ENHANCING THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF RARE EARTH LA MODIFIED HYPEREUTECTIC AL-SI ALLOY USING FRICTION STIR PROCESSING TECHNIQUE

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ABSTRACT

The properties of cast hypereutectic Al-Si alloys are dependent on fine silicon morphology and smaller size of intermetallic formed during solidification. Rare earth (La) elements addition is being used to alter the morphology of silicon particles to small sizes. However, La formed multi-compounds with elements in the base alloy, which appear as coarse needle-like or plate-like structures and can interrupt, and hinder the full modification of Si particles, in turns pose detrimental effect to strength of the alloy. The aim of this study is to enhance the microstructure and properties of Al-18Si-0.9Cu-1Ni-1%La alloy modified with La rare earth element by using FSP technique. FSP is a new processing technique facilitate by the frictional heat generation between tool and the work piece. FSP can triumph over the detrimental properties due to coarse needle-like or plate-like intermetallic structures and coarse large size non-uniformly distributed Si particle in the Al matrix, creates finer and smaller sizes of particles and uniform distribution, thus enhanced properties. The surfaces of cast Al-18Si-0.9Cu-1.5Ni-1%La were subjected to single pass stir FSP using two tool traverse speeds and fixed tool rotation speed. Optical and scanning electron microscopy analysis, tensile test, and hardness test were used to correlates between metallography examinations, and mechanical properties, to quantify the effects that the process FSP induced on the alloy. The microstructure, tensile strength, and micro hardness, of the alloy can be enhanced after FSP. Tensile strength, elongation, and hardness were increased to about 1.7 times, 5 times, and 5.37% respectively compared to the unprocessed base alloy. Presence of fine microstructure constituent, fragmenting of silicon, La intermetallic particles to fine and smaller sizes, and elimination of porosity was found to be important factors for the enhanced properties. Silicon, intermetallic particles, and porosity decreased to 51.14µm², 25.0µm², and 13.03 ECD were achieved with higher tool traverse speed 42mm/min. The challenge within this research is to extend the application of hypereutectic Al-Si alloys.

