INVESTIGATION OF MECHANICAL & WEAR CHARACTERISTICS OF T6 HEAT TREATED THIXOFORMED ALUMINIUM ALLOY COMPOSITE

A.A. Rahman[1], M.S. Salleh[1], I.S. Othman[1], S.H. Yahaya[1], S.S. Al-Zubaidi[2] and K. Zulkifli[3]

[1]Faculty of Manufacturing Engineering,
Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.

[2]Al-Khwarizmi College of Engineering,
University of Baghdad, 10071
Baghdad, Iraq.

[3]Crusk Assembler Sdn. Bhd.,
Jalan Tunas Baru Seksyen 1/5,
Kawasan Perindustrian MIDF,
78200 Masjid Tanah, Melaka, Malaysia.

Corresponding Author's Email: [1]shukor@utem.edu.my

ABSTRACT: Thixoforming of aluminium alloy has been making a strong presence in the near net shape part production for the automotive and aerospace industries due to its process superior properties output in comparison to the other conventional methods. Improvement of the properties would surely further add to the already advantageous process above others. This study investigates the mechanical and wear behaviours of thixoformed Al-Si-Cu alloy when multi-walled carbon nanotubes (MWCNT) infusing into the thixoforming process. A new approach established using the mechanical stirring casting techniques produces a well-bind composite mixture of non-dendritic aluminium mixed with different content (0.5, and 0.75) of MWCNTs. The approach enclosed the MWCNTs with 0.5 wt.% of Magnesium (Mg) powder as a wetting agent into an aluminium wrapping and stirred it with LM4 for 10 and 15 min interval inside a furnace. The feedstock produced would be thixoformed before finally being applying T5 and T6 heat treatment. The microstructure of each specimens is characterised while the mechanical (tensile and hardness) and wear properties are measured according to each requirement. The outcome shows formation of

non-dendritic microstructure with homogeneous distribution phase of the MWCNTs within it. The DOE analysis of the highest MWCNT content and stirring time after a T6 heat treatment reveals the highest hardness and UTS result of 94.6 HV and 246.2 MPa respectively. The Taguchi analysis also reveals that heat treatment was the most significant factor contributing towards the improvement. The heat-treated samples increase the hardness and tensile strength approximately 30% compared to an as-cast and give around 20% increment of wear resistance to the specimen.

KEYWORDS: *Semisolid Metal Processing; Aluminium Matrix Composite; Multi-Walled Carbon Nanotubes; Mechanical Test; Wear Test*

1.0   INTRODUCTION

The composition of cast aluminium LM4 alloy of 92.14% Al, 4.155% Si, 0.554% Fe, 2.553% Cu, 0.352% Mn, 0.083% Mg, 0.015% Zn, 0.003% Cr, 0.0031% Ni, 0.123% Ti, 0.0213% Pb is susceptible to fluidity and are commonly used for casting process with thixotropic properties. The intrinsic properties of Al-Si alloy that include high strength to weight magnitude relation, exceptional solidity and pressure tightness, low thermal expansion coefficient, good mechanical properties and resistance to corrosion makes it widely and commercially utilised [1]. Under these characteristics, the semi-solid processing such as thixoforming of aluminium alloys is paving its way into the aerospace and automotive industries due to the properties improvement it can give and the problematic issues from a conventional casting process that it can overcome. The laminar flow from a liquid to solid fractions of 0.3-0.5 alloy would ensure a reduction in gas entrapment during solidification [2].

On the other hand, producing a Metal Matrix Composite (MMC) would improve further the strength and physical properties of the metal while maintaining the metal's lightweight character. The interest for CNT-metal composites as a potential mechanical reinforcement has grown significantly. This trait can achieve when the matrix and reinforcement gain a uniform dispersion of CNTs in the matrix and an excellent bonding at the interface [3]. However, the segregation of CNTs due to their strong van der Waals forces could inevitably produce material defects that would affect the properties due to any inhomogeneity dispersion.

