JMME. Vol. 13, April, 2021
pp. 9 –20

ISSN: 2006-1919

EFFECT OF INTERRUPTED QUENCHING ON HARDNESS, TENSILE STRENGTH AND MICROSTRUCTURES OF AL-SI-MG/10% LOCUST BEAN WASTE ASH COMPOSITE

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ABSTRACT

This study investigates the potentials of Neem(AzadirachtaIndica) seed oil as quenchant for precipitation hardening of Al-Si-Mg/10% locust bean waste ash composite. This was with a view to improving the precipitation response leading to smaller grain size of the Al-Si-Mg/locust bean waste ash composite. The composite consists of a matrix of Al–Si–Mg and the locust bean waste ash particulates as reinforcement. The composite material was then subjected to thermal treatment through solution heat treatment at 540^{o}C, soaked for 1 hour and then quenched simultaneously in neem seed oil and water at 30^{o}C. Some of the quenched samples were delayed for 0.02hr, 0.5hr, 1hr, 1½hr 2hrs and 3hrs respectively and then age hardened at 180^{o}C for 2 hours, followed by air cooling in order to evaluate the properties as well as the resulting microstructures. The mechanical properties of the composites examined include the hardness and the tensile strength.The microstructures of the heat-treated composite were also examined using OPM and SEM while the phases developed were revealed using XRD. The results of the microstructures of the composite show a uniform dispersion of the reinforcement along the grain boundaries of the alloy. The results show that the composite with the intermediate natural ageing time of 3hrs has the highest properties in both quenchants. The hardness value and tensile strength in neem seed oil and water are 29.2HRF & 28.1HRF, 2.58 & 2.3kN/mm^2 respectively, which corresponds to 93.4% & 86.1%, 207% & 173.8% incrementin hardness and tensile strength. Within the α-aluminium solid solution, the phases are indicated by XRD with Mg_2Si, Mg_2Mn, $Al_{12}Mg_{17}$, SiAl and $Al_8Mg_3FeSi_6$ intermetallic compounds being formed. Thus, these confirmed the SEM and OPM microstructure examined which can be said to be present in the thermally treated composite leading to improved properties. From these results, it can be concluded that the locust bean waste ash particulates can be used to enhance the properties of Al–Si–Mg alloy using indigenously sourced quenchant for engineering applications.

1.0 INTRODUCTION

Metal matrix composites (MMCs) represent a relatively new class of materials characterized by lighter weight and greater wear resistance than those of conventional materials (Włodarczyk-Fligier, 2008). Metal matrix composites have potential advantages over monolithic alloys and this has activated considerable attention in the past years. These composites have provided solution to the problem of increasing service requirements for various structural applications through the reinforcement by metallic or non-metallic materials (Adeosun*et al.*, 2014). The driving force behind the development of most of the existing composites has been their capability to be designed to provide needed types of material behaviour. Discontinuously reinforced metal matrix composites have virtually isotropic properties and lend themselves to metallic design methodologies (Aigbodion and Hassan, 2007).

Among the various types of metal matrix composites, aluminium matrix composites (AMCs) have received considerable attention for military, automobile and aerospace applications because of their low density, high strength and high

stiffness relative to unreinforced materials (Mayas *et al.*, 2012). The major advantages of AMCs compared to unreinforced materials are improved high-temperature properties, controlled thermal expansion coefficient, thermal/heat management, improved wear resistance and improved damping capabilities (Suleiman *et al.*, 2018). In recent years, Aluminium alloys have been identified by many researchers, engineers and inventors as a capable structural material for different industries like aerospace and automotive (Xavior et al., 2015). The particle reinforced aluminium alloy composites which are among the most widely used composites materials are rapidly replacing the conventional materials in various industries like aerospace, marine, and automotive (Abdulwahab*et al*., 2015; Rohatgi*et al*, 2007).

Age hardening behaviour of aluminium metal matrix composite has been of great interest of present research. The nature of change in kinetics and magnitude of hardening during ageing of composites depends on matrix material, type of reinforcement including its size, shape and volume fraction and method of synthesizing the composite, post fabrication treatment and temperature of ageing (Mackenzie and Totten, 2000). It has been conclusively shown that the presence of ceramic reinforcement such as SiC/Al_2O_3 (whiskers, particle or short fibers) lead to acceleration in the ageing kinetics when compared with the unreinforced alloy. This behaviour generally has been attributed to enhanced nucleation and growth due to the presence of high matrix dislocation densities, which is generated due to the thermal mismatch between the matrix and the reinforcements (Ch-Shoba*et al.*, 2014). Proper selection of appropriate quenching media can result in improved mechanical properties of aluminummatrix composite after solution heat treatment (Odusote*et al.*, 2015).

Therefore, the present study investigates the influence of interrupted ageing time on the hardness, tensile strength and microstructures of Al-Si-Mg/10% Locust Bean Waste Ash composite using neem seed oil and water as quenchant.