Keywords: Modification, Rare earth La, FSP, Microstructure, Tensile, Hardness

1 INTRODUCTION

In cast Al-Si alloy system, chemical modification treatment is used to change the shape of eutectic silicon particles from a flake like or acicular to a fine fibrous silicon structure [1], It enhances the mechanical properties of aluminium silicon alloys, such as the fatigue crack growth resistance because coarse silicon structure raises the internal stress in the microstructure and yields easier path for fracture and thus limits the maximal strength and ductility[2]. Modifiers are added to the melt either in element form, in master alloys or through fluxes [3]. Each modifier element has advantages and disadvantages over the other. Na and Sr are the most common and extensively used in the foundry industry to change the form of the silicon from flake to fibrous shape. The selection of a modifying agent also depends on some features like ease of dissolution, and stability in the melt [3]. For Na both its addition and fading in the melt are difficult to control. Sb was introduced as an alternative element for modifying the silicon eutectic [4]. However, it has been reported that Sb refines rather than modifies the eutectic flake structure, produces lamellar structure [5]. Rare earth (RE) elements addition was found to alter the morphology of the silicon structure. Small addition of misch metal (0.03-0.09 wt.% La RE content) can be enough to modify the eutectic Si, provided a critical cooling rate is reached. The RE-treated alloy has a low fading effect compared to Na-modified alloys, as it maintains the modified structure much longer [6, 7]. The RE modifying element should display some tendency for compound formation with aluminium matrix also have low solubility. La and Ce are reported to meet most of those conditions. Properties of Al-Si-Cu-Ni system are dependent on silicon morphology and intermetallic formed during solidification. Therefore, any process that can change the size, distribution, and shape of these constituents will result in changes in the mechanical characteristics. Hongkun *et al*., [2] had studied the La phase in Al-17Si alloys . They reported that the La phase grows into a variety of morphologies, such as needle-like, broken rod like in pores, spherical, flat platelets in the microstructure, some of La phases were observed to enfold some small polygonal silicon particles and the sizes of these silicon primary particles are comparatively small. The size of the La-rich phase is generally bigger than that of the silicon phase and they also reported the long straight intermetallic compound (gray contrast in the optical micrograph) represented as $Al_7Si_7Cu_2La_{3.5}$ in A390 Al-Si-Cu [8]. Tsai *et al.,* [9] investigations observed that the eutectic silicon of A356 alloy does not properly refined until the La concentration approaches 1.0 wt.%, in which the silicon displays fibrous morphology in the microstructure. Hongkun *et al.*, [10] claimed that they had found the optimal amount of La needed to obtain full modifying effect which is rather large i.e., 3.3 wt.%, whereby the morphologies of the eutectic and primary silicon are totally changed to finer silicon particles having smaller aspect ratios in Al-Si17 and Al-Si25 metals [10]. On the other hand, in the case of A390 alloy the addition of 3.0 wt.% of La into the metal displayed but little modifying effect on silicon phases in the A390 alloy as expected with the increase of defects [11]. Misch metal addition of above 1.0 wt. % was observed to decrease the tensile properties, but increased the hardness. RE can form compounds with alloying elements in base metal, which appear as needle-like or plate-like structures and may interrupt the modification morphology of Si [12]. Friction stir processing can be employed to modify microstructure and remove casting defects at preferred locations, where property enhancements could improve part service [13]. Ma *et al.*, [14] applied FSP to as cast A356 Al-Si alloy in order to examine modification influence of FSP on the alloy microstructure. They reported that FSP homogenizes and refines the as cast microstructure, totally removes pores, and produces a microstructure with finer silicon particles distributed in a fine aluminium grain. Additionally, FSP parameters considerably effects microstructure development within the processed region of cast Al Al-7Si. They observed that, higher tool rotation speed (700-900rpm) produces more uniform microstructure, and at lower tool rotation speeds (300-500rpm), a visibly macroscopic banded structure was noticed within the stir region, depicted by a low density of coarser particles. Karthikeyan *et al.*, [15] subjected the surfaces of A319 cast alloy plates to single pass FSP as means decrease the porosity level and grain size and porosity level and increases the material properties under various combination of tool travel speeds and tool rotation speeds. They found that at some combinations of the parameters, the processed plate shows an improvement of about 20 percent in micro hardness and 50 percent in tensile strength compared with those of the unprocessed plate. Also the ductility of the processed plate had improved, vary from 1.5 to 5. Mahmoud

[16] In FSP of A390 cast aluminium silicon alloy, observed that the aspect ratio and size of silicon particles of the alloy reduced from 59μm to about 9μm and from 3.5μm to about 1.4μm, respectively. They also observed more uniform micro hardness within the stir zone of FSP processed alloy than that of the unprocessed one. It is evident from the past studies that friction stir processing (FSP) has the potential of locally modifying the microstructure of cast aluminium silicon alloy and in turns improve properties. Number of investigations have looked into the effect of friction stir processing specifically on the mechanical properties and microstructure of cast aluminium silicon alloys, and these experiments have mainly been on low Ni content alloys [17]. In this study the Influence of FSP on microstructures transformation, strength, and hardness of as-cast hypereutectic aluminium silicon alloy with high Ni content and modified with rare earth La is been reported.