CNTs have high potential of usability in the industry of nanotechnology, optics, electronics and other fields. CNTs have a large

aspect ratio thus making it possible to alter the microstructural of its matrix [4]. The carbon nanotubes are available as cylinder single-walled carbon nanotubes (SWCNT) or a multiple concentric cylinder known as multi-walled carbon nanotubes (MWCNT). Although the cylindrical wall is varied, both MWCNT and SWCNT have the same purpose, which are to strengthen the mechanical properties and enhance thermal responses of metal matrix composite materials. The linked hexagonal shapes give the strong molecule structure yet can be bent and back to its original shape when released. Although SWCNT has a tensile strength 10 times, better that MWCNT but MWCNT are cheaper and has better dispersibility in solutions compared to SWCNT.

A grain refinement can contribute to improve strength so long that the reduction does not create microporosity. It is also known that modification of the morphology from columnar to the equiaxed grain would increase the mechanical properties quality and uniformity of a cast alloy [6]. Heat treatment has shown its ability to modify the microstructures without changing the form of the entity [5-6]. The most used heat treatment for aluminium alloys is T5 and T6 heat treatment. T5 heat treatment includes cooling after casting or hot working prior to artificially aging, while T6 heat treatment requires three-step treatments of solution treatment (ST), quenching and artificial aging [7-9]. T6 heat treatment is considered the most popular choice for increasing the hardness, strength, temperature resistance and ductility of a sample [10-12]. ST is intentionally applied to dissolve the Cu-containing particles during solidification process is slowly dissolve by solution treatment and removed by a high temperature close to the eutectic temperature. Many attempts were recorded in short ST time that would suggests the recommended 8-10 hours standard is unnecessarily long [13-14]. This is further proven by some research that saw the same materials integrity while reducing the ST time [7]. Even though $Al_8Mg_3FeSi_6$ and $Al_5Cu_2Mg_8Si_6$ are hard to dissolve, it is sufficient to expose the alloy at high temperature in a short time to avoid local melting of Cu-containing phase during the solution treatment [15-17].

Conventionally, the components that are widely manufactured using die casting method carries defects such as porosity, macrosgregation and forming forces leading to inferior material characteristics. Since it has been widely recorded that thixoforming and heat treatment can enhance the mechanical properties, the incorporation of CNTs with aluminium metal matrix have not seen proper investigation especially

towards the tribology and coefficient of friction of Al MMC. Hence, this study assesses the effectiveness of mechanical stirring casting technique for feedstock production. Thixoforming process is to investigate the improvement of the mechanical properties and wear behaviour of thixoformed samples after heat treatment.

2.0   METHODOLOGY

2.1   Feedstock Preparation

The initial step for performing a thixoforming process is by preparing the LM4 + MWCNT billet through a mechanical stirring casting process under the defined material and process parameter setting shown in Table 1. These samples would later be subjected to heat treatment after the thixoforming process.

Table 1: Design of Experiment using Taguchi Method

Sample	CNT content (wt. %)	Stir Time (minutes)	Heat Treatment
1	0.5	10	T5
2	0.5	10	T6
3	0.5	15	T5
4	0.5	15	T6
5	0.75	10	T5
6	0.75	10	T6
7	0.75	15	T5
8	0.75	15	T6

2.2   Thixoforming Processing

The process is performed on a T30-80KHz thixoforming machine. The feedstock billet is placed on a pneumatic cylinder ram in between an induction coil as shown by Figure 1. Then, it is heated to a semi-solid state (580°C) indicating a 50% liquid fraction from the Differential scanning calorimetry (DSC) curve [18].

The reheating was controlled by the current frequency that increased at 50 A per minutes until the intended thixoformed temperature, T_{thixo} was reached. A K-type thermocouple measured the billet temperature throughout the process. Subsequently as the T_{thixo} was reached, the ram pushes the semi solid feedstock with 5 tonnes forging load into the steel mould to perform the thixoforming before finally being cooled down to ambient temperature.
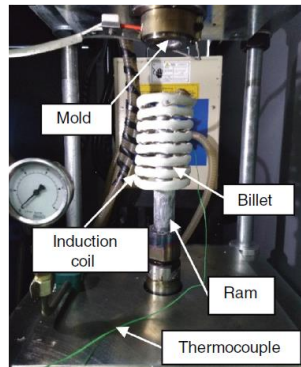
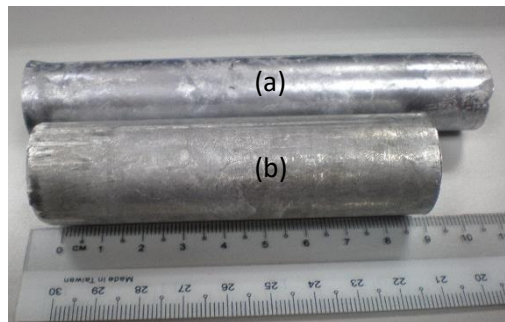Figure 1: Thixoforming process machine setup [19]

