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Experimental Analysis of Microstructure and Mechanical Behaviour in Fine Grained Al-Zn-Mg Alloy

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ABSTRACT

The high strength Al-Zn-Mg alloy is widely used for structure components in critical aerospace applications. Small additions of scandium (Sc) often refine cast microstructure and mechanical properties by forming metastable Al_3Sc dispersoids. The main role of the Al_3Sc dispersoid is to prevent recrystallization during hot deformation and solution treatment. A new technique to apply for surface modification of cast aluminium alloy is usually called friction stir processing (FSP) but basic principle follows by friction stir welding (FSW). The main aim of the present work is to discuss the recent advances in the area of FSPed fine grained aluminium alloy and to highlight the modern trends in research. Highlighting will be focus on the main strategies to increase strength of the fine grained aluminium alloy without loss of ductility, microstructure-property relationship and enhanced mechanical properties, as well as innovative potential of the fine grained aluminium alloys. It is experimentally proven that the higher Zn content (7.35 wt.%) with high Sc content (0.83 wt.%) alloy after FSPed enhanced as much as 0.2%PF 165.5 MPa, UTS 325.2 MPa and ductility 8.5 % respectively. Moreover, several characterizations has been done such as OM, XRD, TEM and fractographs analysis through EDAX and SEM analysis also revealed Al_3Sc agglomeration after solution treatment and cavity formation due to Zn vaporization at high rotational speed and traverse speed of FSPed alloys. But mechanisms states that the combined effects of chemical properties, fine grained due to dynamic recrystallization, $MgZn_2$ hardening precipitates and Al_3Sc dispersoids of anti-recrystallization results. The several mechanical testing has been done such as hardness measurement along the stir zone, tensile properties and toughness. The aim has to enhance functional properties of aluminium alloy through new innovation technique of FSPed.

Keywords: Al_3Sc dispersoids, solution treatment, FSPed, FSW, fine grained, rotational speed and traverse speed, dynamic recrystallization, stir zone, toughness.

I. INTRODUCTION

The modern commercially available high strength Al-Zn-Mg alloy usually contains Sc which form extremely fine, thermally stable Al_3Sc phase. This combination of alloy components results in grain refinement, anti-recrystallization and enhanced dispersoid strengthening [1]. It is more important to mention that the GP zones and the metastable ή phases are mainly responsible for the peak hardening effect of this cast alloy. Moreover, a dimensional and morphological change of GP zones, ή and η precipitates were generated in order to important to examine and clarify the microstructural changes that occur in this alloy during artificial ageing. The Al_3Sc particles did not supress or retard the formation of ageing precipitates in Al-Zn-Mg alloy. While, a fine dispersion of coherent Al_3Sc precipitates enhanced strengthening of the alloy. Some researchers have reported that the dispersoid of Al_3Sc particles perfectly correspond to the $L1_2$ structure when they form during homogenization treatment [2]. Experimentally proven that during decomposition of richer solid solution a higher density of Al_3Sc precipitates is formed, generating higher age hardening response at 120 ^oC ageing temperature. In generally, the ageing treatment relies on the fine precipitation from the supersaturated solid solution (SSS) which is associated with nucleation and growth process. The main precipitates are the

$Al_{32}(MgZn)_{49}$ and $MgZn_2$ phases with Al_3Sc form in α-Al matrix. In literature mention that the τ- $Al_{32}(MgZn)_{49}$ phase is considered as a non-stochoimetrically intermetallic phase having a variable composition around the $Al_2Mg_3Zn_3$ formula. These precipitates strained the crystallographic planes of the aluminium matrix [3]. In addition, the Sc grain refinement effect is observed for content of 0.83 wt.% in aluminium alloy. Increasing the Sc content at 0.83 wt.% does not influence the mechanical properties due to formation of Al_3Sc chunky particles which have identified through optical microscopy, SEM and tensile fractographic studies on friction stir processed (FSPed) alloy. These examinations also revealed on Zn vaporization like black cavities in FSPed alloy. However, microstructure is developed during casting, heat treatment (i.e., solution treatment) and friction stir processing, respectively. Thus, Sc addition improved technological characteristics of this alloy and promoted strengthening after FSPed and heat treatment. This paper presents the effect of Sc on the microstructures and mechanical properties by FSPed fine grained Al-Zn-Mg alloy.