2. MATERIALS AND METHODS

The materials used in this study are high purity aluminium wires which were obtained from Northern Cable company (NOCACO) Kaduna, Silicon and Magnesium powder obtained from NMDC Jos, Locust bean agro waste was gathered from Bomo Village of SabonGari Local Gov't Area of Kaduna State and neem seed oil was sourced from NARICT Basawa.

2.1 Equipment

The equipment used in this research include:

Metal mould, manual stirrer, electrical resistance furnace, graphite crucibles, digital weighing balance, XRD analyzer (Model XRD-320), Rockwell Hardness tester (Model PR-21), Hurston computerized tensile testing machine (Model PSPL-2000), Avery Denison impact testing machine (Model IT-30), grinding and polishing machine, optical metallurgical microscope with inbuilt camera connected to a computer system (model NJF -120A), simultaneous SEM/EDS analyzer (Model Topcon SM-300), Computer system with installed CAD software.

2.2 Methods

2.2.1 Locust Bean Waste Ash (LBWA) Preparation

The locust bean waste ash (LBWA) used was obtained from the burning of locust bean husks sourced from Bomo village of SabonGari Local Government Area of Kaduna state. The husks were completely burnt under controlled atmospheric condition within a temperature range of

700 – 800^{0}C for about 7 - 8hrs measured with a thermocouple. The ash obtained was sealed in plastic bags and transported to the laboratory. The ash was passed through British Standard No 200 sieve (75 µm aperture) and kept to be mixed with the Al-Si-Mg alloy in the appropriate percentages.

2.2.2 Production of Aluminium Matrix Composites

The aluminium matrix composites used in this research was produced using stir casting method at the Department of Metallurgical and Materials Engineering Workshop, Ahmadu Bello University, Zaria, Nigeria. The specimen was produced with constant percentage of aluminium, silicon and magnesium at 92.7%, 7% and 0.3% respectively with 10 wt.% locust bean agro waste ash.

High purity aluminium electrical wires were charged in the graphite crucible in the resistance furnace. After the melting of the aluminium, the temperature of the furnace was raised to about 700^{o}C for the purpose of superheating the aluminium melt. The required 7%Si and 0.3%Mg alloying elements were added to the melt. The molten metal was continuously stirred to ensure uniform distribution of the alloying elements. The Al-Si-Mg alloy was cast into cylindrical bars. The composition of the alloy is shown in Table 1.

Table 1: Composition of Al-Si-Mg Alloy produced (wt.%) using spectroscopy (spark test)

Al	Si	Mg	Fe	Cu	Mn	Zn	Ti	Na
92.69	7.0	0.3	0.08	0.03	0.03	0.03	0.03	0.01

Weighed amount of the alloy was then charged into the graphite crucible in the resistance furnace and re-melted. The melt was removed from the furnace and 10wt% of the preheated locust bean agro waste ash particles was added, mixed manually for about 3 minutes and then returned to the furnace at a temperature maintained between 680^{o}C and 700^{o}C for about 3 minutes. The produced composite was cast into bars of dimension of 20mm x 350mm using a split mould.

2.2.3 Thermal ageing treatment of the composite

One hundred and twenty Samples were subjected to ageing treatment consisting of solution heat treatment at 540^{o}C for 1hr, quenching, intermediate natural ageing at varying time of 1 minute, 30 minutes, 60 minutes, 90 minutes, 120 minutes and 180 minutes each, and then finally aged at180^{o}C for 120 minutes as shown Figure1.

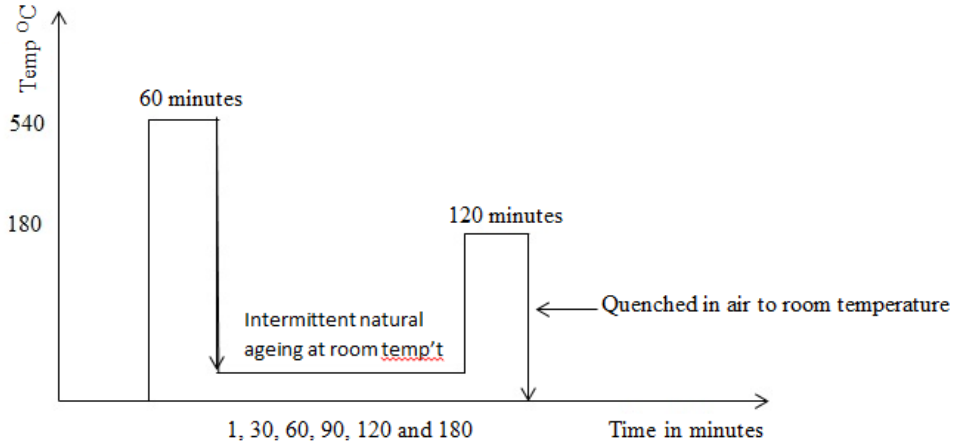

Figure 1: Schematic diagram of the thermal ageing heat treatment

2.3 Mechanical Testing

2.3.1 Tensile Strength Determination

The tensile properties test was conducted using a computerized Hurston Universal Testing Machine of load capacity 100kN. The tensile test was conducted in accordance with ASTM D3039-76 (2000) specification. The Al-Si-Mg/10wt%LBWA composite samples with dimension 100mm x 20mm x 3mm were prepared by making the specimen gauge length with pink punch mark and measuring the X – sectional area of the reduced section.

