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Damage sensing and mechanical properties of laminate composite based MWCNTs under anticlastic test

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THE MANUFACTURE AND CHARACTERIZATION OF ALUMINUM ALLOY METAL MATRIX NANO COMPOSITES

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ABSTRACT

In this research the Composites of A356- Nano Al_2O_3 reinforced with different Wt. % were fabricated by (semi solid) double stir casting technique in order to improve the wettability and distribution of reinforcement particles within the matrix,. Semi-solid stir casting (SSC) techniques have proven useful in the mass production of high integrity castings for the automotive and other industries. In this process, the A356 aluminum alloy is heated to above its molten temperature. The melt is then cooled down to ($580^{\circ}C$) a temperature between the liquid and solid points to a semi-solid state. At this point the preheated Nano Al_2O_3 reinforcement particles are warped with aluminum foil added and mixing at 450 r. p.m. the slurry is heated to a fully liquid state once again and mixed thoroughly for 30 mints. After applying Full heat treatments (T6) on a part of cast Nano composite ingots will provide for the samples; solution treatment consist of heating in an electrical furnace at $540^{\circ}C$ ($\pm 1^{\circ}C$) for 6 hours and then quenching in water at room temperature. The Artificial aging treatment done at $180^{\circ}C$ for 3 hours. The effect of heat treatment (T6) on the microstructure and mechanical properties of the as caste and A-356 aluminum matrix composites was studied. The microstructure ,porosity and distribution of Al_2O_3 nanoparticles for all samples A356 alloy were studied by optical microscope (OM) equipped with image analyzer, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD), Also, the hardness and tensile strength of samples was investigated. The results showed, after full heat treatment the microstructure consists of hard intermetallic Mg_2Si . The structure of the eutectic Si form to crash and spheroid sing and the Si particle become rounded). Significant agglomeration can be found in composites with different reinforcement percentage Nano particles Al_2O_3 . Porosity will increase with the increase in the volume fraction of Nano Al_2O_3 . Hardness, yield strength and tensile strength increase, but the elongation decreases with the increase in the wight fraction of the Al_2O_3 particles, indicating that increasing the wight fraction of the Al_2O_3 particles can improve the strength but degrade the plasticity of the composites. The mechanical strength of

composite A356 alloy increased considerably by combining T6 heat treatment and different Wt. % of Nano- Al_2O_3 particle reinforced aluminum alloy.

Key words:-A356 alloy, composite material, semi-solid technique, heat treatment, Nano alumina.

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1. INTRODUCTION

Metal matrix composites (MMCs) reinforced by Nano-particles are very promising materials, suitable for a large number of applications. These composites consist of a metal matrix filled with Nano or micro -particles featuring mechanical properties more different from those of the matrix. The Nano-particles can improve the base material in terms of mechanical strength, fracture and fatigue, damping properties, wear and corrosion resistances [1-3].

Al-Si alloys such as A356 Aluminium alloy with Si as the major alloying element form a class of material providing the most significant part of all casting manufactured materials. This alloy has a wide range of applications in the automotive and aerospace industries due to an excellent combination of castability and mechanical properties, good corrosion resistance and wear resistivity. Additions of minor alloying elements such as Mg improve the mechanical properties and make the alloy responsive to heat treatment. [4] Stir casting technique is generally accepted as an alternative low cost and simple route which can be successfully used for mass production of AMCs. Due to particle agglomeration and clustering problems, achieving a uniform dispersion of nanoparticles is one of the principal purposes in the fabrication of AMNCs with stir casting methods. A recent development in stir casting process is a double stir casting or Semi-solid process (semi –solid state) [5, 6]. This process, the matrix material is first heated to above its molten temperature. The melt is then cooled down to a temperature between semi-solid states. At this point, the preheated reinforcement particles are added and mixed. Once again, the slurry is heated to a fully liquid state and mixed thoroughly. In double stir casting the resulting microstructure has been found to be more uniform as compared with conventional stirring [7-9]. The potency of this two-step mixing method is mainly due to its ability to break the gas layer around the particle surface which otherwise impedes wetting between the particles and molten metal. Thus, the mixing of the particles in the semi-solid state helps to break the gas layer because of the abrasive action due to the high melt viscosity. Furthermore, particles tend to sink or float according to their density relative to the liquid metal or will be pushed by the solidification front. Although it is a challenging subject, it would be advantageous to improve and optimize stir casting process to commercialize the production process of low-cost as cast bulk light weight high performance components of AMNCs [10, 12]. Reinforcement of A356 alloy by nanoparticles and heat treatment are the two main processes used to improve the properties of this alloy. Attempts are made to increase the strength of Al-Si-Mg by various manufacturing processes, heat treatment and reinforcement of hard particles. The use of ceramic particles to improve the yield and ultimate strength (UTS) of cast Al alloys has been investigated by many researchers [13, 14]. In recent years, widely used nanoparticles as refining and reinforcement agents to gain the improved performance of A356 cast alloys by adding Al_2O_3 , Sic ,carbon Nano tube and TiO_2 particles have gained significant interest recently [15]. The Nano-particles can improve the base material in terms of mechanical strength, fracture and fatigue behavior, damping properties, and wear and corrosion resistances [16-18].