II. EXPERIMENTAL PROCEDURE

The aluminium alloy containing 0.83 wt.% Sc was cast using Al-2 wt.% Sc master alloy with pure Zn and pure Mg. The alloy was melted in a graphite crucible using an electrical resistance muffle furnace and poured into a mild steel mould to obtain 160 × 90 × 8 mm^3 size cast plate. For the alloy production the Al melt was heated up to 780±5 °C for 3 h and the Al-Sc addition was incorporated to carefully handle Sc fading effect during melting. The composition of the alloy was examined by ICP-AES and OES methods and the following chemical composition are Zn- 7.35, Mg-3.40, Sc-0.83, Si-0.04 and Fe-0.04 (all in wt.%). The as-cast alloy was subjected to solution treatment at 465±5 °C for 1h followed by water quenching and kept for one week in room temperature for natural ageing and then prefers artificial ageing at 80, 120, 140 and 180 °C for 16h in air atmosphere, respectively. In each time, Vicker's hardness (Model no.: FIE-VM50 PC) measurements were performed on T_4 alloy (solution treated + water quenching + room temperature ageing) at 10 kg. load with15 sec dwell time. The Vicker's hardness measurements on ageing kinetics have been shown in graphical representation in Fig.-1 for four ageing temperatures upto 16h ageing time. The Vicker's hardness values were selected of average six indentations of each time in hardness measurement. Consequently, the friction stir processing has been conducted on T_4 plates as dimension 150×90×8 mm^3 and then post heated up at 140±2 °C for 2h. During FSPed the following parameters have adjusted to 1025 rpm on clockwise direction and traverse speed to 70 mm/min setting in vertical milling machine. In contrast of processing it has to mention as advancing side (AS) is defined as the direction of tool rotation with the same direction as processing direction. Consequently, as retreating side (RS) is defined as the direction of tool rotation with the reverse direction of processing direction. With tool pin height 3.5 mm and shoulder diameter 20 mm which made of martensitic stainless steel on tempering as shown in Fig.-2. The optical microstructures revealed using LEICA DMI 5000M (Leica Microsystems, Buffalo Grove, IL) microscope after thoroughly metallographic polishing and etching by modified Keller's reagent (2.5 ml HNO_3(70%)+1.5 ml HCl(38%)+1 ml HF(40%)+ 175 ml water). The SEM with EDS (Model no.: ZEISS EVO MA and LA Series) analysis of T_4+FSP sample has been examined after fine metallographic polishing. Several phases are identified by XRD analysis. TEM analysis has been carried out to study the precipitation morphology, size, and orientation using at Techai G^2 20 S-TWIN at 200 kV. Similarly, the fracture surface of tensile specimens have studied by scanning electron microscopy (SEM) (Model no.: LEO 435 VF) analysis. The tensile test was conducted in Instron (universal testing machine) UTM (25 KN, H25 K-S, UK). The tensile samples (flat shape sample dimensions in mm): full length 58, gauge length 26, width 4, thickness 2) had collected from stir zone, and tested with cross head speed 1 mm/min at room temperature.