Tensile test was carried out by gripping the end of the specimen in a tensile testing machine and applying increase pull on to the specimen till it fractures. During the test, the tensile load as well as the elongation of a previously marked gauge length in the specimen was measured with the help of load dial of the machine and extensometer respectively. These readings help plot stress- strain curve. After fracture, the two pieces of the broken specimen were placed as if fixed together and the distance between two-gauge marks and the area at the place of fracture were noted.

2.3.2 Hardness Value determination

The hardness test was carried out by using the Rockwell hardness machine (Model PR-21 Durometer) in accordance with ASTM D2483 (2000) using 1.56mm steel ball indenter, minor load of 10kgf, major load of 100kgf and hardness value of 101.2HRB as the standard bloc. The sample has a dimension of 10mm x 3mm. Before the test, the mating surface of the indenter, plunger rod and test samples were thoroughly cleaned by removing dirt, scratches and oil. The samples were placed on the anvil, which act as a support for the test samples. A minor load of 10kgf was applied to the sample in a controlled manner without inducing impact or vibration and zero datum position will be established, and then major load of 100kgf was then applied. The reading was taken when the large pointer came to rest or had slowed appreciably and dwelled for up to 2 seconds. The load was then removed by returning the crack handle to the latched position and the hardness value read directly from the digital scale.

2.4 Microstructural Examination

2.4.1 Optical Microscope Examination

The produced composite specimens in as-cast and heat treated forms were prepared for metallographic examination. Standard method of specimen preparation for microstructural examination was adopted which includes; mounting, grinding, polishing and etching. The samples were mounted using a Bakelite, followed by grinding and polishing using silicon carbide abrasive papers different grit sizes. The samples were then etched in 0.5 ml hydrofluoric acid and 99.5ml of distilled water. The structure obtained was photographically recorded using metallurgical microscope with built – in camera.

2.4.2 Scanning Electron Microscope and Energy Dispersive Spectrometry Examination

The microstructures and the chemical compositions of the phase present in the test samples were studied using a scanning electron microscope (SEM). The samples were washed, cleaned thoroughly, air-dried and sputter-coated with 100 $\overset{o}{A}$ thick palladium to achieve good electrical conductivity. The SEM was operated with accelerating excitation energy of 10.0kV to 12.0kV. The digitized SEM images were recorded.

2.4.3 Phase Examination

X-ray diffractometer (XRD) was used to examine the elements and chemical

compositions/phases of the Al-Si-Mg/LBWA composites after being subjected to thermal treatment. The X-ray diffractograms were taken using Cu Kα radiation at scan speed of 3^o/min. The samples were rotated at precisely one-half of the angular speed of the receiving slit so that a constant angle between the incident and reflected beams is maintained. The receiving slit is mounted in front of the counter on the counter tube arm, and behind it is usually fixed a scatter slit to ensure that the counter receives radiation only from the portion of the specimen illuminated by the primary beam. The intensity of rays diffracted at the various angles were recorded automatically on a chart and the appropriate (θ) and (d) values were obtained.

3. RESULTS AND DISCUSSION

Tables 2 and 3 show the hardness and tensile test results of the heat treated composite quenched in oil and water respectively. It was observed that the hardness and tensile strength increase as ageing time increases with highest properties at 3hr of ageing time. From Table 2, the hardness value at 3hr of ageing time in both the water and oil are 29.2 and 28.1 HRF respectively, while Table 3also compares the tensile strength results of the water and oil quenched samples. The results show that the tensile strength increases as ageing time increases up to 3hr with peak tensile strength of 2.58 and 2.3kN/mm^2 in water and oil respectively. This trend is also depicted in figure 2. The XRD result is displayed in Figures 3 (a-f) which shows the distribution of the phases present in the produced composite. The phases present in the XRD on the result include Al_3Si_2, Mg_2Si, Si_8Ti, AlCaFe. These may have resulted in the appreciable mechanical properties experienced in the result.