2 METHODOLOGY

The Al-Si hypereutectic ingot was first cut into smaller pieces, cleaned and dried, weighted and melted in a silicon carbide crucible employing induction furnace. The melt temperature was maintained at 577°C [18] and increases the temperature to 750-760°C for an adequate time to allow for melt homogenization [19]. The molten alloy was poured at temperature of 730 ± 5°C into pre-heated steel mould. The La addition level was 1.0 wt. % [20]. The material addition was allowed to 10 min for homogenization before pouring [19]. The as cast Al-18Si-0.9Cu-1.5Ni-1%La material was prepared into plates of size 150mm x 30mm 7mm, and subjected to for friction stir processing on vertical milling machine as shown in Figure 1, using 800rev/min rotational speed, 22mm/min and 42mm/min traverse speeds. The FSP processed samples were cut in the transverse direction, ground using grades of sand papers, polished with 0.5μm alumina paste, and etched, using 0.5 M hydrofluoric acid to reveal the microstructures. The as cast base material, and as FSP processed samples microstructure examinations was conducted using optical, and scanning electron microscope (SEM). Image analysis technique was used for determining the sizes of the silicon particles, intermetallic particles and porosity. To measure the tensile properties, tensile specimens were prepared as per ASTM: E8/E8M-11, with the gauge length and gauge width of 25mm and 4mm respectively, using 1mm/min cross head speed. Tensile test was done on both the as cast alloy and the FS-processed samples. At every condition, two specimens were tested recorded their average values. Fracture surfaces of the tensile specimens were examined using SEM. Samples for micro hardness test were grinded and polished. Vickers micro hardness was measured across the samples processed zone, using a load of 500g and dwell time of 10s.

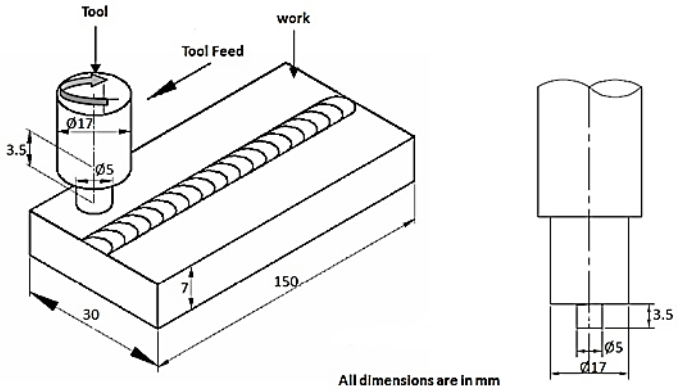

Figure 1. Schematic representation of the FSP process, dimension of work piece, and tool

3 RESULTS AND DISCUSSION

3.1 Microstructure of Al-18Si-0.9Cu-1Ni- 1%La base alloy

Plate 1, present the optical micrograph of the 1.0%La modified base alloy. The microstructure consists of flake like, irregular primary silicon particles, and needle/platelet-like intermetallic structures,

and primary silicon morphology in the microstructure. The Silicon particles size; obtained as mean area and aspect ratio, measured by image analysis technique were 136 μm^2 and 2.67. Porosity of up to ~182 ECD μm was detected in the alloy.

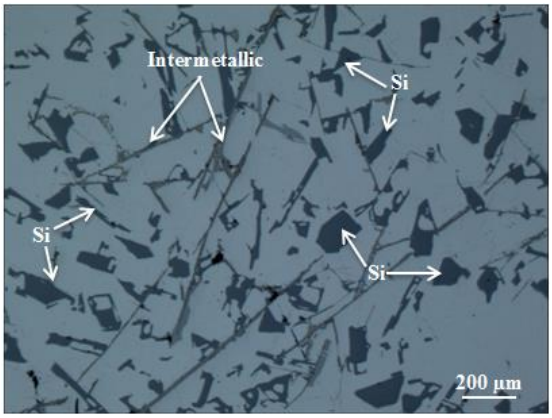

Plate 1. Optical micrograph of Al-18Si-Cu-Ni-1.0 wt. %La alloy

SEM micrograph was shown in Plate 2, it can be seen that the microstructure consists of (bright) long needle/platelet-like intermetallic phases, having mean area of 519 μm^2 and aspect ratio of 18; measured by image analysis. EDS point analysis indicates that the bright needle and platelet shaped compounds were formed by La with Al, Si, Cu, and Ni. It can be observed that the needle-like or platelet second phases are distributed across eutectic Si, and some phases interfaces at edge of the primary Si crystals. Similar result was reported by Hongkun *et al.*, [8], when studying the compounds and microstructures of La and Cu containing aluminium silicon alloy (A390). Intermetallic compound is vital parameter for the modification of silicon. From the SEM micrograph Plate 2, the AlLaSiNiCu observed near Si, which could have disturbed the silicon modification. Hongkun et al, [2] agreed with the result, where Al-La-Si-Ni-Cu makes La harmful to modify the Si structure. The tendency of La element to form these phases is attributed to the difference in atomic radius [21]. However the harmful effect of this intermetallic is that they interrupt the modification of Si [2], and may result to poor tensile properties [12].