Nano Al_2O_3 particles with high specific stiffness, superior high temperature and excellent mechanical properties are used as ceramic reinforcement phases in the A356 alloy. Eutectic

structure of A356 can be refined and its properties can be improved by optimized heat treatment [19, 20]. T6 heat treatment is usually used to improve fracture toughness and yield strength. It was reported that the factors influencing the efficiency of heat treatment of Al-Si hypoeutectic alloys include not only the temperature and holding time, but also the as-cast microstructure and alloying addition [22-24].

The T6 heat treatment of A356 alloy includes two steps: solution heat treatment and artificial aging. The solution heat treatment, generally, is carried out at a temperature slightly under the eutectic temperature at about 540°C for (4 to 8) hours. This treatment dissolves the hardening elements (Mg and Si) in the Al matrix, homogenizes the casting and spheroidizes the eutectic silicon. After the solution treatment, castings are quenched from this high temperature in order to avoid the precipitation of dissolved elements and to create a supersaturated solid solution at room temperature, an essential condition for further aging. Artificial aging is the step where the hardening elements are precipitated in order to improve the strength of the aluminum matrix. This process is carried out at relatively low temperatures (150°C - 200°C) [24-28].

The aim of the present investigation is to study the effect Nano particles at different volume fraction reinforcement and heat treatment on the microstructure and mechanical properties of A356 /Al₂O₃ nano composites Fabricated by double stir casting technique(semi solid technic) was investigated.

2. EXPERIMENTATION

2.1. Manufacturing process of nano composite -aluminum alloy

The manufacture of Nano composite -aluminum alloy was done at Mechanical Engineering school in Plymouth University. Bottom pouring stir casting furnace as shown in figure (1) was used to manufacture the nano composite -aluminum alloy.

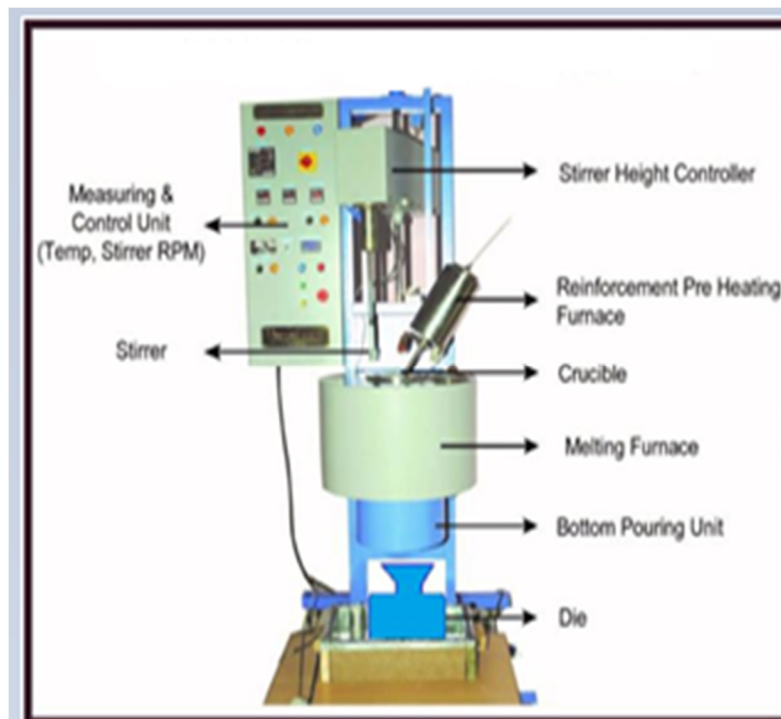


Figure 1 Bottom pouring stir casting furnace.

Bottom pouring stir casting furnace process was performed in a stainless steel crucible placed in a resistance furnace. While the stainless steel crucible was fixed in the middle of furnace, a hole was created in the bottom of the crucible for bottom pouring of the composite slurry. The

hole was closed during the melting, injection and stirring process with a stainless steel stopper. Also, a high frequency stainless steel stirrer system was placed on the top of the furnace. Injection of the reinforcement particles into the melt was carried out using a stainless steel injection tube and inert argon gas. In this part of equipment, the reinforcement powder is placed in a chamber and injected to the melt by pressure of the inert gas. This chamber also has the ability to heat treat the particles in an inert atmosphere before the injection process started. (Semi-solid) Stir casting technique was used to fabricate the A356 alloy reinforced with (0.5-1-2) wt. % nano- Al_2O_3 composites. In table (1) lists the chemical composition of A356 alloy used as a matrix and the table (2) shows the properties of nano- Al_2O_3 particles with average size of 60 nm used as reinforcement powders.