Table 2: Comparison of hardness value between the samples of Al-Si-Mg/10%LBWA Composites quenched in water and oil

Ageing time (hr)	Water quenched (kN)	Oil quenched (kN)
0	21.9	21.1
0.5	22.4	21.8
1	23.7	22.2
1.5	27.8	26
2	28.9	27.8
3	29.2	28.1

Table 3: Comparison of tensile strength value between the samples of Al-Si-Mg/10%LBWA Composites quenched in water and oil

Ageing time (hr)	Water quenched (kN)	Oil quenched (kN)
0	1.56	1.51
0.5	1.78	1.7
1	1.92	1.84
1.5	2.23	2
2	2.35	2.11
3	2.58	2.3

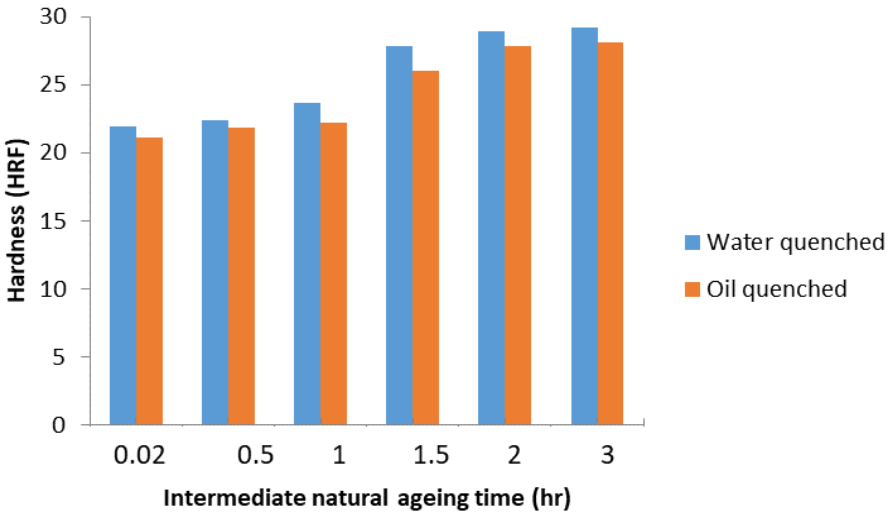

Figure 2: Comparison of hardness value between the samples of Al-Si-Mg/LBWA composites quenched in water and oil with intermediate ageing time.

3.1 Microstructural Examination

The distribution studies of LBWA in the matrix of Al-Si-Mg alloy are examined using optical photographic microscope (OPM) and scanning electron microscope (SEM). The micrograph shown in Plate I, represent the image of the unreinforced Al-Si-Mg alloy while Plates II-VII depict the microstructures of thermally treated Al-Si-Mg/10%LBWA composites at variable pre-ageing time. Plates VIII (a-e) shows micrographs of the SEM result. A uniform distribution of locust bean waste ash (LBWA) particles without voids and discontinuities can be observed from these micrographs along with good bonding between matrix material and LBWA. This observed result is due to the heating of LBWA particulates prior to dispersion and the addition of magnesium in small quantities during stirring which improved wettability between matrix and LBWA particles.

The microstructure of the unreinforced Al–Si–Mg alloy is shown in Plate I. The structure consists of essentially α-Al matrix and Mg_2Si and Al_6SiMg4 intermetallic compounds distributed within the grains (Oghenevweta *et al.*, 2016).

Plates II - VII show the microstructures of the heat-treated composites. The microstructures consist of small phase discontinuities (phase heterogeneity) and a reasonably uniform distribution of locust bean waste ash particles in the matrix (phase homogeneity).

It was also observed that the reinforcing phase is shown as dark, while some of the metal phases are either shown as black or white (see Plates II - VII). Besides, the microstructures also show a tangible agglomeration and segregation of the ashed reinforcing particles in the Al–Si–Mg/10%wt. locust bean waste ash particle as the pre-ageing extends to 3hrs (see Plate VII). But other developed and heat treated composites (Plates II-VI) show reasonably good distribution of the locust bean waste ash particles in the Al–Si–Mg alloy. Because of the wettability of magnesium in the matrix excluding the mechanical stirring, there is a tendency of the Al–Si–Mg alloy with the reinforcing particles to experience interfacial bonding. These occurrences are in agreement with the previous reports (Abdulwahab *et al*., 2015; Siddharth*et al.*, 2017; Oghenevweta *et al.*, 2016; Saravanan *et al.*, 2014 and Aigbodion *et al.*, 2010).

Plate I: Micrograph of unreinforced as-cast Al–Si–Mg alloy, showing magnesium silicide, (Mg_2Si) (black) and α-Al matrix (white). x100, acid etchants (0.5ml HF acid and 99.5ml distilled water).