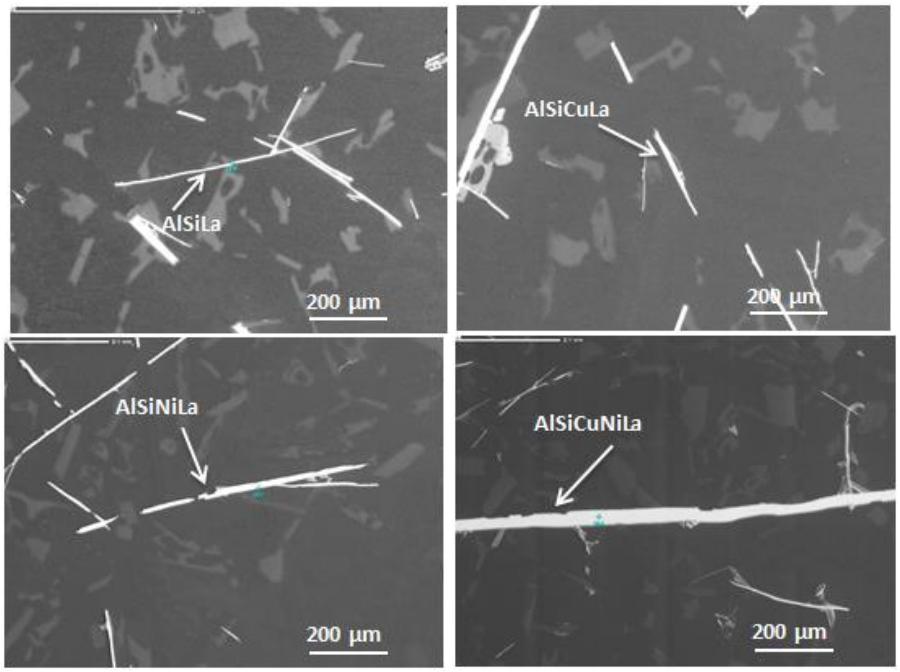

Plate 2. SEM-EDS micrograph of base alloy showing; AlSiLa, AlSiCuLa, AlSiNiLa, and AlSiCuNiLa

3.2 Microstructure of Al-18Si-0.9Cu-1.15Ni-1La after FSP

Plate 3, presents optical micrographs of the Al-Si-Cu-Ni-1.0wt%La alloy samples after FSP. The graphs display the significant influence of FSP on the morphology (shape and size) of silicon particles. High density of finer silicon particles are distributed within the processed zones [22].

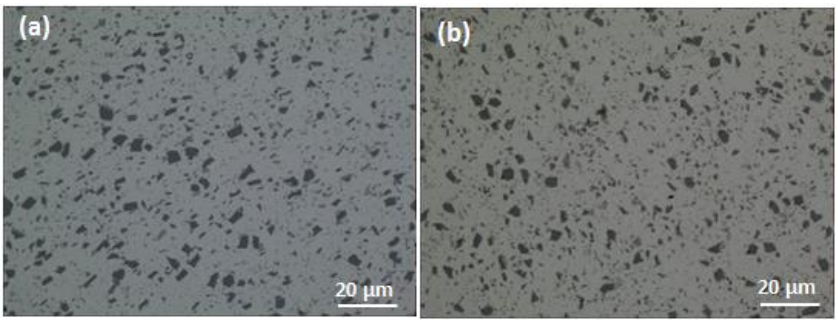

Plate 3. Optical micrograph of Al-18Si-0.9Cu-1Ni-1%La alloy after single pass FSP using 800RPM: (a) 22mm/min (b) 42mm/min tool speeds