Table 1 The chemical composition of A 356 alloy

Alloy	weight %							
	Al	Si	Mg	Fe	Mn	Ti	Zn	Cu
A356	Bal.	7.44	0.53	0.27	0.22	0.01	0.01	0.2

Table 2 Properties of Nano alumina [20]

Reinforcement type	γ - Al_2O_3
Average density (g/cm ³)	3.6
Young's Modulus (GPa)	380
Average particle size (nm)	60
Melting Point (°C)	2045
Hardness Gpa	18-23

The experiment of Nano composite A356/ Al_2O_3 was done by employing the semi-solid stir casting technique. In this case, the A356 alloy played a role as matrix. The Al_2O_3 played a role as the reinforcement in the A356 alloy matrix. The variations of nano Al_2O_3 employed are 0.5, 1, and 2 wt. %. To manufacture the nano composite aluminum alloy 1 kg of A356 aluminum alloy charge was used and then heated to the temperature 800°C in a Stainless-steel crucible inside the furnace to get a liquid state. On the other hand, the temperature was lowered to 580 °C to get a semi- solid state. Meanwhile, different wt. % nano alumina powders was heated by reinforcement preheating furnace as shown in figure (1) at a temperature of 550 °C for 2 hours. After that, the nano Al_2O_3 was added to the electric stir casting furnace by injector tube made of quartz and stirred thoroughly by using a mechanical mixer. The mixing speed of the mixer was 450 rpm for 30 mins. After all materials were mixed and stirred in semi-solid state, the temperature was increased to 750 °C to get the casting condition as shown in figure (2) .

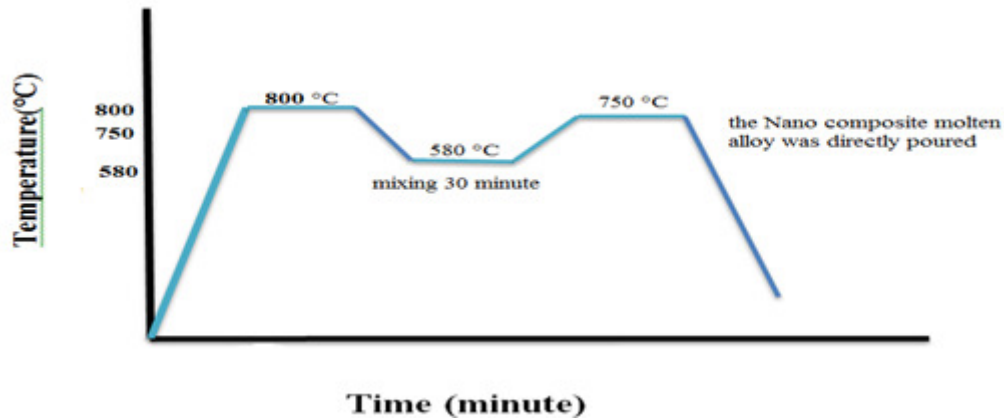


Figure 2 The semi-solid graph of temperature and time

The composite of mixture melted, and the nano particles started to distribute around the alloy sample. The metallic mold was also heated to the temperature 350 °C, and the Nano composite molten alloy was directly poured from the bottom to a st-steel die casting at 750 °C as illustrated in figure(3). Casting product was cooled at room temperature at 25 °C. After that, the Nano composite ingots were cut according to the standard test.

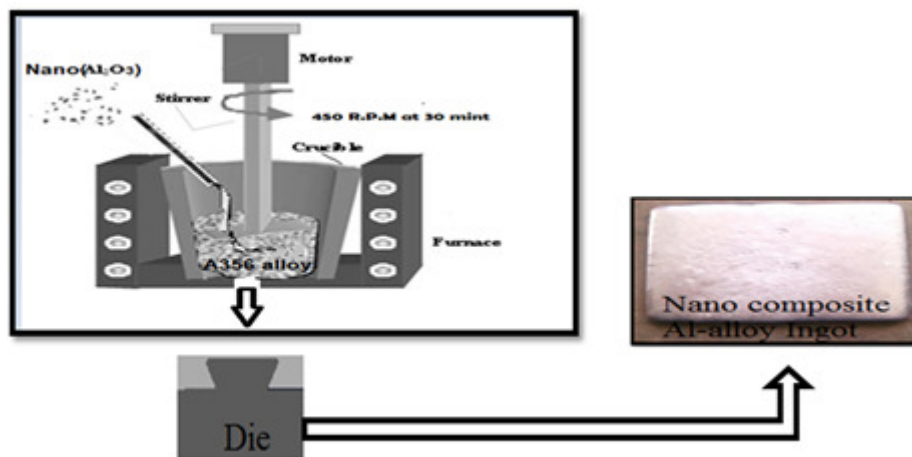


Figure 3 illustration of the casting process.

2.2 Density and porosity measurement

The density of Nano composites alloy was measured experimentally using the typical Archimedes (water displacement) method according to ASTM B311-08 (26). The specimens for the density test had the size of 2 x 2 x 2 cm cut from the composite alloy and weight them first in air and then in water by using digital balance of ± 0.0001 g accuracy. The theoretical density was calculated using the mixture rule according to the weight fraction of the nano particles. The porosities of the produced composites were evaluated from the difference between the expected and the observed density of each sample according to equation 1 below:-

$$\left[1 - \frac{\rho_{exp}}{\rho_{th}}\right] \times 100 \% \quad (1)$$

Where,

ρ_{th} : is the theoretical density of the composite (g/cm^3).

ρ_m : is the density of the matrix (g/cm^3).

2.3 Samples preparation, heat treatment and testing

Samples from the cast Nano composite ingots were designed by solid work and machined by computer added manufacture (CAM) by different mining machining. After machining, the samples were heat treated T6 using electric furnace. The T6 heat treatment for specimen included solution heat treating at 540 °C for six hours and quenching in water at room temperature. After cooling specimens were artificially aged for 3 hours at 180 °C as, shown in figure (4).