Figure 2: The aluminium MMC billet produced by: (a) mechanical stirring casting and (b) thixoforming process

2.3   T5 and T6 Heat Treatments

Table 1 shows the T5 and T6 heat treatment time for heating the sample. A T5 heat treatment will consist of only an artificial aging where the alloy will be heated up to 160°C for 6 hours to cause precipitation of fine particles of the beta phase. Meanwhile the T6 heat treatment process consists of three steps: (i) solution treatment where the alloy will be heated up to 540°C for 8 hours using the Nabertherm furnace to dissolve the beta phase, (ii) quenching at room temperature to build a supersaturated solid solution and, (iii) artificial ageing at 160°C for 4 hours. Finding by many researchers proved that the silicon particles would start growing and change to a more globular microstructure right from the first 5 minutes solution heat treatment [8].

Table 1: T5 and T6 heat treatments' parameters

Treatment type	Solution Treatment	Quenching	Artificial Aging
T5	-	-	160°C 360 minutes
T6	540°C 480 minutes	27°C (room temperature)	160°C 240 minutes

2.4 Hardness Test

Vickers Hardness Tester is used to test the hardness of the MMC. The hardness is measured with an applied load of 200g for 20 seconds. The T5 and T6 thixoformed samples are firstly grinded by silicon carbide (SiC) paper of 240, 400, 600, 800 and 1200 grit size. The samples polished with a polishing cloth and diamond paste of 6, 3 and 1 micron prior to perform the hardness test. The mean values are determined from the 10 measurements attained off a single sample.

2.5 Mechanical Test

The samples were cut using Wire Electrical Discharge Machining (WEDM), with the dimension of 10 mm diameter and minimum length of 60mm. Dog-bone shape tensile specimens were primarily used for the tensile tests which was fabricated from a CNC turning machine. The specimens were fabricated in accordance with ASTM: E8M standard as shown by Figure 3 and tested using a Universal Testing Machine (UTM). The test determines the mechanical behaviour such as ultimate tensile strength (UTS), yield strength and percentage of elongation for fracture.
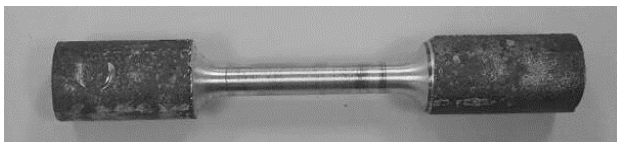
Figure 3: Dog-bone shaped specimen

2.6 Wear Test

The samples are prepared for the test by grinding them using five different grit size papers ranging 240 – 1200 before polishing with diamond paste. Three different microns are used for the polishing which are 6μm, 3μm and 1μm. The samples are then cleaned and dried before undergoing an etching process. The samples are immersed in Keller's reagent for 10 seconds.

The performance of wear rate and the coefficient of friction of the MMC are tested by a dry sliding wear test in accordance with the ASTM-G99-05 standard. A micro ball-on-disc tribology machine as shown by Figure 4 is used to measure the wear rate at ambient temperature. The samples with a height of 5 mm and diameter of 4 mm are used as the disks while a 12.7 mm steel ball as the pin. All samples undergo wear testing for 30 minutes. The wear test is performed at a 15 N applied load, 5 mm/s sliding speed with a set stroke of 2 mm. The specific wear rate is expressed on a volume loss basis (derived from the test before and after weight difference and the density) over the product of unit applied normal force and unit sliding distance. The coefficient of friction (CoF) is determined from the following equation:

$$\mu \, (CoF) = F/W \tag{1}$$

where F = Frictional force (N) and W = Applied load (N).