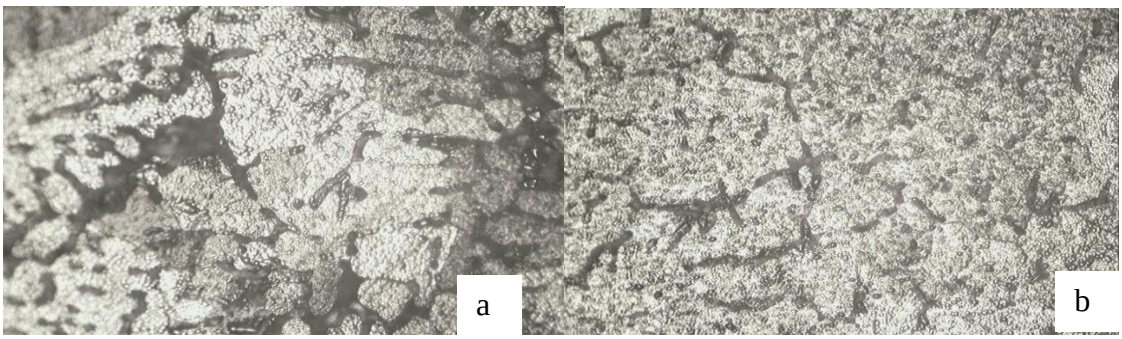

Plate ***II: Micrograph of Al–Si–Mg/10%LBWA composite, (0.02 hrintermediate natural ageing time); (a) oil quenched (b) water quenched, showing Mg_2Si (black) and uniform dispersion of the LBWA particulate along the grain boundaries (black) and in the grains of the α-Al matrix (white). x100, acid etchants (0.5ml HF acid and 99.5ml distilled water).***

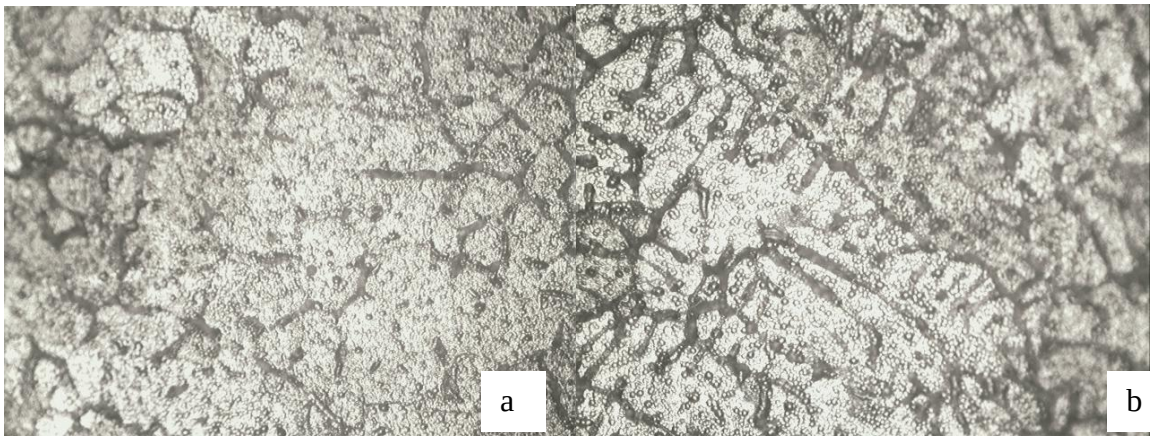

Plate III: Micrograph of Al–Si–Mg/10%LBWA composite, (0.5hrintermediate natural ageing time); (a) oil quenched (b) water quenched. (white). x100, acid etchants (0.5ml HF acid and 99.5ml distilled water)

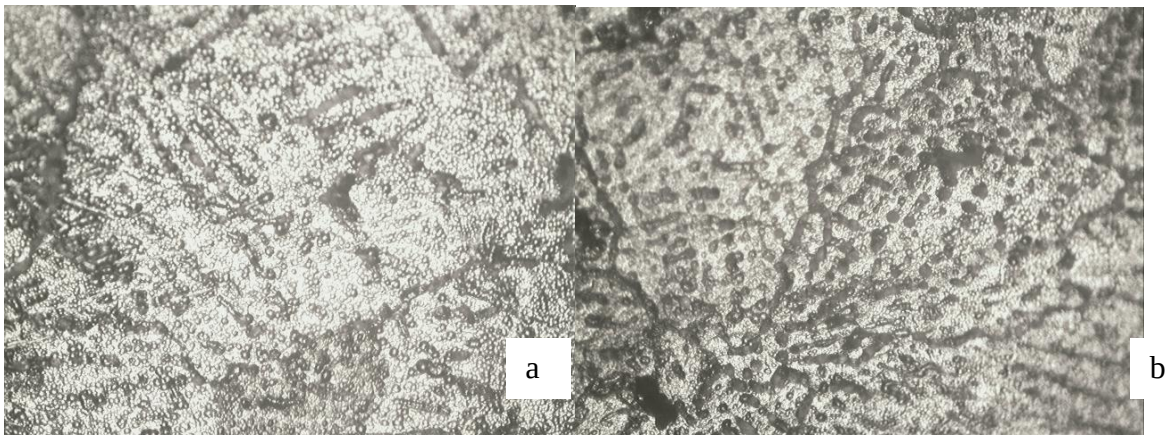

Plate IV: Micrograph of Al–Si–Mg/10%LBWA composite, (1 hrintermediate natural ageing time); (a) oil quenched (b) water quenched. x100, acid etchants (0.5ml HF acid and 99.5ml distilled water)