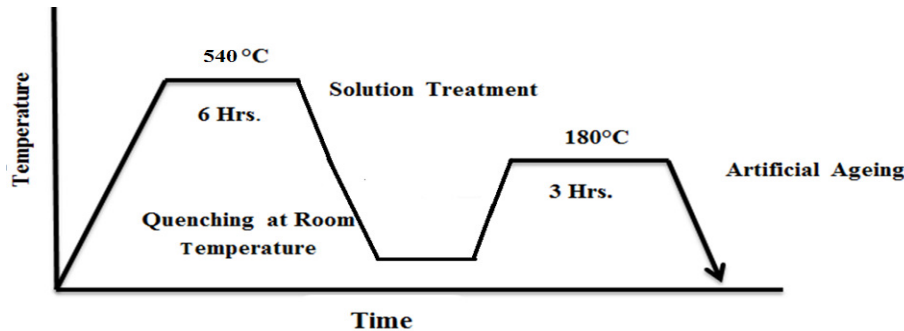


Figure 4 Typical T6 heat treatment cycle used for A356 cast alloy.

The microstructure for heat treated Nano composite aluminum alloy samples was investigated by optical microscopy (OLUMPES N334) as shown in figure (5) and (Scanning electronic microscopy (SEM, model: JEOL-7001-FE-SEM) linked to Energy Dispersive Spectrum (EDS) by INSPECT F50 FE-SEM, and the phase identification was examined by X-ray diffraction (XRD). The specimens were prepared by grinding through 200, 400, 600, 800 and 1200 grit emery papers followed by polishing with 6µm alumina paste and 1 µm diamond paste and finally polished by electro polishing machine.as shown in figure (6).



Figure 5 Optical microscope



Figure 6 Electro polishing machine

Micro hardness was measured on polished samples using the (OLYMPES) micro hardness tester with a high digital resolution camera shown in figure (7). The tests were carried out by applying an indentation load of 0.3Kg with a Vickers indenter. Five readings were conducted for each specimen directly and the average value was considered.

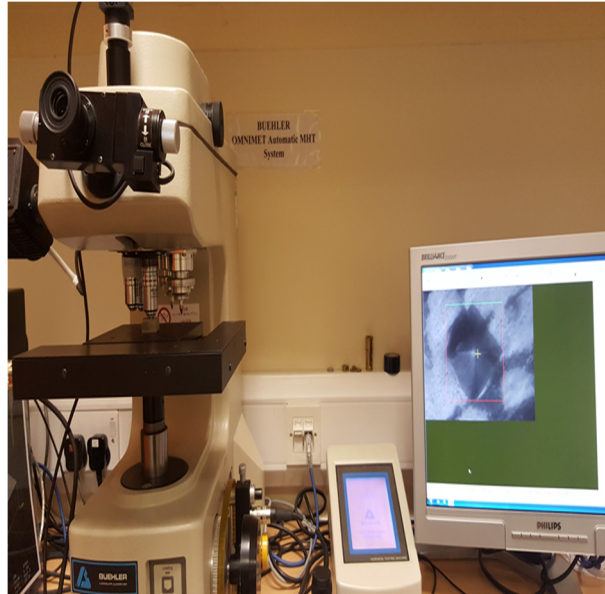


Figure 7 Micro hardness tester

The tensile tests were carried out using an Instron machine type 5582 100 kN universal testing machine as shown in figure (8), and the samples prepared according to ASTM E8 was used in all tensile tests at 1 mm/min. The ultimate tensile strength (UTS), 0.2% yield stress (YS) and ductility (%El) were measured, and the elongation % was calculated for each reinforcement nano alumina specimen percentage. Minimum three specimens were tested, and the average value was calculated. All mechanical tests and Microstructure examinations were conducted at the Mechanical Engineering School in Plymouth University.



Figure 8 Tensile test machine

3. RESULTS AND DISCUSSION

3.1. Density and porosity

Porosity is a defect formed by interfacial reactions, which cause a decrease in the mechanical properties of metal matrix composite. Operating stirring process with a high velocity will form a swirl on the surface of the slurry. The expansion of the swirl is noticed to be helpful for conveying the particles into the matrix melt as the pressure difference between the inner and the outer surface of the melt absorbs the particles into the liquid. This will cause porosity formation in the slurry

[26-27]. Table (3) presents with the comparison of theoretical density obtained by rule of mixture and measured density values by experiment and percentage of porosity for different filler percentages for Al₂O₃ Nano particles composites alloy, it can be noticed that the densities of composites are higher than that of their base matrix and the experimental densities are lower than the theoretical densities, further the density increases with increased percentage of filler content in the composites.