Figure 4: Pin-on-Disk tribo tester machine

3.0   RESULTS AND DISCUSSION

3.1   Microstructural Analysis

The morphology of dendritic features (Figure 5(a)) of the as-cast composite has changed after the thixoforming process. The microstructures changes to a non-dendritic structure that constitutes of two phases: primary α- Al phase (white area) and Si-based eutectic phase (dark area) with more spherical grain structure and no indication of acicular phases as shown by Figure 5 (b). A superior homogeneous globules size microstructure feedstock is observed clearly due to the proper heating rates during the thixoforming process which increases the α-Al grains sizes.
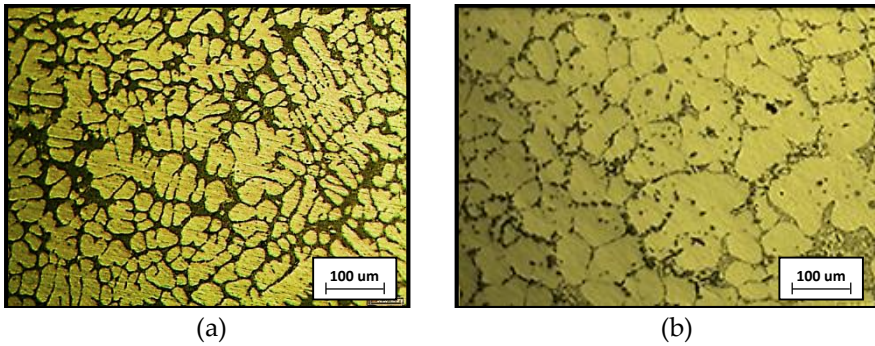
(a)   (b)

Figure 5: The microstructure under optical microscope of: (a) as-cast LM4 aluminium alloys and (b) thixoformed LM4/MWCNTs composites

Figure 6 shows the T5 heat-treated thixoformed samples. It was clearly seen that there is a significant grain growth in the structure, however, the grain size is decreased after underwent a T6 heat treatment. Hall-Petch strengthening mechanism can be used to predict the effect of grain size on the strength of a material [18]. As can be observed in the Figure 6, the microstructure of eutectic Si particles had changed and becomes more globular and more evenly distributed due to the solution heat treating and artificial aging. The uniform distribution of fine Si particles embedded around the α-Al globules would inarguably enhance the mechanical properties of the composites.
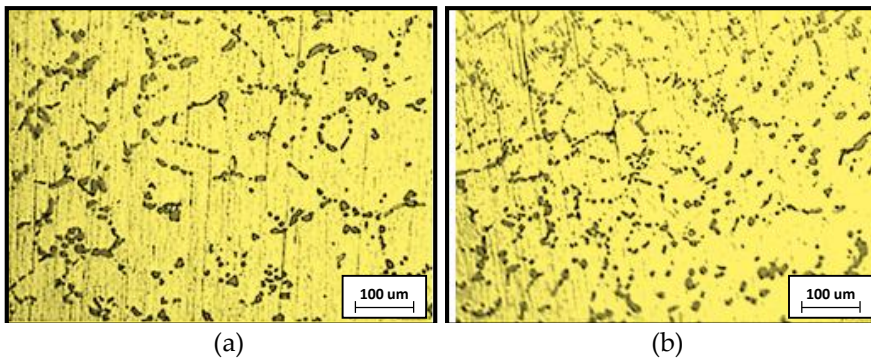

(a)   (b)

Figure 6: Optical micrograph at 100X magnification of the thixoformed LM4/MWCNTs composites after: (a) T5 and (b) T6 heat treatment

Figure 7 is the EDX analysis of LM4/MWCNTs composites after a T5 and T6 thixoformed process which shows Si Cu, Mg, and Fe elements present in certain areas within the Al matrix. The type of intermetallic phases precipitated in the thixoformed T5 and T6 composites contain Mg_2Si, $Al_8Mg_3FeSi_5$ and $Al_5Cu_2Mg_8Si_6$ that exists in the eutectic region.

These phases accumulated heavily at the grain boundaries with fine dispersion in theT6 thixoformed composite.

