

Investigation of Microstructure And Mechanical Properties of Al-Zn-Mg and Al-Zn-Mg-Sc Alloys After Double Passes Friction Stir Processing

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Abstract: In this study, the two types of aluminium alloys namely Al-Zn-Mg and Al-Zn-Mg-Sc were developed by casting route. The cast plates were subjected to double passes friction stir processing (FSP) by in-situ vertically fitted milling machine. The tool design and process parameters were found to affect the microstructural and mechanical properties of these aluminium alloys. Therefore, the experimental samples were collected from the stirring zone and selected for mechanical testing. The FSP caused intense plastic deformation, material mixing, and thermal exposure, resulting in significant microstructural refinement, densification, and homogeneity of processed zone. It has been observed that the mechanical properties improved as a result of the complex interactions due to FSP, thermo-mechanical effects, dissolution, coarsening and re-precipitation of the strengthening precipitates in these two experimental aluminium alloys. It can be concluded that the strengthening effect of the Al-Zn-Mg and Al-Zn-Mg-Sc alloys after FSP are associated with the fine grain strengthening, subgrain strengthening and precipitation strengthening of Al_3Sc and $MgZn$ precipitates. Finally, the resultant properties have been evaluated by FSP parameters such as rotational speed of 1000 rpm and traverse speed of 70 mm/min and specified tool design. In addition, the detrimental effect of high Zn content (~8.20 wt.%) and high Sc content (~0.87 wt.%) aluminium alloy may be attributed to the vaporization of Zn at the time of the FSP. The experimental samples were collected from double passes FSPed zone and critically examined for optical microscopy (OM), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), transmission electron

Keywords: Double passes FSP, Sc particles, Zn vaporization, fractographic analysis.

1. Introduction

The Al-Zn-Mg and Al-Zn-Mg-Sc alloys are exhibited age-hardening effects and good mechanical properties. Scandium (Sc) used for grain refinement agent in cast aluminium alloys [1]-[3]. The main functions of these alloys are to increase strength without affecting other properties. The Zn/Mg ratio is the major criterion to hardening alloys. The peak hardness is increased with increasing Zn/Mg ratio. The particles of Al_3Sc are the main stages involved in 7xxx series aluminium alloys [4]-[6]. Impurity elements like Fe and Si are formed intermetallic compounds and degrade the fracture toughness properties. Moreover, solution treatment at 465 for 1h then immediately water quenching (T_4) reduces the cast inhomogeneity, grain boundary segregation and

$\text{Al}_2\text{Zn}_3\text{Mg}_3$ phases in the temperature range 435-445°C in the matrix [7]. Loss of thermal conductivity is significantly degraded by multiply alloying precipitation hardening elements such as Mg, Fe, and Si, [8]. FSP is the advanced solid state technique to modify cast surface layers and to enhance specific properties to some considerable depth. Although, the basic principle is based on FSW (friction stir welding). FSW was first developed at The Welding Institute (TWI) in Cambridge, UK in 1991 [10], [11]. This process is particularly suitable for welding/processing of non-ferrous metals with low melting points such as magnesium, aluminium, brass, titanium, zinc and lead. Because of melting does not occur and processed area takes place below the melting point ($\sim 0.6-0.8 T_m$ of T of the base material), a good quality of processed or weld zone is created. The heat input during FSP creates from friction and plastic deformation which indicates FSP is a green and energy-efficient technique without harmful gas, radiation and noise. The component shape and size is remaining same after FSP. A specially designed cylindrical tool, consisting of a shoulder and a profiled pin is inserted into the work piece by a rotational movement with the axial force. A temperature rise of 400°C to 500°C has been reported within the SZ (stir zone) of Al-Zn-Mg alloys [12], [13]. Main parameters have been too applied such as rotational speed, traverse speed and specific tool design. It is also well understood that the effect of these important parameters on the FSP properties are the major topics for researchers. Obviously, FSP parameters were selected by trial and error to fix the process to obtain sound and defects free materials. FSP achieved three distinct zones, namely as nugget or SZ (stir zone), TMAZ (thermo-mechanically affected zone) and HAZ (heat affected zone) due to the stirring action of a rotating tool with axial force and the plate travel speed [14]. FSP causes grain refinement due to the dynamic recrystallization in the SZ which increase the tensile properties without loss of ductility [15]. According to Mishra *et al.* (2000) has been reported that the Al-Zn-Mg alloys subjected to FSP exhibits superplastic formation at a 480°C and the thermal stability of refined grains achieved during FSP at around $\sim 500^\circ\text{C}$ [16]. This is considering a high temperature process when fine-grains formed during FSP grow rapidly under the influence of thermal cycles in air cooling. Thus, FSP Al alloys results in the formation of a homogeneous fine grained structure at around $\sim 80-90\%$ developed high angle grain boundaries about $\sim 80-90\%$. Notably, the grain refinement caused by secondary Al_3Sc nanoparticles, precipitation strengthening and sub-grain strengthening mechanisms. The presence of such nanoparticles or dispersoids is known to restrict the grain growth through Zener pinning [17]. The present work has two highlights on some deleterious effects like Zn vaporization due to high rotational speed of 1000 rpm and traverse speed of 70 mm/min and formation of Al_3Sc chunky particles due to high Sc (0.87 wt.%) content in aluminium alloys. Such defects have been identified through OM, FESEM, TEM and SEM fractographs analysis. Investigations determine the mechanical properties should be optimized when process parameters are avoided to Zn vaporization or hair line crack forming on the stir region during FSP [18]. The present research has to determine the effects on microstructure and mechanical properties under T_4 +FSP and T_4 +FSP+Aged at 140°C for 2h conditions of the 7xxx series of Al-Zn-Mg alloys.