III. RESULTS AND DISCUSSION

In this work an Al-Zn-Mg cast alloy has investigated in order to identify the best solution treatment and ageing treatment and to better understand its precipitation hardening behaviour. Therefore, multi-step ageing is the most effective method to influence the structure and properties of multi-phase ageing alloy. As characteristic features of heat treatment of aluminium alloy on Sc inoculation is connected mainly with a presence of the general hardening phase $L1_2$ (Al_3Sc) in solid solution. For the first time the step ageing has been developed for aluminium alloy. A considerable influence of preliminary zone ageing on the following phase ageing was established for alloy. Nevertheless, the advantages of this alloy are high strength, wide solution range for solution treatment, age hardenability and weldable. The technical literature states that in this alloy, the strengthening effect is given by different types of precipitates: as binary precipitates, i.e., $MgZn_2$, MgZn, Zn_3Mg_7, Al_3Mg_2, Mg_2Zn_{11}, Al_3Sc, Al_2Sc, AlSc; ternary precipitates as τ ($Al_{32}(MgZn)_{49}$) [4-6]. It is also mention that these phases identified by XRD analysis. The Al-Zn-Mg-Sc quaternary alloy recently became commercially available and other different chemical compositions are still under development to mainly address to the aerospace and automotive industry. The leading mechanism to enhance hardening is probably a complex interaction between added elements with vacancies, which favours dispersion and refinement of the precipitates. Thus, small Sc addition (at 0.83 wt.%) has been improved aluminium alloy properties, including mechanical strength [7]. The metallurgical investigations have been performed on the samples (T_4 condition) to assess ageing effectiveness. Thus, the ageing kinetics is studied by hardness test (HV10) on small samples (10×10×10 mm^3) at temperatures of 80 °C, 120 °C, 140 °C, 180 °C, respectively. On the other hand,

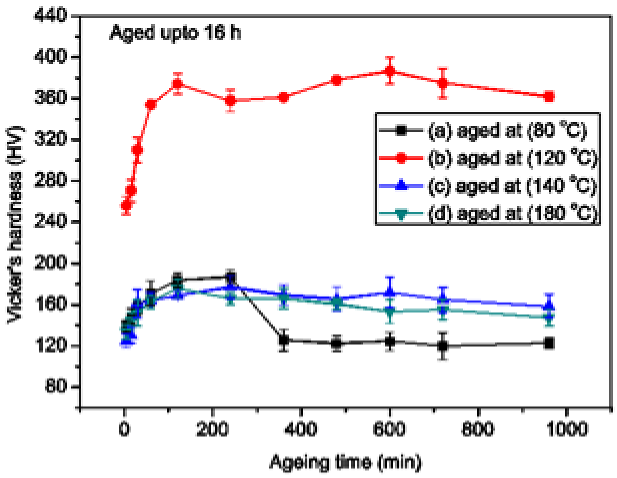


Figure 1 : Illustration of artificial ageing curves of studied alloy at different temperatures: (a) aged at 80 °C,
(b) aged at 120 °C,
(c) aged at 140 °C,
(d) aged at 180 °C.

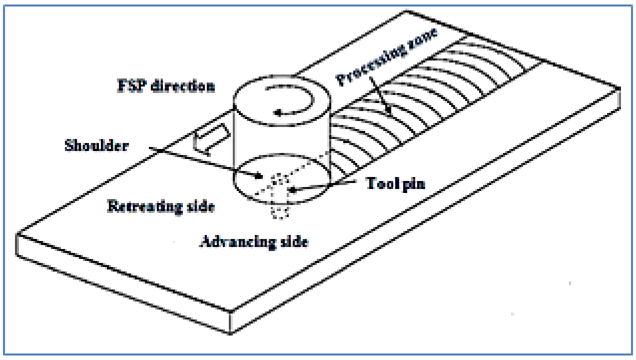


Figure 2 : The diagram of friction stir processing set-up.