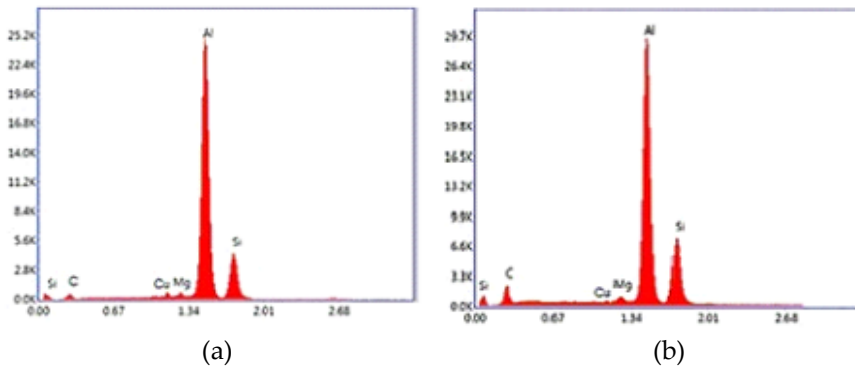(a)   (b)

Figure 7: EDX analysis of LM4/MWCNT composite undergone: (a) T5 and (b) T6 heat treatment

There are Mg that reacted with Si to produce Mg_2Si phases that dispersed consistently within the composites. A certain amount of Mg and Si is left in the solid solution during T6 heat treatment at quenching stage. Therefore, α-Al phase contains excellent saturate Mg and Si phases that could lead to mechanical properties improvement. The remaining Mg reacted with Cu and Fe to produce an $Al_5Cu_2Mg_8Si_6$ and $Al_8Mg_3FeSi_5$ intermetallic compound, respectively. $Al_8Mg_3FeSi_5$ has a compact morphology and the existence of Fe which is known to be the most destructive impurity in aluminium alloys could be a damaging factor towards the tensile strength and ductility of the composites [20].
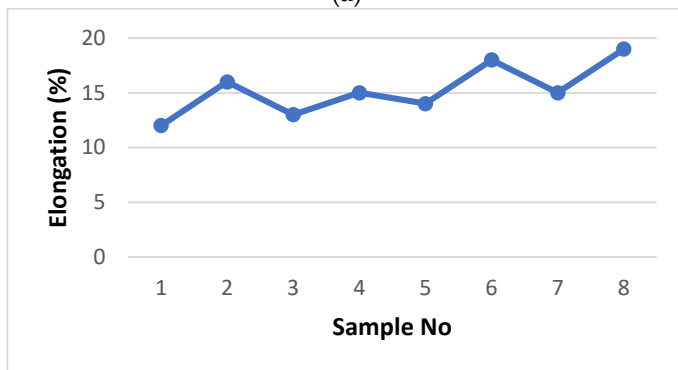
3.2   Mechanical Properties

The highest hardness value of 94.6 HV was attained by the composite with 0.75 wt.% CNT, stirring time of 15 minutes in T6 condition. The thixoformed samples hardness perform extremely better than as-cast samples due to lower porosity from the process and existence of CNT. The heat treatment has an important role in enhancing the hardness of the thixoformed sample, as the dendritic microstructures in as-cast sample are eliminated and spheroidization takes place. Comparison between a T5 and T6 of the same thixoforming parameter sample shows an increase of 38%. This notably was due to the precipitation of an intermetallic phase among the α-Al globules in the alloys. The second step of the T6 heat treatment had also contributed to improve the matrix strengthening properties. The hardness of the composite increased with an increase of MWCNT content as it acts as

reinforcement to the LM4 alloy while the longer stirring time has helped to distribute the elements in the molten matrix and prevented density segregation.

Figure 8 implies that the strength of the composite increased significantly after the heat treatments. The tensile strength has increased with the increase of CNTs content and stirring time, reaching the optimal value at 0.75 wt. % CNTs and 15 min respectively while a T6 heat treatment showed a superior tensile strength to a T5. The composite shows 30% increments as compared to an as-cast LM4 alloy. A sizable change in the sample's UTS after a T6 meant that the heat treatment had enhanced the mechanical properties of LM4/MWCNTs composites. The result also indicates that the stirring time influence the least towards the mechanical properties of thixoformed LM4/MWCNTs.
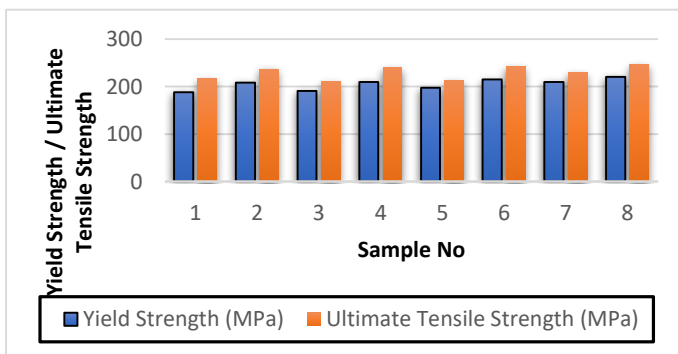
(a)