2. Experimental Procedure

The present experimental works were carried out on aluminium alloys through cast metallurgy route and alloy compositions were tested by ICP-AES and AAS methods shown in Table 1. The physical and thermal properties of aluminium alloys are mentioned in Table 2. In general, thermal conductivity depends on solute content, while Mg content increasing with thermal conductivity decrease. The evolution of microstructure was investigated by optical microscopy (OM), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) tensile fractographs analysis. The properties of the prepared aluminium alloys were evaluated at T_6 , T_4 +FSP and T_4 +FSP+Aged at 140°C for 2h conditions, respectively. The metallographic specimens were collected from

as-cast plate and subjected to solution treatment at 465°C for 1h and immediately quenched in water (A). Similarly, sample was collected from FSP region and mechanically polished using different grades of emery papers with the help of rotating wheel using water +Al₂O₃ powder. Final polishing was done using diamond paste with particle size of 1-µm on a rotating wheel. Then the samples were etched by using 5% HCl solution (volume fraction) for reveal microstructures. The T₄ sample was selected for artificial ageing treatment.

Table 1. Chemical Composition (in wt.%) of Studied Aluminium Alloys

| Alloy no. | Zn | Mg | Sc | Si | Fe | Al | Zn+Mg | Zn/Mg |
|-----------|------|------|------|------|------|------|-------|-------|
| 1 | 8.34 | 3.30 | - | 0.02 | 0.04 | Bal. | 11.64 | 2.53 |
| 2 | 8.20 | 5.70 | 0.87 | 0.88 | 0.42 | Bal. | 13.90 | 1.44 |

Table 2. Physical and Thermal Properties of Zn-Mg Alloy

| | |
|---|-------------------------|
| Density (g/cc) | 2.72 |
| Melting point (°C) | 652 |
| Modulus of elasticity (GPa) | 68.9 |
| Poissons ratio | 0.33 |
| Thermal conductivity (W/m -K) | 167 |
| Thermal diffusivity (m ² /s) | 5.37 × 10 ⁻⁵ |
| Specific heat capacity (J/g°C) | 0.869 |

Table 3. FSP Tool Characteristics and Process Parameters

| | |
|--|--------------------------|
| Shoulder diameter (mm) | 20 |
| Pin diameter (mm) | 3 |
| Pin height (mm) | 3.5 |
| Clockwise and unidirectional, tool rotational speed(rpm) | 1000 |
| Tool traverse speed (mm/min) | 70 |
| Downward pressure (kN) | 15 |
| Tool tilt angle (°) | 2.5 |
| Number of passes | two |
| Plate dimension | 150×90×8 mm ³ |