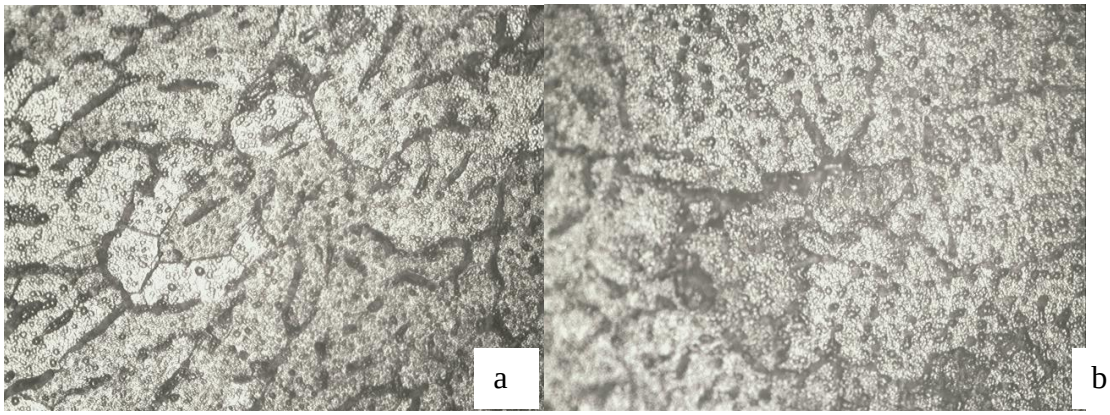

Plate V: Micrograph of Al–Si–Mg/10%LBWA composite, (1½hr intermediate natural ageing time); (a) oil quenched (b) water quenched. x100, acid etchants (0.5ml HF acid and 99.5ml distilled water)

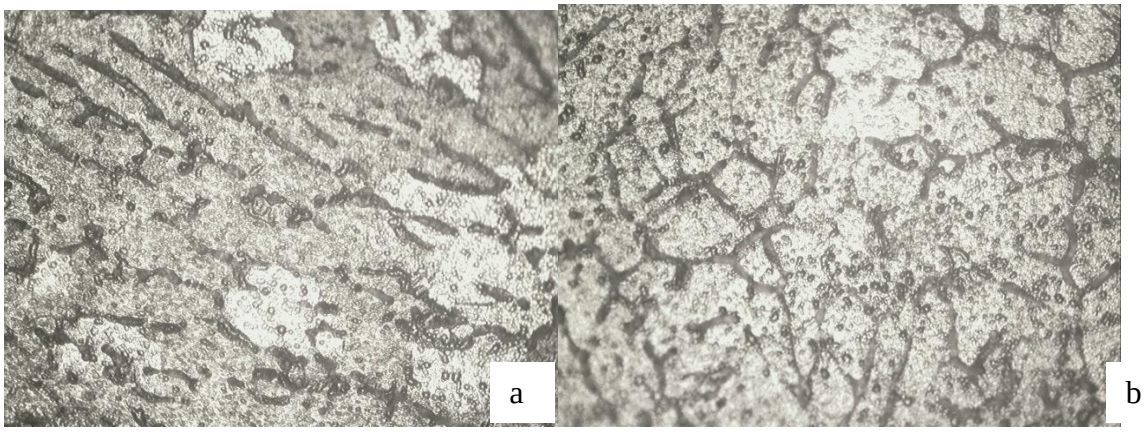

Plate VI: Micrograph of Al–Si–Mg/10%LBWA composite, (2hrs intermediate natural ageing time); (a) oil quenched (b) water quenched. x100, acid etchants (0.5ml HF acid and 99.5ml distilled water).

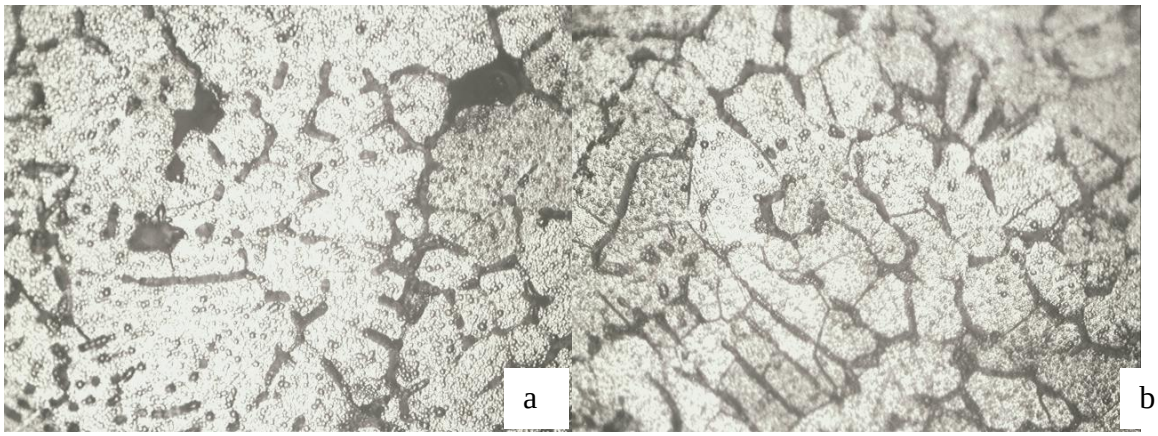

Plate VII: Micrograph of Al–Si–Mg/10%LBWA composite, (3hrsintermediate natural ageing time); (a) oil quenched (b) water quenched. x100, acid etchants (0.5ml HF acid and 99.5ml distilled water)