when increasing the alloying elements in the aluminium alloy, the Vicker's hardness increased enormously due to large precipitation provokes a lattice compression, since the minimum point of the lattice parameter coincides with maximum point of the ageing . Therefore, the main precipitates are GP zones, ή, η, τ and Al_3Sc particles. It is also kinetically proven that ή and Al_3Sc particles are perfectly coherent in matrix then maximum hardening effects possible. Moreover, at the overageing stage the coalescence and growth of the precipitates cause their incoherence and the stresses tend to disappear [8]. In the present study, the Al alloy has successfully fabricated by double passes FSPed. The technique is energy efficient, environmentally friendly and versatile which basic principles originated from FSW. FSP has great advantages such as solid-state microstructural evaluation, adjusting mechanical properties by optimizing tool design and process parameters, the depth of processed zone and location. Several researchers (Colligan et al., 1999; Ma et al., 2002; Kwon et al., 2003; Nascimento et al., 2009; Wang et al., 2009) have proposed that the possible strengthening mechanisms are attributing to grain and subgrain structure and dislocation distribution of the modified surfaces [9]. It has successfully improved mechanical properties and also induced super plasticity (Elangovan and Balasubramanian 2007, Santella et al. 2005, Cavaliere and Squillaace 2005) [10]. On the other hand, finely dispersed Al_3Sc particles can limit the grain growth and resulted in an ultrafine grain size of FSPed Al-Zn-Mg-Sc alloy [11]. In this paper highlighting on two fundamentals aspect on aluminium alloy development with Sc to refine grains and ultra-fine grain achieved by FSPed. Therefore, it is also necessary to study the precipitation of Al-Zn-Mg-Sc alloy natural aged (i.e., room temperature ageing) plus artificially aged enhances hardening effects as shown in graphical presentations on continuous hardness plotting in Fig.-1. Ageing at 80 ^{o}C curve (in Fig.-1.a) show that the effect of hardening occurs (i.e., 187.2±6.8 HV at 4h ageing time) during the formation of coherent precipitates of GP zones but that it decreases gradually in occurrence of incoherent precipitates. At this low temperature ageing $ScAl_3$ particles remain in solid solution may not take part in ageing kinetics. Ageing at 120 ^{o}C curve (in Fig.-1.b) show that the effect of maximum hardening occurs at 386.7±8.8 HV at 10h ageing time but first ageing effects come out at 374±9.8 HV due to combine effects of coherent precipitates of GP zones plus ή-phase synchronising effects with Al_3Sc particles high density to results shown in continuous flow of hardening curve. Ageing at 140 ^{o}C curve (in Fig.-1.c) show that the effect of hardening occurs at 177.3±7.8 HV in 4h ageing time) due to high Sc content (0.83 wt.%) incoherence come off at faster rate as a results of Al_3Sc particles agglomeration main cause of lower hardening effects. The agglomeration of Al_3Sc chunky particles existence proven by EM with EDX analysis even after T_4+FSPed condition as well as tensile test fractography surfaces. Ageing at 180 ^{o}C curve (in Fig.-1.d) show that the effect of hardening occurs (i.e., 175.8±9.8 HV at 2h ageing time) during the formation of coherent precipitates of η-phases with Al_3Sc agglomeration comes faster rate of incoherent of precipitates to shown as lower hardening effects. In Fig.-3 shows the TEM micrograph with charged coupled device (CCD) analysis on T_6 alloy (i.e., solution treated then aged at 100 ^{o}C/6h). At this ageing temperature and time is kinetically favourable to formation of fine dense precipitates. The TEM micrograph exhibited on aged properties where fine distribution of uniform Al_3Sc precipitates in white spots also dense populated of GP zones with ή-phases formation in matrix. On the other hand, CCD pattern has shown uniform distribution of precipitates as arranging encircled with some bright spots nothing but some Al_3Sc chunky particles indication. In Fig.-4 shows the SEM micrograph with EDS analysis of T4+FSPed alloy where Al_3Sc chunky particles formed (shown in red arrows) as well-defined geometry in matrix. The EDS analysis is shown to Sc-34.94 wt.% optimal concentration. On the other hand, blue arrows have indicated to black spots owing to Zn vaporization as a result of enormous heat generated in stir zone (or nugget zone) as much as 400 – 480 ^{o}C in aluminium alloy [12]. In Fig.-5 shows the optical micrographs of aluminium alloy at different conditions as Fig.-5.a exhibited optical micrograph at T_4 condition (or no FSP). The solution treatment exhibited fine grains as Sc inoculated, also homogenization of grain boundary segregation with Al_3Sc chunky particles (as shown in red arrows) formation.