(b)

Figure 8: Mechanical properties performance of LM4/MWCNT composite of (a) yield / ultimate tensile strength and (b) elongation

3.3   Coefficient of Friction (CoF)

The CoF improves with the addition of MWCNT as compared to a pure as-cast aluminium indicating that the inclusion of CNT into the structure has increase the wearability of the surface by 7-10%. Furthermore, the CoF of the Al matrix composite is seen to improve more after undergoing thixoforming and heat treatment that alters the microstructure and dissolves and homogenize the soluble phases especially the T6 heat treatment. The CoF of T6 heat treated LM4-MWCNT has the highest improvement of approximately 20% as compared to the as-cast sample as illustrated in Figure 9.
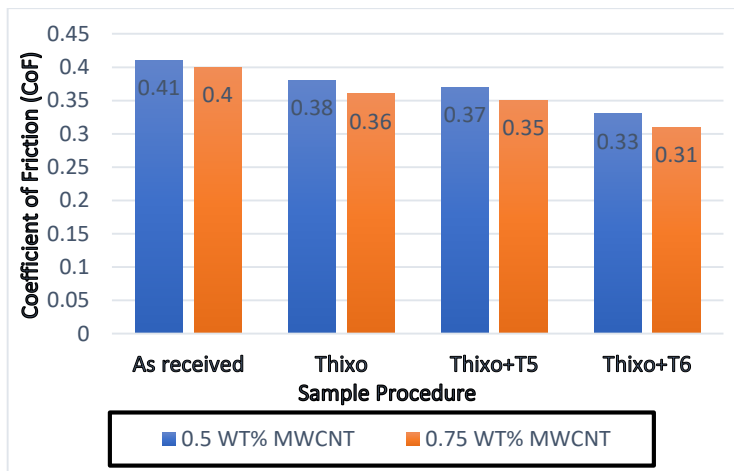

Figure 9: Graph coefficient of friction against LM4-MWCNT composite

Artificial ageing in the T5 heat treatment helps produce new and smaller precipitate. The diffusion effect that occurs due to low temperature and slow ageing time enlarge the already large Si particles. On the other hand, the T6 heat treatment involves a processing route of solution treatment that aids in homogenizing the spheroidizing of eutectic Si. The eutectic Si is more uniformly distributed, and the particle size increases. Si is a hard phase where fine and uniform distribution improves the wear properties of MWCNT-LM4 composite [21]. The quenching step further helped keep the Mg_2Si and Al_2Cu precipitate in the solid solution before finally the artificial ageing process increases the number of Mg_2Si and Al_2Cu precipitate, which contributes towards hardness increment and thus a longer wear rate of the LM4-MWCNT composite.

4.0   CONCLUSION

The best parameters of the thixoforming feedstock are determined using Taguchi analysis. The study proved that thixoforming and T6 heat treatment of the aluminium alloy composite produced a significant improvement on the hardness and mechanical properties. Furthermore, the heat treatment especially T6 improves the wear resistance of Al-MWCNT composite. The result also showed that the CNTs addition helps to increase the mechanical properties and wear behaviour of the thixoformed samples. The best CoF is achieved when 0.75 wt% of MWCNT is added to the alloy.

ACKNOWLEDGEMENT

REFERENCES

[1]   M. Poornesh, N. Harish, and K. Aithal, "Study of Mechanical Properties of Aluminium Alloy Composites," *American Journal of Materials Science*, vol. 6, no. 4A, pp. 72–76, 2016.

[2]   L. Gan, H. Lu, X. Hu, F. Lin, X. Li and Q. Zhu, "Current Progress in Rheoforming of Wrought Aluminum Alloys: A Review", *Metals*, vol. 10, no. 2, pp. 1-23, 2020.

[3]   A.M.K. Esawi, K. Morsi, A. Sayed, M. Taher and S. Lanka, "Effect of carbon nanotube (CNT) content on the mechanical properties of CNT-reinforced aluminium composites," *Composites Science and Technology*, vol. 70, no. 16, pp. 2237–2241, 2010.