The artificial ageing was performed in electrical resistance muffle furnace with precision controlled temperature at 140±2°C. The FSP samples were collected such as T₄+FSP and T₄+FSP+Aged at 140°C for 2h and stored for different types of testing. Differential scanning calorimetry (DSC) was performed on the EXSTAR TG/DTA 6300 equipment with a heating rate 10°C/min. The FESEM micrograph with EDAX analysis was presented at T₄+FSP+Aged at 140°C for 2h condition of aluminium alloys. The transmission electron microscope (TEM) analysis was performed using twin-jet electro-polishing (solution was 75% CH₃OH and 25% HNO₃) at 12 V and 35 °C. All the imaging was carried out using at Techai G20 S-TWIN at 200 kV. The hardness measurements of specimens were carried out in longitudinal direction. The specimens for tensile tests were cut in the longitudinal direction parallel to the direction of processing. The dimensions of tensile sample (ASTM: E-8/E8M-11sub-size) has shown in Fig. 9. The process parameters were kept on constant at every unidirectional pass of FSP of 1000 rpm and 70 mm/min as shown in Table 3. The mechanical testing was carried out an electro-mechanically controlled universal testing machine (25 kN, H25 K-S, UK) with cross head speed 1 mm/min after FSP and results are calculated and exhibited at bar diagrams format in Fig. 10.

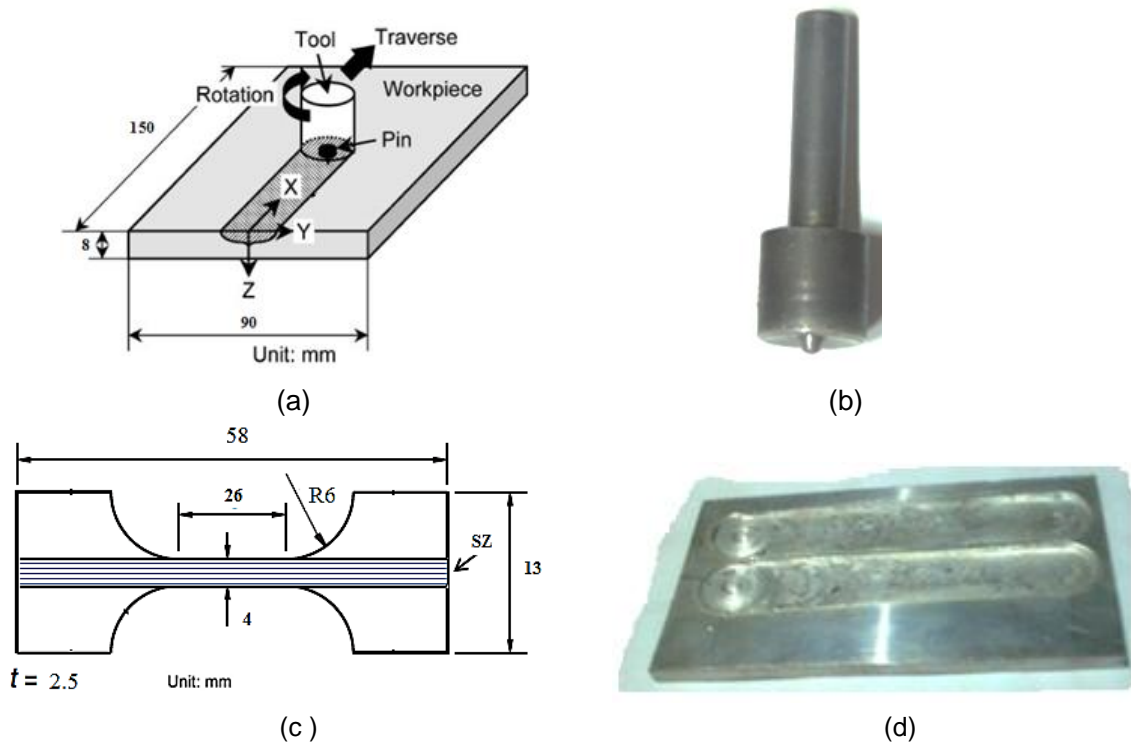


Fig. 1 (a) Schematic diagram of FSP set-up (150×90×8 mm³), (b) Illustration of tool configuration, (c) Geometry and dimensions of the tensile test sample (58×13 mm²), (d) Illustration of double passes FSP plate (150×90×8 mm³).