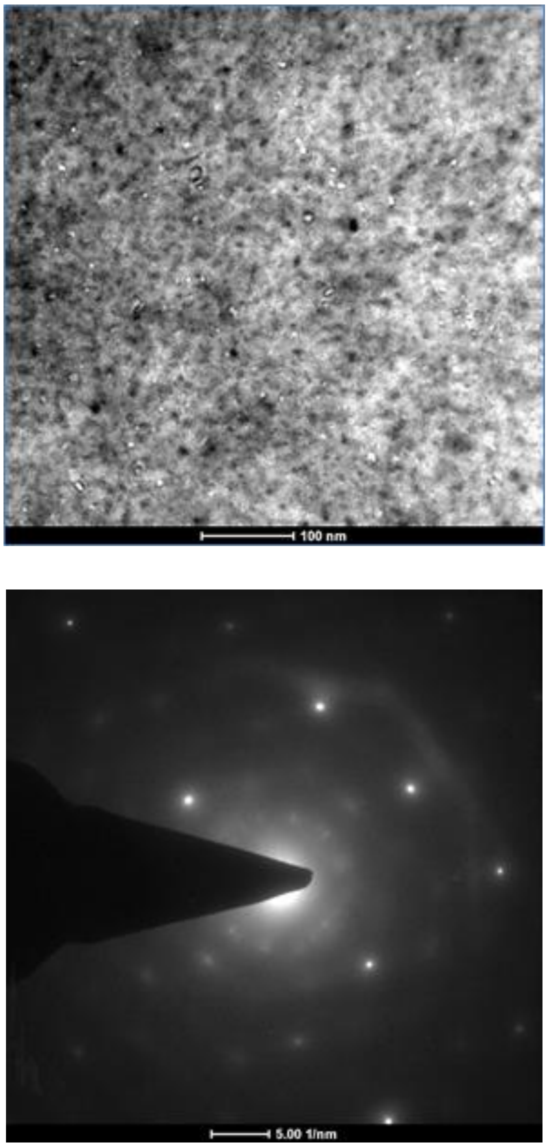
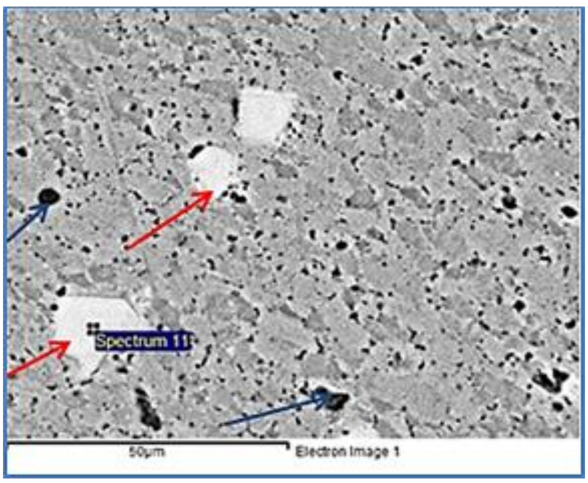


Figure 3 : The TEM micrograph with CCD pattern at T6 condition (aged at 100 oC/6h).



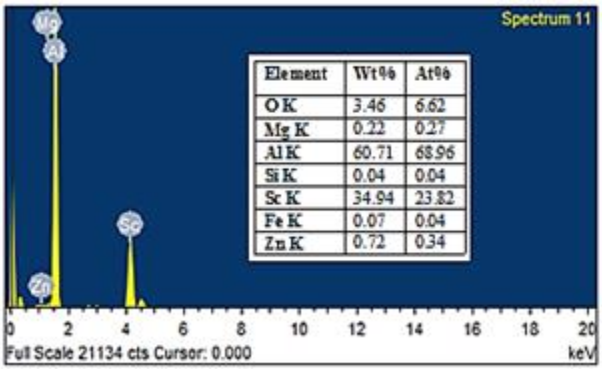


Figure 4 : The SEM with EDX analysis of T4 + FSPed alloy.