Stirring effect of the FSP tool results in breakdown of the coarse needle type eutectic silicon and the coarse primary silicon particles to finer particles [14, 23] also produced a enhanced distribution of fine and near circular silicon particles in the aluminium matrix. Quantitative measurement of the silicon particles sizes, and aspect ratio, relative to a fixed tool rotational speed (800RPM), and traverse speeds, were measured to be: (52.11µm2 /1.90), for 22mm/min travel speed, and (51.14µm2 /1.85), 42mm/min travel speed respectively. Similarly, porosity has significantly decreased from 182µm ECD, in the as cast condition to as low as (15.89 ECD, for 22mm/min travel speed, and (13.03 ECD, for 42mm/min travel speed, after FSP. This is consistence with the finding in [24], influence of FSP on aluminium silicon Al-7Si-Mg cast alloy. It can be seen that all the combination of rotational and traverse speeds used in this study, induced considerable effect on the Si, aspect ratio, and porosity of the base alloy [25]. Plate 4 shows the SEM micro graph for the FS-processed Al-18Si-0.9Cu-1Ni-1.0wt%La. It can be seen that all the white long platelet-like La-intermetallic phases (AlSiLa, AlSiCuLa, AlSiNiLa, and AlSiCuNiLa), observed in the as cast alloy Plate 2, have been fragmented into smaller sizes and redistributed in the FS-processed samples, showing the influence of tool speeds on morphology, size and distribution of the intermetallic particles [26]. The decreasing of the RE intermetallic particle size within stir zones is as a result of increased particle fragmentating througout FSP [27]. That results to a rise in the density of smaller La-rich particles, and volume of larger La-rich particles decreasing. Thus, the accompanied deformation conditions were enough to promote more fragmentation of the La-rich intermetallic [14]. The particle measured mean areas and aspect ratios were measured to be: (28.4µm^2 /12.62); at 22mm/min tool travel speed, and (25µm^2 /2.43); at 42mm/min tool travel speed respectively. As conceptualized by Mishra et al. [28], APR values lead to higher residence time for the particles in the shear zone and higher process strain, leading to more fragmentation and higher dispersion of the RE intermetallic particles [27, 28].

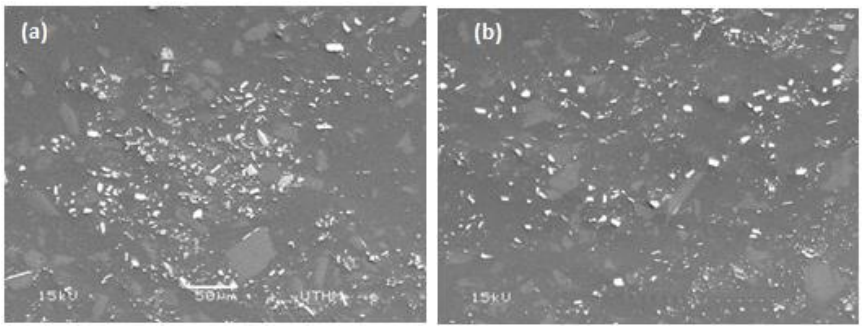

Plate 4. SEM micrograph from the stirred zone SZ of Al-18Si-Cu-Ni-1.0wt%La alloy after single pass FSP using 800RPM: (a) 22mm//min, (b) 42mm/min