3. Results and Discussion

The Al-Zn-Mg is a novel alloy with light weight, high specific strength and good mechanical properties. A little amount of Sc utilized in alloys with specific control even with a little variation possibly may have a great impact on material performance. Zn and Mg elements also cause of quench sensitivity, which is the indirect measurement of the phase transformation which indication of the type, size and amount of precipitates formation in aluminium alloys. The aluminium alloy in the solutionizing (T) state has the lowest conductivity due to the decrease of the mean free path of electrons. The main mechanisms are mismatch of sizes between the solute and solvent atoms, and the electron/atom ratio in solid solution is different completely [20]. Many researchers were suggested on the FSP of engineering alloys to promote process windows which have been applied in the industrial application effectively [21][22]. These alloys can be used in many applications such as cylinder heads, engine blocks, aerospace housings, gear pumps, aircraft fittings, general automotive castings, and marine structures [23]. Sc has been found to result in grain refinement through the eutectic form at 655°C of Al₃Sc compound favours the nucleation of aluminium grains while Ti, V, Cr forms peritectic with aluminium. The primary particles (Al₃Sc) are formed in different shapes in different cooling rate, which act as a grain refining of aluminium alloys. Grain refinement by Sc inoculation has been commonly used to improve the mechanical properties of aluminium alloys. It is fact that increase in Sc initially strength increases, however more than 0.6 wt.% mechanical properties remain same. Sc is the costlier element and it can substitute by other rare earth elements and higher order alloy combination. The addition of Mg in binary Al-Sc alloys has been shown to form primary particles (Al₃Sc) much lower than the eutectic composition of Sc in such aluminium alloys [24]. Mg improves the mechanical properties through solid solution strengthening. There is extensive investigation reported

on cubic Al_3Sc particles (melting point of $1320^\circ C$). Comparative study is reported on crystallographic mismatch between Al & Al_3Sc ($\sim 1.5\%$) is higher than between Al & Al_3Ti ($\sim 1.18\%$) and Al & Al_3Zr ($\sim 0.7\%$) in some specific crystallographic plane and room temperature [25], [26]. Fig. 2 indicates the existence of β , τ , η , δ , ζ , θ , γ , ϵ , ζ_1 , ζ_2 , ζ_3 and ζ_4 phases are involved [27].

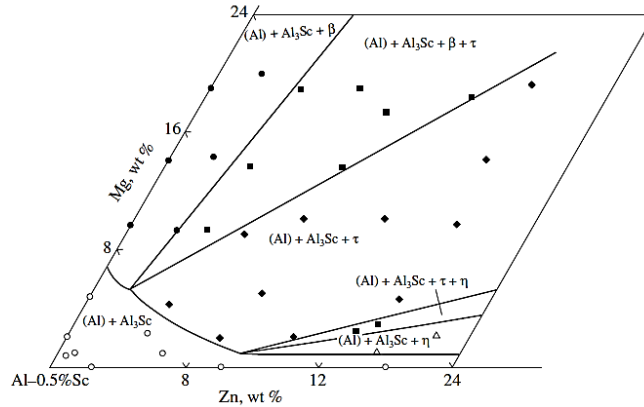


Fig. 2. Section of the isothermal tetrahedron of the Al-Zn-Mg-Sc system ($300^\circ C$) [28].