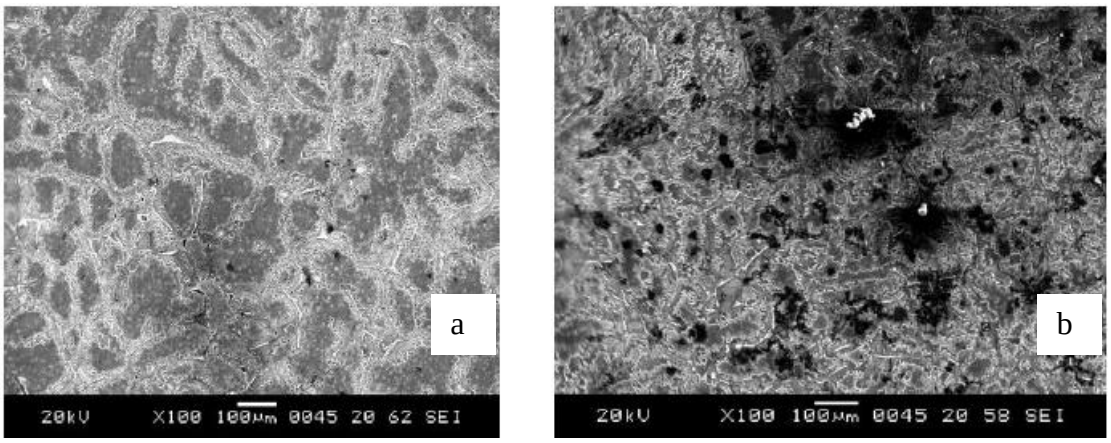

Plate VIII: SEM images thermal treated of Al–Si–Mg/10%LBWA composite(50µm), oil quenched (a)0.02hr intermediate natural ageing time (b) 0.5hr intermediate natural ageing time)

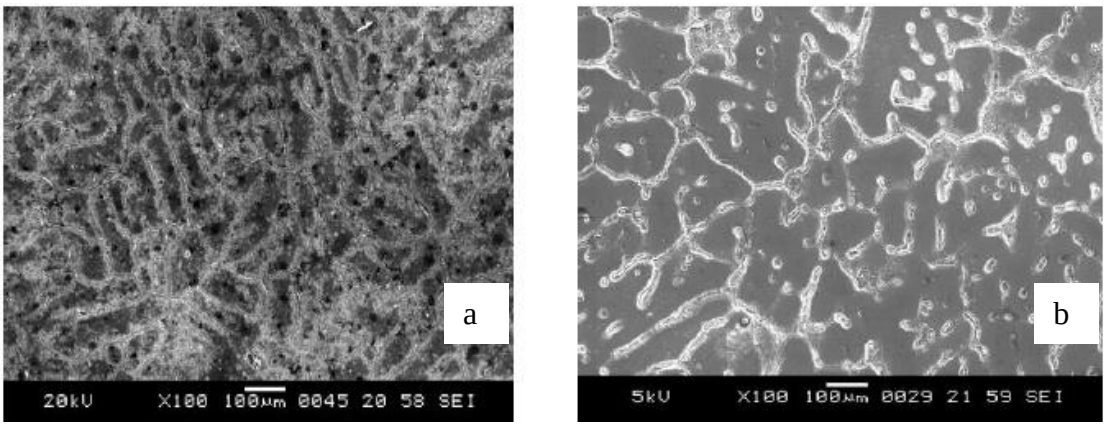

Plate IX: SEM images of thermal treated Al–Si–Mg/10%LBWA composite(50µm), oil quenched (a)1hr intermediate natural ageing time (b) 1½hrintermediate natural ageing time)