3.3 Tensile test

Figure 2 present variation of tensile properties and quality index of the base metal before and after FSP processing at a varied transverse speeds and fixed tool rotational speed. The base metal shows a typical brittle material with lower elongation. As reported by [29], RE can form compounds with alloy elements in base alloys, which appear as needle-like or plate-like structures and can interrupt the modification morphology of Si [12]. These intermetallics also affect the strength and quality of Al–Si–Cu–Ni casting alloy. But after FSP it was transformed to a ductile material with wide strain-hardening region. Evidently, FSP improved the ductility and strength of the alloy simultaneously [30]. It is visible that before FSP the mean tensile strength (UTS), of the base metals (BM-1%La; 102 MPa, mean elongation to failure (%El): 1.28 %, and mean (Q): 118.1 MPa), were lower compared to all the FS-processed samples. These low tensile strength/elongation of the base material could be related with the formation of brittle intermetallic structure [15]. After FSP, samples showed significant increases in tensile properties. The UTS was improved up to 1.7 times, and % elongation about 5 times, as compared with that of the based metal, largely due to the refinement of the microstructure as shown in Plate 3 and 4. Other studies also have found that the tensile properties and fatigue of cast aluminium silicon alloys improved with refined microstructure, and reducing casting defect such as porosity size density [31].

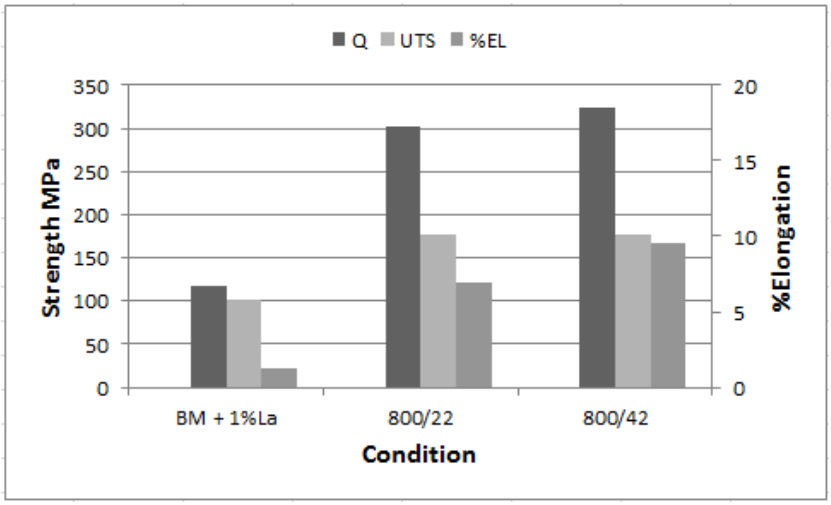

Figure 2. Variation of tensile properties

Referring to Plate 4, enhanced distribution of second phase particles is believed to have influenced both the tensile and ductility in the processed samples [24]. This tensile properties enhancement is related to the RE particles structural changes in the samples alloy [32]. It is evident that the smaller, finer La intermetallic particles sizes were effective in improving the tensile properties of the FS-processed samples. The presence of fine RE containing intermetallic phase with smaller area size in the stir zone microstructure, provides less stress concentration sites and crack initiators with less negative effect on tensile properties [32]. Other studies have shown that the rupture mechanism of aluminum alloy was changed by smaller amount of rare earth intermetallic [33]. FSP facilitates intense plasticization of the RE particles and fragmenting into smaller sizes. Quality index was increased about 2.5 times compare to that of base metal. it can mentioned that thermal treatment by FSP plays a crucial role in the refining of La intermetallic particles [34]. Plate 5, presented the SEM fracture surfaces of the base alloy, and FS-processed tensile samples. It can be seen that Plate 5(a) the surface was dominated by cleavage planes which indicate a brittle failure pattern; results in lower ductility. The cleavage pattern in fracture surface gives the indication of brittle material [24]. Plate 5 (b)-(c), presented the fracture surfaces of FS-processed tensile samples, shows fracture surface with dimple rupture pattern. Formation of dimple pattern generally gives the indication of ductile material [24, 35]. FSP resulted in the refinement and redistribution of eutectic Si particles and breaking of continuous network of brittle second phases particles. When these FSP samples were subjected

to tensile testing the micro voids nucleated after certain level of straining, but interlinking of these micro cracks became difficult and therefore allowed the ligament to deform considerably; resulted in high ductility. The nucleation of micro voids at the particle matrix interface represents dimple like pattern in the fracture surface [24], which is evident in Plate 5(b)-(c). The dimples become finer and smaller as the tool travel speed increases, implying increased material ductility [36].