Table 3 Theoretical and actual densities and percentage of porosity

Composites (wt. %)	Theoretical density (g/cm ³)	Actual density (g/cm ³)	%Porosity
A356 /Al ₂ O ₃ 0 %	2.70	2.67	1.111
A356 /Al ₂ O ₃ 0.5%	2.78	2.74	1.800
A356 /Al ₂ O ₃ 1 %	2.76	2.69	2.508
A356 /Al ₂ O ₃ 2 %	2.84	2.73	3.873

Form the same table 3, it can be seen that the percentage of porosities of the Nano compost alloy product are between 1.800 – 3.873 % which is within the acceptable range of 5% porosity reported in literatures as the maximum Allowed persntage in cast metal matrix composites [26-28]. The porosity content increases with the increasing filler ratio of reinforcement material, as shown in figure 9

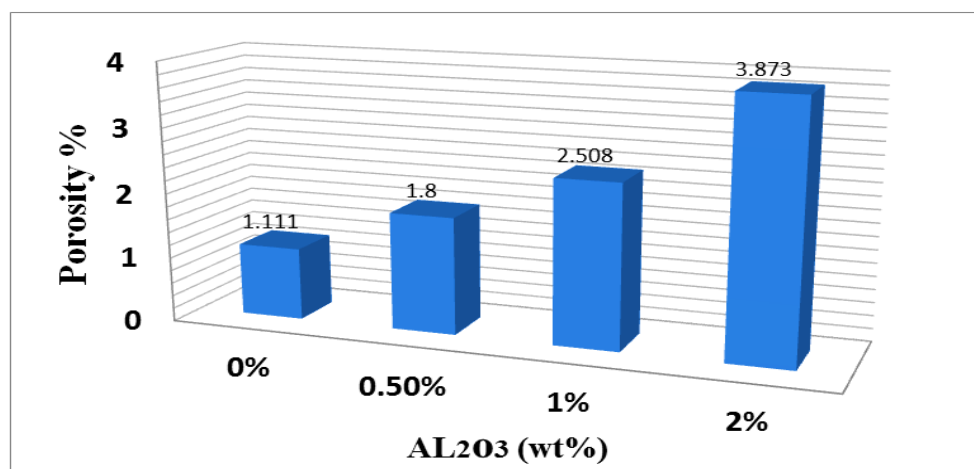


Figure 9 level of porosity composite

The figure shows that the increasing of porosity volume percent has occurred with increasing alumina weight fraction. This is because of low wettability and agglomeration at high filler percentage of reinforcement and pore nucleation at the matrix– Al₂O₃ interfaces. Also, decreasing the liquid metal flow associated with the particle clusters caused the formation of porosity moreover the hydrogen content of molten increases the viscosity due to particles addition and gas entrainment inside the melt and preventing them from floating before it solidifies.

3.2. Microstructure analyses

The evolution of the microstructure of A356 as-cast and nano composite aluminum alloy during the (T6) heat treatment was investigated in details. The temperature of solution treatment is 540°C. For 6 hours and the artificial ageing is at 180°C for 3 hours. The changes in dendritic

composition and eutectic morphology due to solution, aging treatment and distribution of nano particles in structure were quantified by optical and image analyzer and scanning electron microscope with EDS for a wide range of processing conditions. The microstructure of the as-cast A356 consists of a primary phase, α -Al solid solution, and a eutectic mixture of aluminum and silicon as shown in figure 10 a

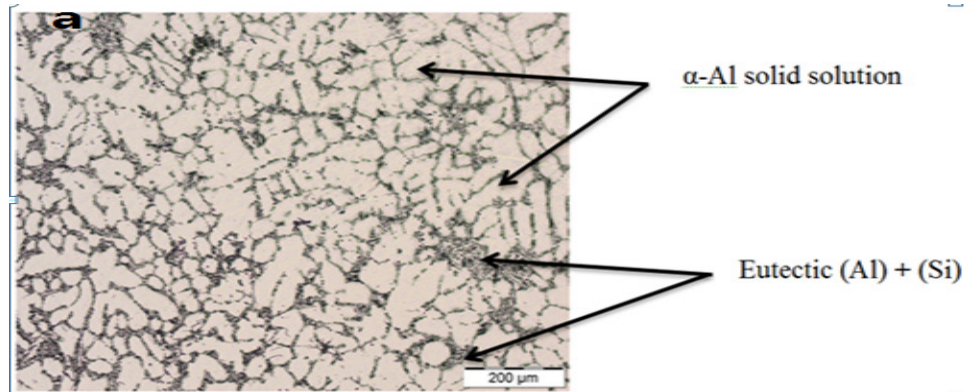


Figure 10 Microstructure of A 356 aluminum alloy.

As shown in Figure (11) (a,b). The primary phase are solid solution and irregular eutectic Al-Si precipitates from the liquid in the form of dendrites containing the Mg_2Si phase. These particles are very small (less than $0.5\mu\text{m}$) [22] and α -AlFeSi particles as shown in figure 12 EDS analyses. The Mg_2Si particles are dissolved during the solution treatment and release magnesium atoms into the solution of the aluminum matrix [22].