[4]   G.Q. Han, J.H. Shen, X.X. Ye, B. Chen, H. Imai, K. Kondoh and W.B. Du, "The influence of CNTs on the microstructure and ductility of CNT/Mg composites", *Materials Letters*, vol. 181, pp. 300-304, 2016.

[5]   M. Zhu, Z. Jian, G. Yang and Y. Zhou, "Effects of T6 heat treatment on the microstructure, tensile properties, and fracture behavior of the modified A356 alloys", *Materials and Design*, vol. 36, pp. 243–249, 2012.

[6]   K.T. Akhil, S. Arul and R. Sellamuthu, "The effect of heat treatment and aging process on microstructure and mechanical properties of A356 aluminium alloy sections in casting", *Procedia Engineering*, vol. 97, pp. 1676–1682, 2014.

[7]   E. Sjölander and S. Seifeddine, "Optimisation of solution treatment of cast Al-Si-Cu alloys", *Materials and Design*, vol. 31, pp. S44–S49, 2010.

[8]   S. Menargues, E. Martín, M.T. Baile and J.A. Picas, "New short T6 heat treatments for aluminium silicon alloys obtained by semisolid forming", *Materials Science and Engineering A*, vol. 621, pp. 236–242, 2015.

[9]   M. Zeren, "Effect of copper and silicon content on mechanical properties in Al-Cu-Si-Mg alloys", *Journal of Materials Processing Technology*, vol. 169, no. 2, pp. 292–298, 2005.

[10]   K. Kashyap and T. Chandrashekar, "Effects and mechanisms of grain refinement in aluminium alloys", *Bulletin of Materials Science*, vol. 24, no. 4, pp. 345–353, 2001.

[11]   S.G. Shabestari and M. Malekan, "Assessment of the effect of grain refinement on the solidification characteristics of 319 aluminum alloy using thermal analysis", *Journal of Alloys and Compounds*, vol. 492, no. 1-2, pp. 134–142, 2010.

[12]   B.M. Afshari, S.S. Mirjavadi, Y.A. Dolatabad, M. Aghajani, M.K.B. Givi, M. Alipour and M. Emamy, "Effects of pre-deformation on microstructure and tensile properties of Al—Zn—Mg—Cu alloy produced by modified strain induced melt activation", *Transactions of Nonferrous Metals Society of China*, vol. 26, no. 9, pp. 2283–2295, 2016.

[13]   D.A. Lados, D. Apelian and L. Wang, "Solution treatment effects on microstructure and mechanical properties of Al-(1 to 13 pct) Si-Mg Cast Alloys", *Metallurgical and Materials Transactions B*, vol. 42, no. 1, pp. 171–180, 2011.

[15]   J.H. Peng, X.L. Tang, J.T. He and D.Y. Xu, "Effect of heat treatment on microstructure and tensile properties of A356 alloys", *Transactions of Nonferrous Metals Society of China*, vol. 21, no. 9, pp. 1950–1956, 2011.

[16]   Y. Birol, "Semi-solid processing of the primary aluminium die casting alloy A365", *Journal of Alloys and Compounds*, vol. 473, no. 1-2, pp. 133-138, 2009.

[17] H. Hashim, M.S. Salleh, M.Z. Omar and A.B. Sulong, "Effects of mechanical stirring and short heat treatment on thixoformed of carbon nanotube aluminium alloy composite", *Journal of Alloys and Compounds*, vol. 788, pp. 83–90, 2019.

[18] A.M.A. Mohamed and F.H. Samuel, "A review on the heat treatment of Al-Si-Cu/Mg casting alloys", in *Heat Treatment - Conventional and Novel Applications*, F. Czerwinski. Hamilton, Ontario: InTechOpen, 2012, pp. 55-72.

[19] E.J. Zoqui, "Alloys for Semisolid Processing", *Comprehensive Materials Processing*, vol. 5, no. 9, pp. 163-190, 2014.

[20] K. Abedi and M. Emamy, "The Effect of Fe, Mn and Sr on the Microstructure and Tensile Properties of A356-10% SiC Composite," *Material Science and Engineering A*, vol. 527, no. 16-17, pp. 3733-3740, 2010.

[21] M. Snajdar and Z. Schauperl, "ECAP-new consolidation method for production of aluminium matrix composites with ceramic reinforcement", *Processing and Application of Ceramics*, vol. 7, no. 2, pp. 63–68, 2013.