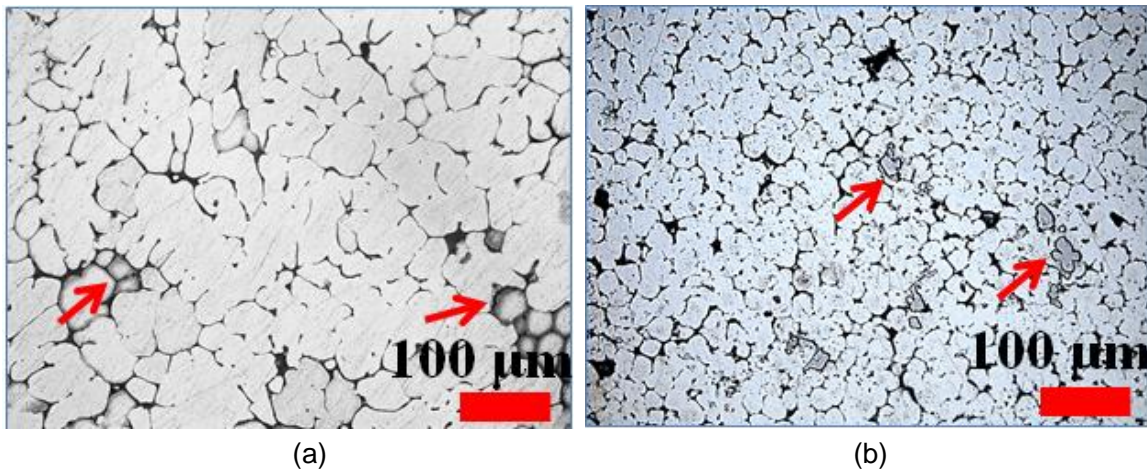


Fig. 3. Optical micrographs of as-cast aluminium alloys (T4 condition): (a) Alloy-1, (b) Alloy-2.

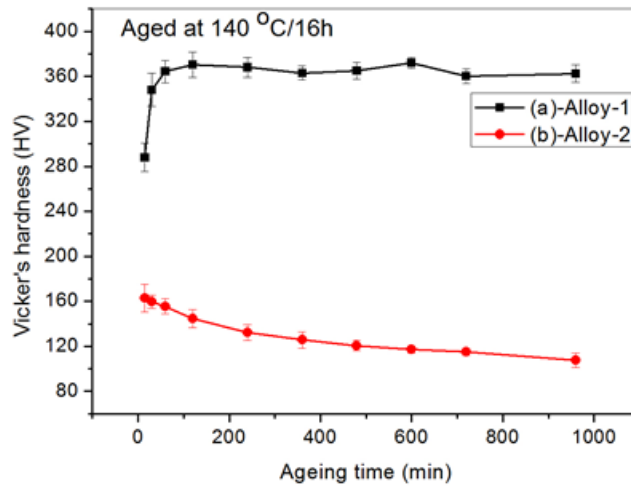


Fig.4. Illustration of ageing curves of aluminium alloys (a) Alloy-1, (b) Alloy-2.

Fig. 3 shows Al alloys exhibited grain boundary segregation with black spots on the boundary regions. Due to high solute contents (11.64 wt.%) prone to segregation of eutectic constituents on the grain boundaries as shown in Fig3(a). Fig. 3(b) shows Al-Sc chunky particles and several black spots on the grain boundaries owing to high Sc content (0.87 wt.%) and high solute content (13.90 wt.%), respectively. Since, both of the alloy constituents are very high levels, so present solution treatment (465 for 1h) may not be feasible to optimum dissolve second phases in the matrix.

Fig. 4 shows ageing curve of Al alloys. The maximum hardness values developed by age-hardening alloys ... MgZn phases as foreign atoms in the lattice of the solvent atoms in the solid solution. These are the main causes of more lattice distortion to make alloys harder. Elements like Zn and Mg are the substitutional atoms in aluminium matrix produced elastic lattice distortions by solid solution strengthening. Hence, the main strengthening mechanisms exhibited in these alloys are an age-hardening by structural precipitates of MgZn formed during artificial ageing treatment. These precipitate fine particles act obstacles to dislocation movement and thereby strengthen the Al-Zn-Mg alloy [29]. Fig. 4(a) shows ageing curve exhibited maximum hardening effects at around 100 min ageing time ... MgZn particles with optimum size in this ageing treatment regime. Fig. 4(b) shows ageing curve exhibited minimum hardening effects due to high Sc content produce Al-Sc chunky particles or agglomeration tendency of Al-Sc particles aggravated with high solute content in this ageing treatment regime.