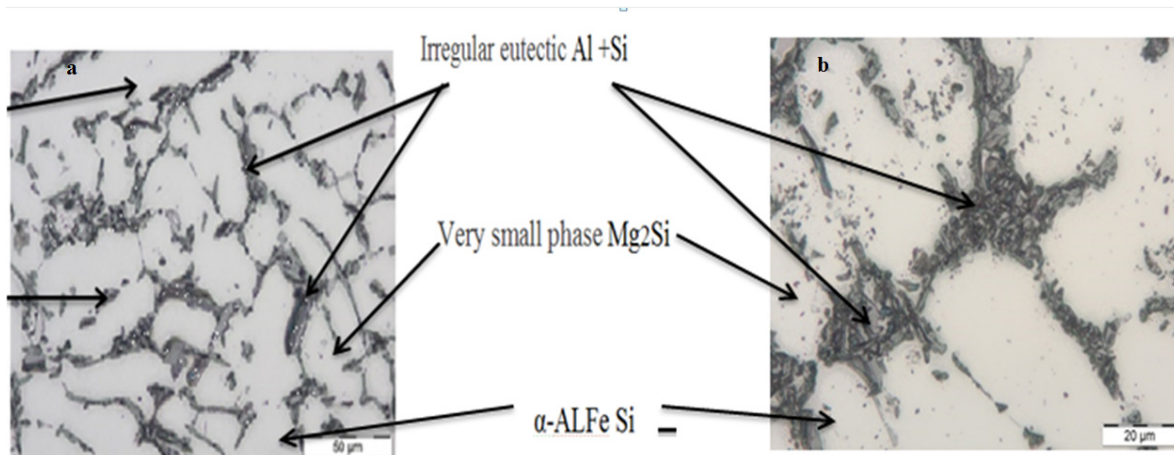


Figure 11 (a,b). Precipitation second phases (a) low magnification (b) High magnification

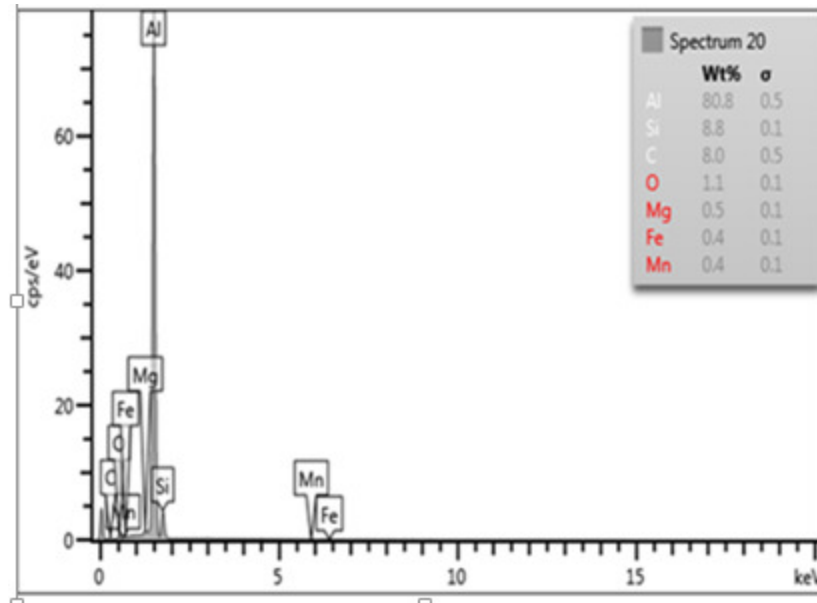


Figure 12 EDS spectrum of the sample (1Wt % Al_2O_3).

For Solution treatment, it is observed that a large quantity of the silicon has spheroidized and the coarsening process has begun. As the end of treatment after 6 hours the silicon has completely become spheroidized and refining takes place as shown in figure (13).

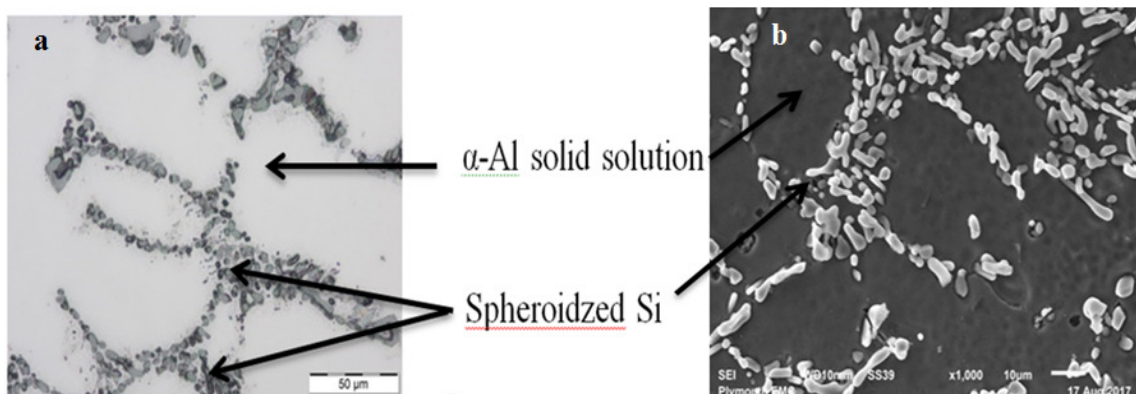


Figure 13 uniformly distributed spheroidized silicon in the Al matrix (a) optical micrograph (b) SEM.