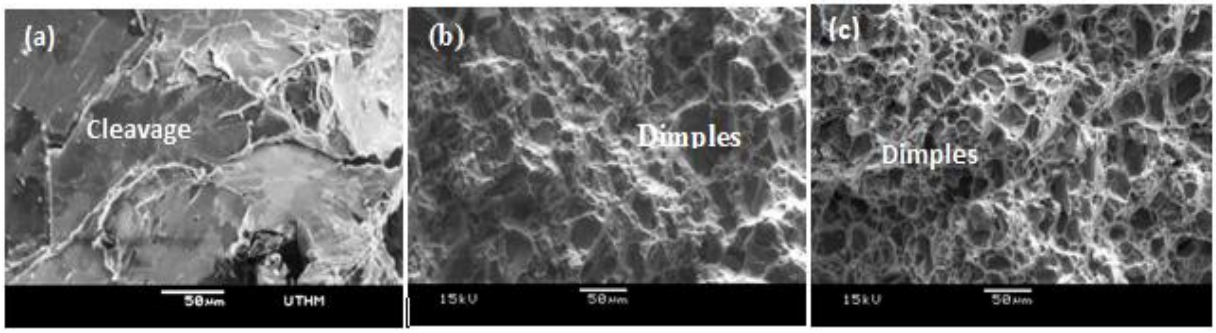

Plate 5. The fracture surfaces of (a) the as cast base metal + 1%La, and FS -processed tensile samples using 800RPM: (b) 22mm/min, (c) 42mm/min

3.4 Micro hardness

The base alloy BM-1%La exhibited measured average hardness values of 64.80 HV. While for the FSP processed samples, mean hardness measured were 67.22HV; under 22mm/min traverse speed, and 68.29HV; under 42mm/min speed. Distribution of Si particles and the intermetallic phases in the microstructure play a vital role in determining the hardness and strength values of the Al-Si casting alloys [37]. FSP enhanced the hardness of the processed samples. This is primarily due to the increase in dispersion of refined and more finer distributed eutectic and primary silicon particles in the SZ Plate 3, by the effect of FSP [38]. Also, the intermetallic phase was refined due to the dynamic recrystallization during FSP [13, 25], which provide additional improvement in hardness values. All the different combinations of processing parameters resulted in nearly similar hardness values. The mean hardness increases with increasing the tool traverse speed [39]. The processed samples show an increased in the hardness of about 3.7% to 5.3%. At the higher tool travel speed 42mm/min sample, the slighter higher values of hardness obtained may be ascribed to the fact that sample possess better combinations of the modified Si structure and a considerable dispersion of the La-rich precipitation-hardening phases as shown in Plate 4. This is due to the fact that the hardening effect caused by the RE phase is greater than the softening effect resulting from a lower coarsening rate of Si particles [40]. There was small difference in mean hardness between the 22mm/min and 42mm/min traverse speeds. Implying that, the 22mm/min and 42mm/min speeds induced nearly the same effect on hardness of the FS-processed samples.

3 CONLUSIONS

This research work was conducted to enhance the metallurgical properties of modified Al-Si Al-18Si-0.9Cu-1Ni-1%La alloy modified La rare earth element. It correlates between metallography examinations, and mechanical properties, to quantify the effects that the process FSP induced on the alloys. The microstructure, tensile strength, and micro hardness, of the alloy can be enhanced after FSP. Tensile strength, elongation, and hardness were increased to about 1.7 times, 5 times, and 5.37% respectively compare to the unprocessed base metal. Presence of fine recrystallized microstructure, breakup of silicon, La intermetallic particles to fine and smaller sizes, and elimination of porosity are found to be important factors for the enhanced properties. Silicon, intermetallic particles, and porosity decreased to 51.14μm², 25.0μm², and 13.03 ECD were achieved with higher tool traverse speed 42mm/min. The challenge within this research is to extend the application of hypereutectic Al-Si alloys.

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