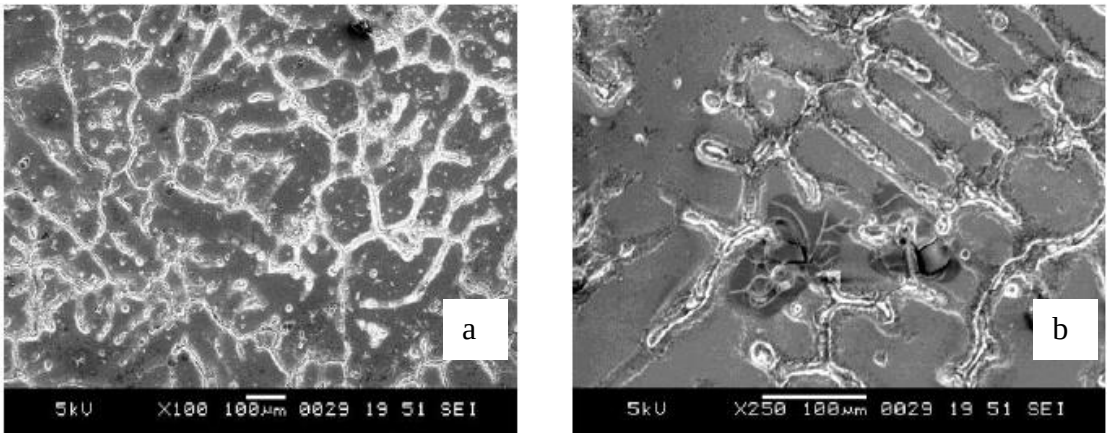

Plate X: SEM images of thermal treated Al–Si–Mg/10%LBWA composite(50µm),oil quenched (a)2hrsintermediate natural ageing time (b) 3hrs intermediate natural ageing time)

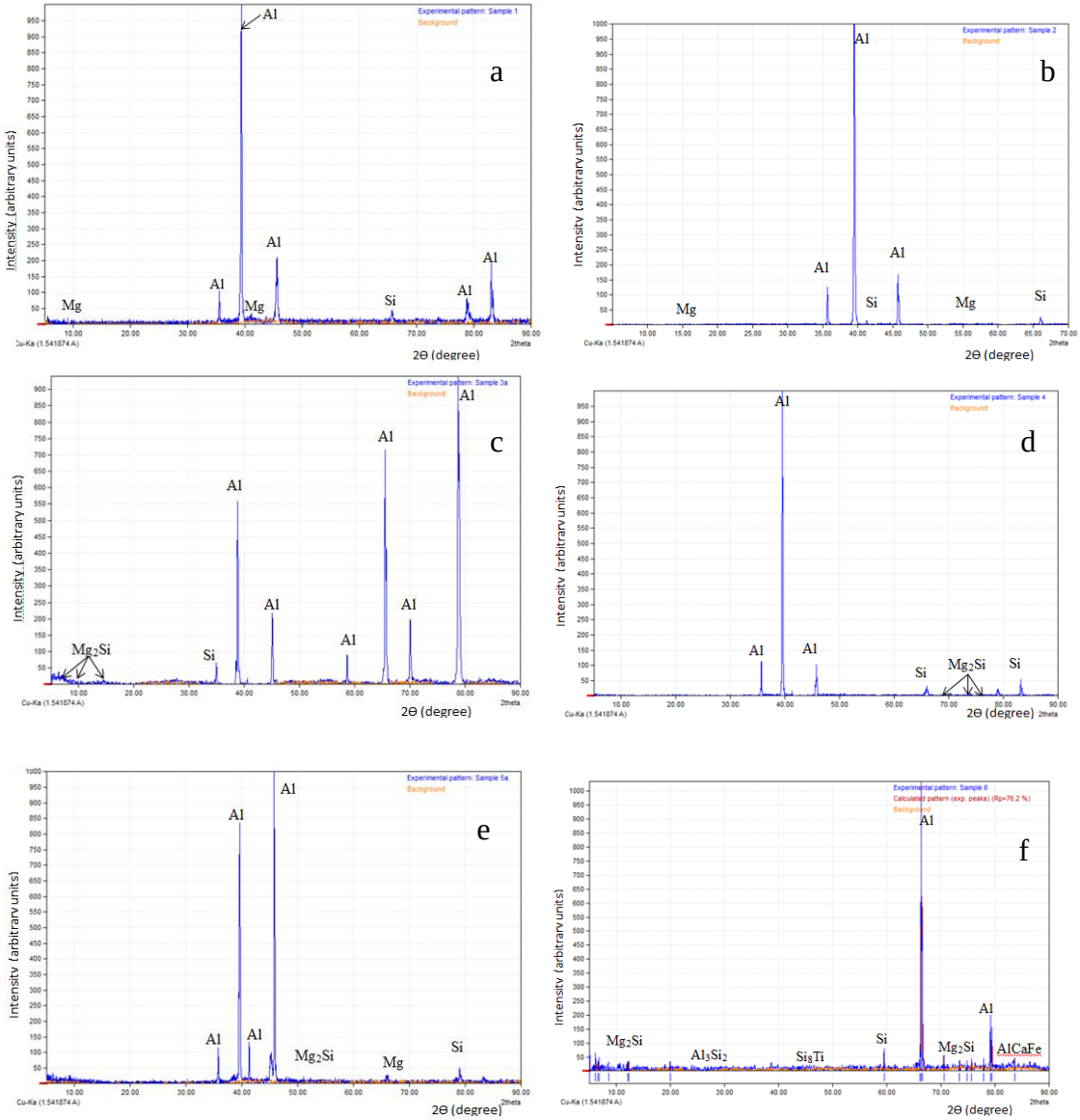

Figure 3: XRD pattern of Al-Si-Mg/10 % LBWA; a-f;1, 30, 60, 90, 120, 180 minutes, intermediate natural ageing time, oil quenched respectively.

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