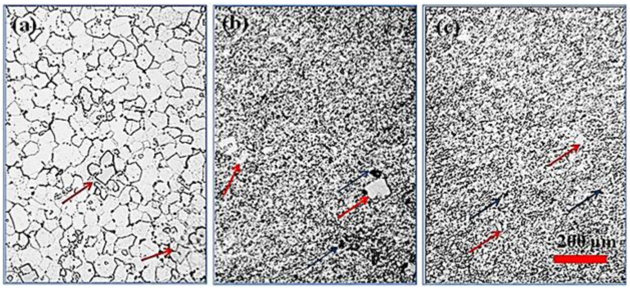


Figure 5 : Illustration of optical micrographs of studied alloy at different conditions: (a) T4 (no FSP), (b) T4+FSPed, (c) T4 + FSPed +Aged at 140 oC/2h.

It is obvious to confirm that the as high Sc (0.83 wt.%) in hypereutectic content in Al-Sc phase diagram [13] ability to refine grains as well as some Al_3Sc chunky particles formed. It is deleterious effect of high Sc content after ≥0.55 wt.% Sc (eutectic point) in aluminium alloy. In Fig.-5.b shows optical micrograph in stir zone (SZ) after T_4+FSPed condition. Categorically, during FSP generated three distinct microstructural zones such as stir zone, thermo mechanically affected zone (TMAZ) and heat affected zone (HAZ) [14]. The stir zone is in center of processing zone and has undergone the most severe plastic deformation in it. The stir zone exhibited very fine grains (>1µm) due to dynamic recrystallization with fine second phase dispersions with well-defined Al_3Sc chunky particles (as shown in red arrows) and several black cavities (shown in blue arrows) in matrix. Notably, it can be explained that due to high Sc content as results to Al_3Sc chunky particles formation and for high Zn content (7.35 wt.%) these defects like Zn vaporization occurred. Moreover, the defects like Zn vaporization occurred during processing because the material undergoes intense plastic deformation with high heat generation resulting in significant grain refinement with such defects [15]. In Fig.-5.c shows optical micrograph in stir zone (SZ) after T_4+FSPed+Aged condition exhibited distinct equiaxed fine grains with uniform fine precipitates with some Al_3Sc chunky particles (as shown in red arrows) and some small black cavities of Zn vaporization (as shown in blue arrows) in matrix. Also, some very fine flaw lines are exhibited due to distortion in plasticised zone with fine Al_3Sc particles anti-recrystallization effect [16]. Therefore, after the post ageing treatment the grains refined around 8-15 µm within the stir zone. In Fig.-6 shows the SEM tensile fractographs morphology presented of larger Al_3Sc chunky particles as shown in red arrows in matrix. The tensile fractographs are mostly ductile mode with crack lines separated like island. In Fig.-6.a shows the microcracks generated as a source of Al_3Sc particles or cracks initiated in vicinity of Al_3Sc chunky particles edges which may debonding from the matrix. In Fig.-6.b shows the microcracks generated clearly visible at the edges of Al_3Sc particles after post ageing treatment. So, both the cases at high Sc content are major deleterious effects of source of cracks generation to diminish the toughness and tensile properties of aluminium alloy. In Fig.-7 shows the bar diagram of mechanical properties of aluminium alloy after double passes FSPed. It has exhibited gradual increment of mechanical properties under FSPed condition. At T_4 (or no FSP) condition the mechanical properties attributed due to the grain refinement of solution treated matrix with high Sc content. At T_4+FSPed condition exhibited higher 0.2% proof stress, tensile stress and ductility but hardness is remaining same in all three cases. The tensile properties enhanced due to homogeneity of chemical composition with dynamic recrystallized fine grained with crushed of Al_3Sc chunky particles fragmentation at this condition. In contrary, hardness falls due to Zn vaporization under FSPed condition. At T4+FSPed+Aged at 140 ^{o}C/2h condition exhibited similar tensile properties like T4+FSPed condition but hardness slightly fall due to may Zn vaporization and high angle grain boundaries cause of faster rate of agglomeration of Al_3Sc particles rather fine precipitates formation during post ageing treatment as examined in Fig.-4 (as SEM analysis) and Fig.-5(optical microscopy at FSPed condition).

