reduce when the process is short. The combustion of power to generate energy also may decrease as well as the production cost. From the business perspective, cost is always being an issue. Every penny that flows out must based on something that may bring back the values. Therefore, currently lightweight components and weight reduction in aluminium composite alloy being the most important aspect that may considered aluminium based alloy as sustainable materials. The greenhouse effect can be an issue as we need transportation to supply the raw materials and also to deliver the end product to users. CO_2 and particles emission can contribute to haze.

Besides that, concept of 6R' should be implemented in engineering and manufacturing works. Inthis study, Reduce and Recycle are the 6R' concept that are implemented to promoting sustainability process and product. The scrap from thixoforming die casting can be recycled and reuse when the leftover being treated and use it back as second sources of materials. By practicing this 6R' method we could save more money and save our environmental by reducing unnecessary processing times in T6 Heat Treatment with harmful yet dangerous gaseous to our nature that may affect living things. But the scrap will be considered as waste if we simply discharge into a scrap bin. Environmental pollution also could be an issue due to material waste and long times process.

5.4 Complexity of this Study

Since there are still limited study about aluminium A319 alloy being used as metal matrix reinforced with CNTs there are some complexities arise during the completion of this study. The formation of magrosegregation and porosity in Al -alloy microstructure because of the coarse primary of α- Al and non – circular shape in eutectic Si phases. Agglomeration of CNTs in A319 alloy occurred as the large different in aspect ratio causing deterioration in hardness, HV. The agglomeration always happened when we inject the CNTs that wrapped with aluminium foil using a plunger as the size of the plunger can become small due to the elements that solidified and stuck in the plunger.

5.5 Life – long learning

Short T6 heat treatment is a new approach technique for producing A319- CNTs composite alloy to increase the mechanical test and improve the microstructure. The obtained results demonstrate that it is possible to reduce time in solution treatment producing semisolid casting process with outstanding properties. This technique can be applied in various field especially in automotive and aerospace as well as engineering and manufacturing factory since aluminium – based multicomponent alloys especially A319 is lightweight but also strong due to changes in Si – morphology in short solution treatment, short quenching and also short artificial aging. Besides, short T6 HT can also consider as green technology as we can practice sustainability from the perspective of economic, environment and social. By using this method in producing A319 -MWCNTs composite alloys we can save more money and time as well as the material used as aluminium alloy is much more lightweight compared to other metals.

This study should not stop until here but in the future research work could be conducted in developing lighter composite alloy using more cost-effective reinforcement agent and alloying elements such as Mn that is most cost effective with leas amounts of embodied energy and eco-friendly compared to other elements. The study about coefficient of friction, C0F can also be further study to indicate the inclusion of CNTs into structures, increasing wearability surfaces for future applications.

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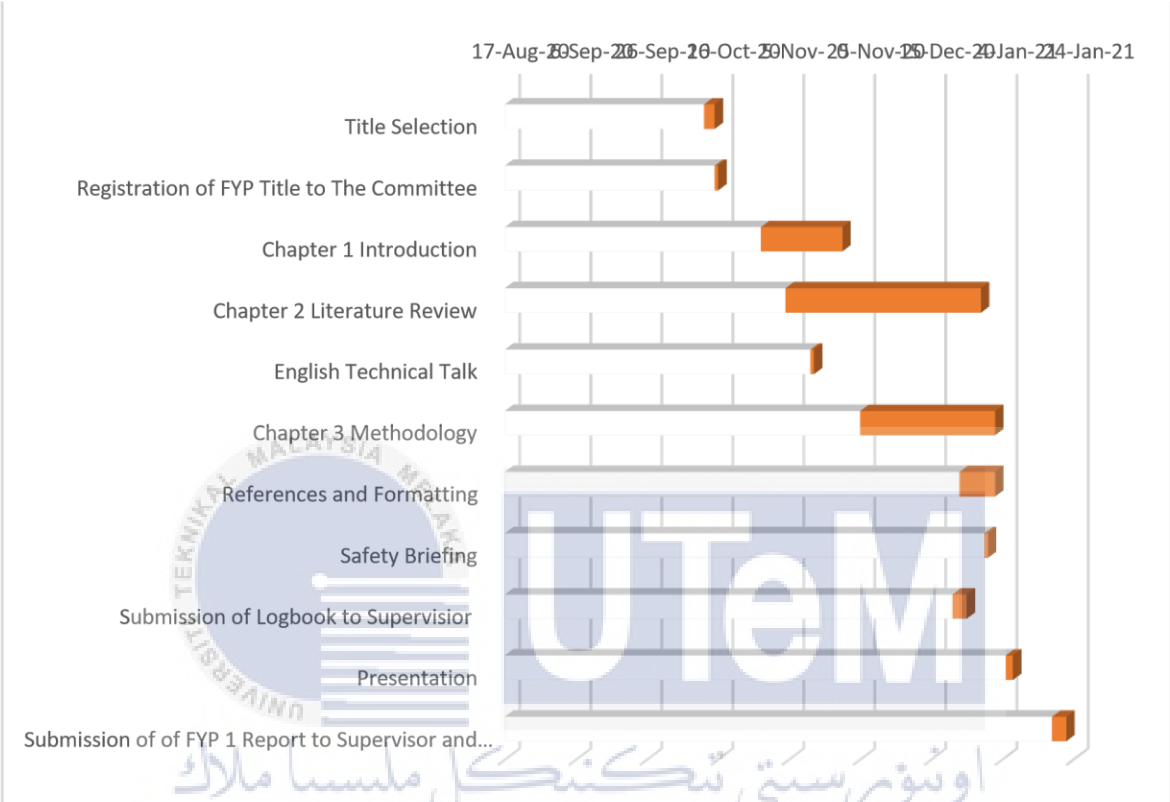
APPENDICES

A Gantt Chart for FINAL YEAR PROJECT 1



A Gantt Chart for FINAL YEAR PROJECT

TASK \ WEEK	01	02	03	04	05	06	07	08	09	10	11	12	13	14	15
1. Cutting the A319 ingot into smaller pieces using bandsaw															
2. Preparing of feedstock for permanent mould casting															
3. Thixoforming process															
4. Short T6 Heat Treatment															
5. CNC and EDM Wire Cut															
6. Testing the sample – Hardness Test and Tensile Test															
7. Microstructure Analysis															
8. Write Chapter 4 and 5															
9. Slide Preparation															
10. Submit Video Presentation															
11. Question and Answer (QANDA S.)															
12. Correction and Improvement															
13. Submission Report															
14. Hardbound															