Similar result was reported in literature of semi-solid casting of A356 (16-21-22). The subsequent quenching will form a supersaturated solid solution and trap excess vacancies and dislocation loops which can later act as nucleation sites for precipitation. The precipitates, however, will form quickly at elevated temperatures, at 180°C (artificial aging). Figure 14 (a,b,c) represents the SEM distribution of nano composite containing different percentages of Nano- Al_2O_3 from 0.5 to 2 wt. % respectively. It has been observed that the distribution of particles in the composite samples is uniform and the un-uniformity increases by increasing the reinforcement percentage above 2 wt. %. The increase in the reinforcement percentage causes clustering and agglomeration of particles due to increase in surface energy and weakens the bonding between reinforcement and matrix thereby wettability decreases.

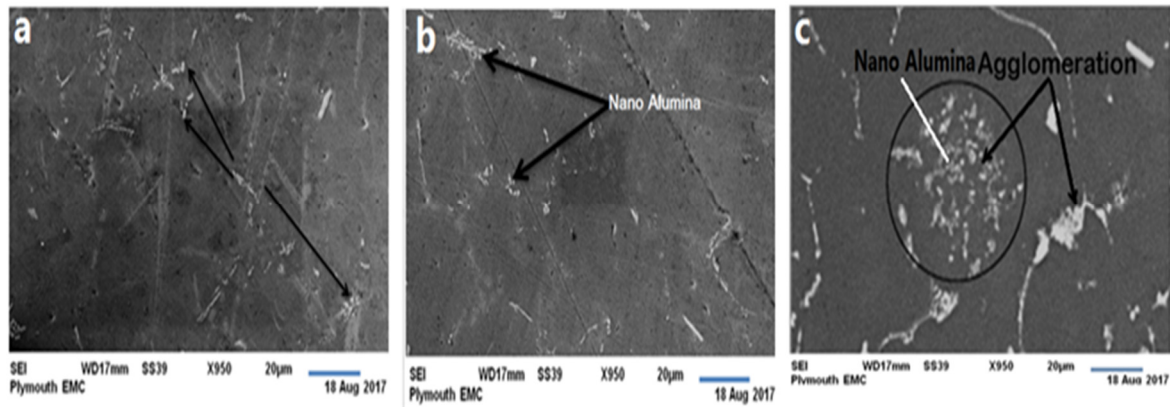


Figure 14 Typical SEM images for nano particles distribution (0.5 ,1,2)Wt.% Nano Al_2O_3 (a,b,c) respectively .

In Fig. (15), it is noticed that there is un- uniformity distribution and agglomeration of Al_2O_3 particles for 2 wt. %. Also the refinement of dendritic structures may result in better improvement of properties.

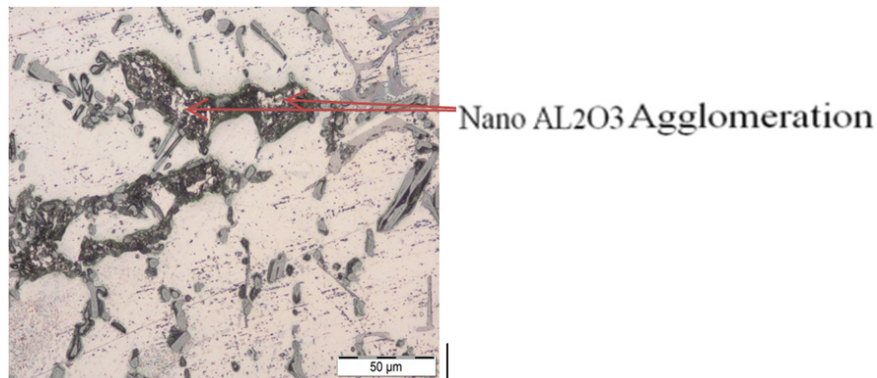


Figure 15 Optical micrograph of (2 Wt. %) nano alumina

3.3. Mechanical properties

3.3.1. Tensile test

The average values of the ultimate tensile strength, yield strength, and % elongation obtained from the tensile test are summarized in table (4).

Table 4 Yield strength, ultimate tensile strength and % elongation values of the Nano-composite alloy

wt.% Al_2O_3	Yield strength YS (MPa)	Ultimate tensile strength UTS (MPa)	Elongation (%)
As cast	85	125	6
0.5	102	161	4
1	116	210	3.3
2	135	245	2.6

Using T6 treatment in this study and different reinforcement by nano alumina, strength is improved significantly. As shown in figure (16), the yield strength and ultimate tensile strength increase, but the elongation decreases with the increase in the weight fraction of the Nano alumina particles, indicating that increasing the weight fraction of the nano alumina particles can improve the strength but decrease the plasticity of the composites. Also figure (16) shows that the increase is more significant for the Al cast with 2%. The increase in tensile strength is due to the presence of the hard and higher modulus Al_2O_3 particles embedded in the Al (356) matrix which act as a barrier to resist plastic flow when the composite is subjected to strain from an applied load. Also, the decreased inter particle spacing, due to the increasing weight percent of Al_2O_3 reinforcement, creates increased resistance to dislocation motion, which contributes to the enhanced strength of the composites [22].