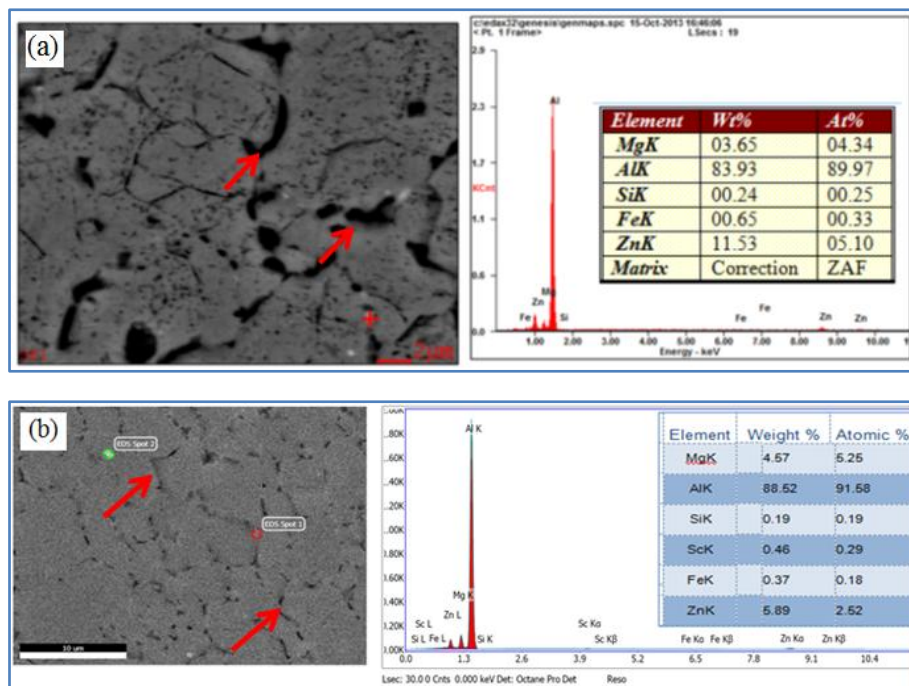


Fig. 5. The FESEM micrographs exhibiting black holes with EDAX analysis at FSP+Aged at 140°C condition: (a) Alloy-1, (b) Alloy-2. (1000 rpm and 70 mm/min).

Fig. 5 shows FESEM micrograph with EDX analysis after FSP+Aged at 140°C for 2h condition. Fig. 5(a) shows FESEM micrograph exhibited several black spots (indicated by red arrows) indicate Zn vaporization may be occurred due to high heat generation (~550°C) during FSP. This deleterious effect main causes of low mechanical properties of FSP aluminium alloy. Fig. 5(b) shows FESEM micrograph exhibited formation of grain boundaries with continuous network of black lines (indicated by red arrows) may be same reason

like Zn vaporization aggravated with high density of Al₃Sc particles during FSP aluminium alloy.

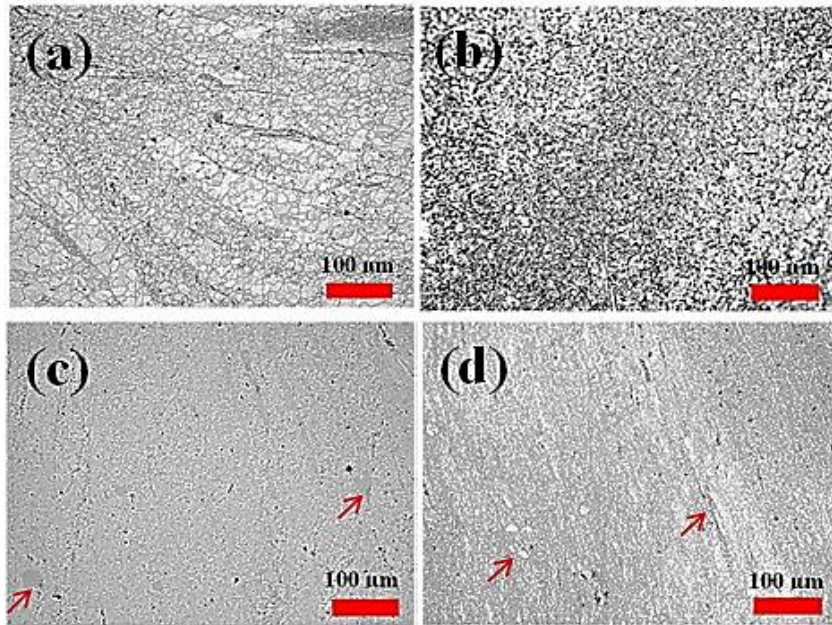


Fig. 6. Illustration of optical micrographs collected from SZ for Alloy-1 at (a) T₄+FSP, (b) T₄+FSP+Aged at 140 °C/2h condition; for Alloy-2 at (c) T₄+FSP, (d) T₄+FSP+Aged at 140 °C/2h condition. (1000 rpm and 70 mm/min).