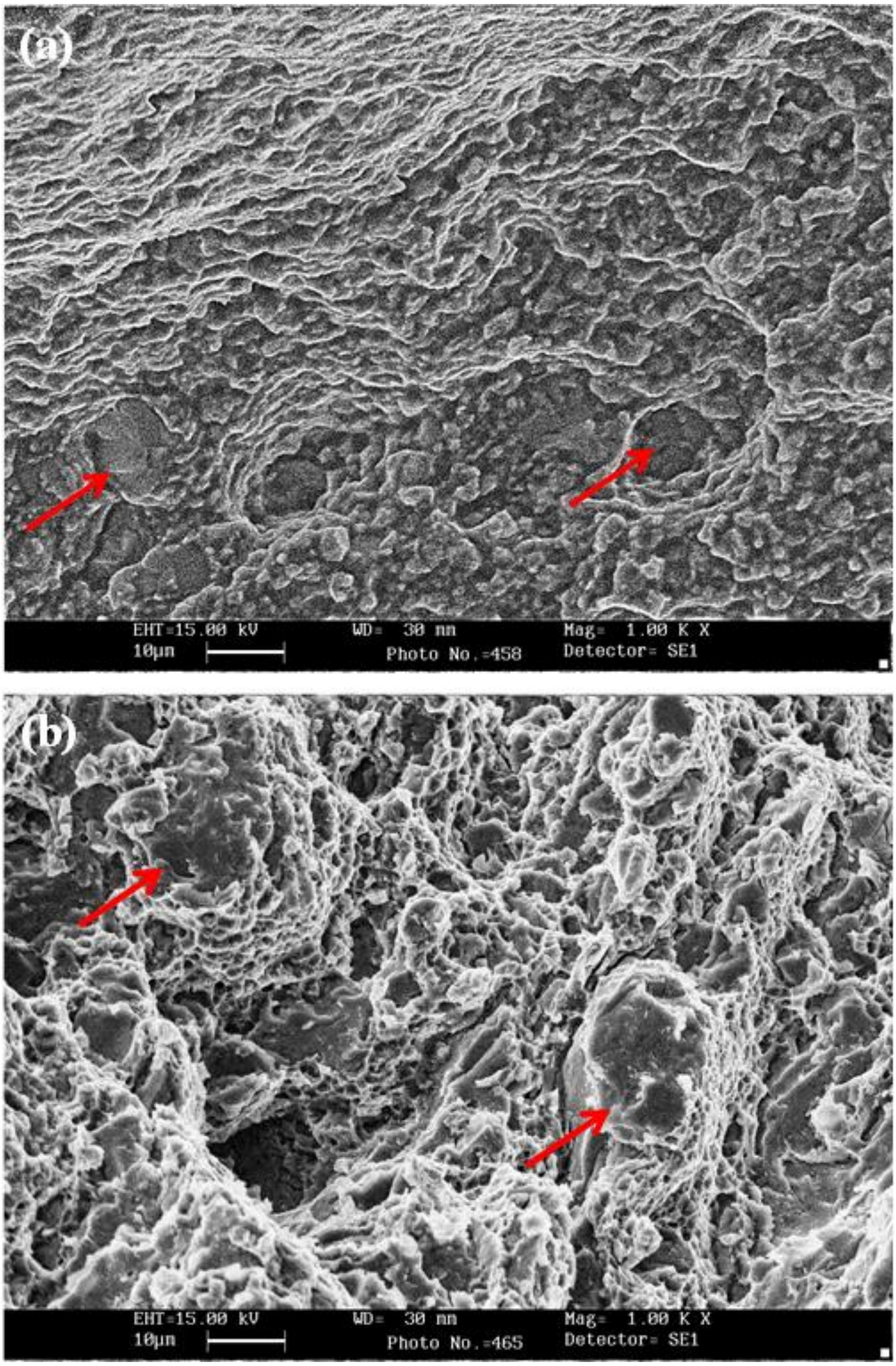


Figure 6 : The illustration of SEM tensile fracture surfaces after (a) T4+FSPed, (b) T4+FSPed+Aged at 140 oC/2h condition