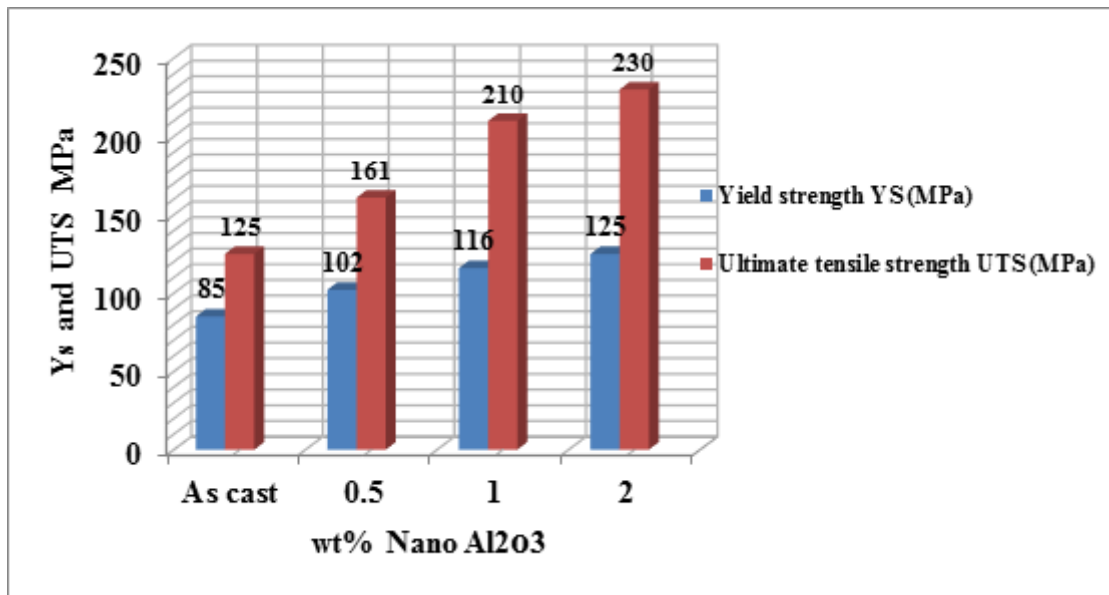


Figure 16 The effect of wt. % fraction of Al_2O_3 Nano-s in A356 on the (YS) UTS.

3.3.2. MicroHardness

Figure (17) summarised the micro hardness values for different weight fractions of nano Al_2O_3 reinforcement. It's noted that the micro hardness values increase along with percentage of reinforcement when compared with base alloy. Hardness value of A365 base alloy was 70. VHN. Composites reinforced with Al_2O_3 particles at 2 wt. % showed the highest hardness value (123VHN) while composites reinforced with Al_2O_3 particles at 0.5 wt. % showed the lowest hardness (88. VHN). The maximum observed increase in hardness of composites compared to unreinforced base alloy was due to the presence of hard Al_2O_3 particles, which aid to the load bearing capacity of the material and also restrict the matrix deformation by constraining the dislocation movement [18-19]. It may also be noted that T6 heat treatment is able to provide hardening effect by precipitation of constituents from the solid solution the precipitated constituents are believed to account for increase in hardness and also the increase in reinforcement results on increase of hardness [15].

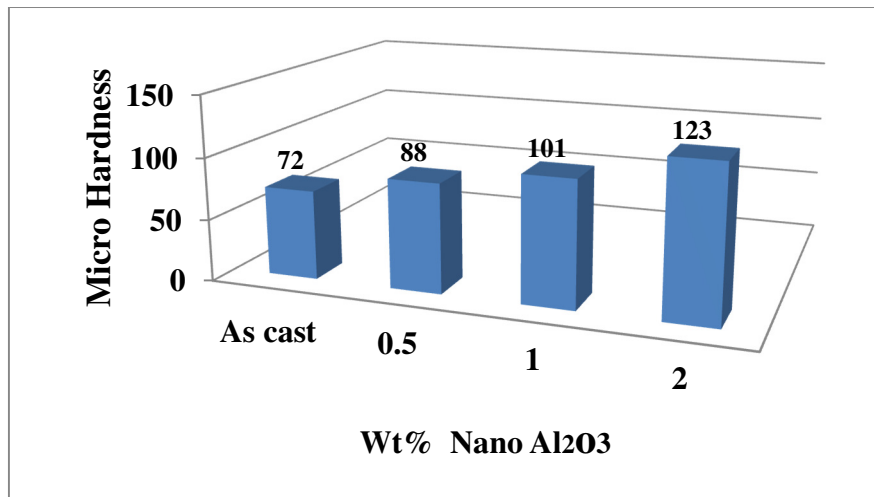


Figure 17 The effect of wt. % Nano Al₂O₃ on Hardness of MMC.

4 CONCLUSIONS

Semi solid stir casting technique is useful for manufacture nano composite alloy due to the following conclusion.

1. The solution treatment at 540 °C for 6 hours and aging at 180 for 3 hours can reach fulls periodization of Si particle, over saturation of Si and Mg in α (Al).
2. The distribution of nano particles is not uniform and can make cluster and agglomeration at high weight fractions reinforcement.
3. The porosity content increases with the increasing filler ratio of reinforcement material.
4. The T6 treatment and different wight fractions of nano alumina can improve strength and hardness but reduce plasticity elongation for A356 alloys nano composite.

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