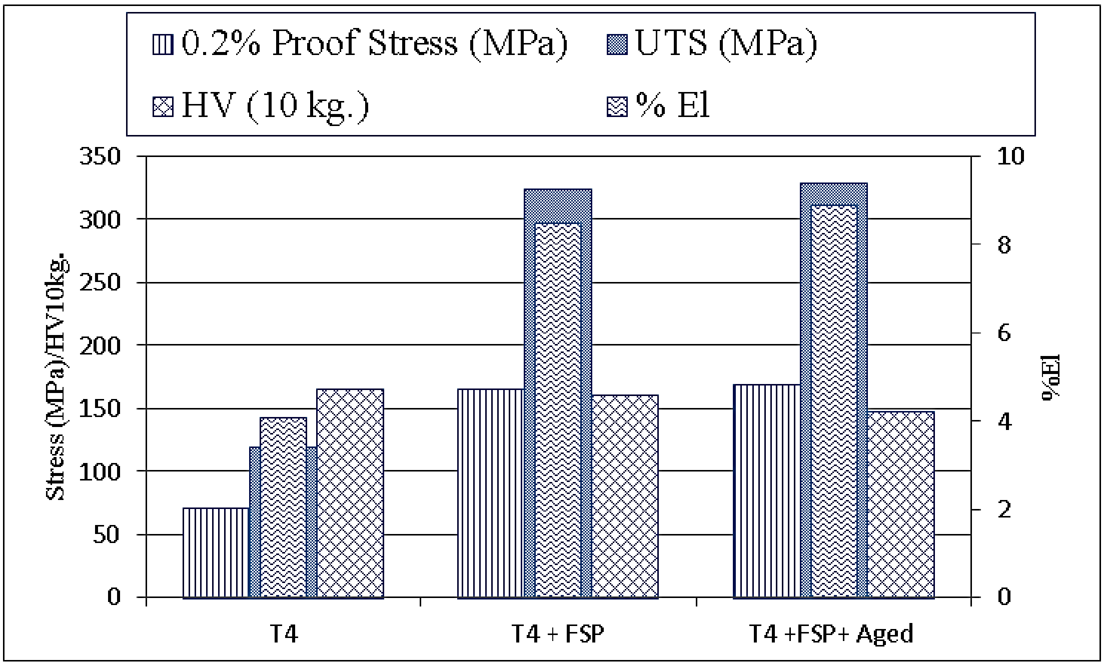


Figure 7 : The bar diagrams represented the mechanical property of studied alloy after double passes FSPed at different stages.

IV. CONCLUSION

1. The high number density (approximately 1022 – 1024 per m3) of coherent precipitates (L12 based ScAl3) achievable in this alloy also inhibits migration of grain boundaries due to Zener-drag effect which resulting in a high recrystallization resistance.
2. The double passes FSPed can be locally eliminated casting defects, homogenized precipitates, refine grains and thereby improving tensile properties and hardness.
3. In general, the stir zone has been characterized by a recrystallized fine grained structure and uniformly distributed second phase particles and Al3Sc dispersoids.
4. So, at T4 condition with Sc inoculation grain size achieved 35-50 µm, after T4+FSPed condition grain size refined 2-10 µm and after T4+FSPed+Aged at 140 oC/2h condition grain size refined 8-15 µm, respectively.
5. It can be concluded that the heterogeneous nucleation and growth of Al3Sc precipitates in the FSPed zones. FSPed involves a heat input (i.e., 400-500 oC within a few seconds) from frictional forces and dynamics recrystallization processes occurring within the friction stir zone, which provide Sc with sufficient mobility for precipitation and growth to results Al3Sc chunky particles (in high concentration of 0.83 wt.% Sc content) and Zn (7.35wt.%) vaporization revealed by optical microscopy and SEM examinations after T4+FSPed and T4+FSPed+Aged at 140 oC/2h in both the cases.
6. After T4+FSPed+Aged at 140 oC/2h condition, nucleation and growth of Al3Sc precipitates are heterogeneous during friction stir processing plus post ageing treatment owing to existence of dislocations and the large angle grain boundaries are facilitated to coarsening of Al3Sc precipitates within the friction stirred zone as a results no improvement of mechanical properties as shown in bar diagrams (in Fig.-7).

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