Fig. 6(a) shows optical micrograph exhibited fine and equiaxed grains of SZ after FSP condition of Alloy-1. The microstructure of present alloy consists of equiaxed grains that can be induced during the dynamic recrystallization process. It is well known the FSP generates substantial fictional heat and intense plastic deformation for creating fine recrystallized grains in the SZ. Therefore, the FSP improves the grain boundary formation and increases suitable sites for the precipitation of nucleation in post ageing treatment at 140°C for 2h as shown in Fig. 6(b). Fig. 6(c) shows the optical micrograph exhibited several chunky particles (marked by red arrows) even after T₄+FSP condition. Also, some fine cracks are exhibited due to FSP torsional effects could not be resisting such cracks as presence of coarse Al₃Sc particles. Both the causes are pertinent of high Sc content (0.87 wt.%) due to formation of Al₃Sc chunky particles as proven by TEM analysis and mechanical properties. In case of post ageing treatment at 140°C for 2h for Alloy-2 (Fig. 6.d) exhibited fast coarsening effects due to Al₃Sc chunky particles and high Zn content (8.20 wt.%) formed high fraction of high angle of grain boundaries, high mobility of atoms would affect its thermal stability adversely during FSP. These types of Al₃Sc chunky particles are unable to the pinning action to refine grains by Zener drag mechanism. Moreover, dynamic grain growth mechanism is prominent rather than recrystallization process. There are three types of adverse effects have been postulated such as hair line cracks generation, several black spots due to Zn vaporization, and several Al₃Sc chunky particles formation (all marked by red arrows). The DSC curve of the experimental alloys in the present state is presented in Fig. 7. The DSC curves show the main two transition points at around 475-510°C (exothermic reaction) and 615°C-620°C (endothermic reaction), respectively. Two alloys show the similar trend and almost the reaction temperature (475°C-510°C) in this state, which indicates that the combined effects of high Zn content and high Sc content main causes of formation of high density of Al₃Sc phases exhibit anti-recrystallization effects. The endothermic reaction (615°C-620°C) is mainly caused by the formation and growth Al₃Sc phases. The precipitate characteristics in the experimental alloys have been shown in the TEM analysis in

Fig. 8. It is observed that the precipitates in the post ageing treatment at 140°C for 2h alloy have a coarse and spherical shapes and an average grain size ~20-45 nm as shown in Fig. 8(a). Some of needle shape precipitates are long size ~130-200 nm. Due to post ageing at 140°C for 2h, homogeneous nucleation sites are activated in the matrix and consequently the precipitated can be formed in the mostly spherical shape within the matrix with a uniform distribution. The high solute content (11.64 wt.%) with high temperature generates during plastic deformation are the main causes of formation of coarse Mg₂Zn precipitates. This is attributed to the loss of metastable age-hardening precipitates during FSP, which reduced the precipitation hardening contribution [30], [31].

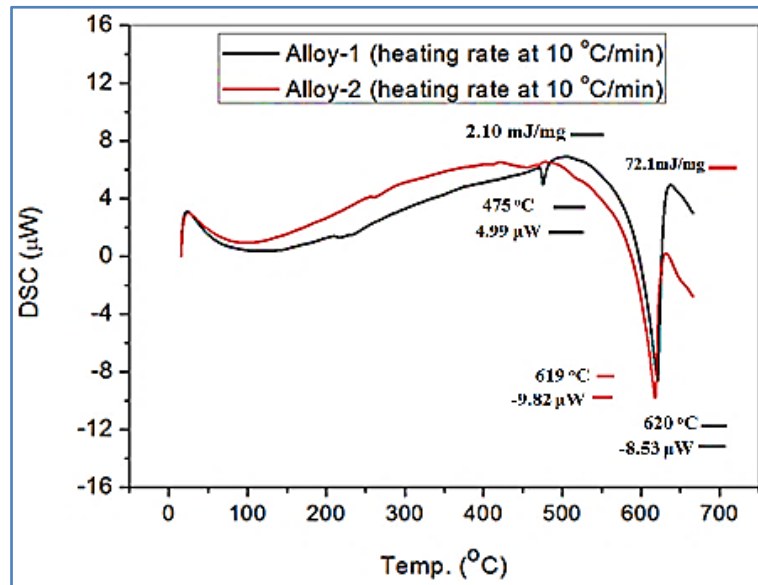


Fig. 7. DSC analysis of aluminium alloys after FSP+Aged at 140°C/2h condition. (1000 rpm and 70 mm/min)

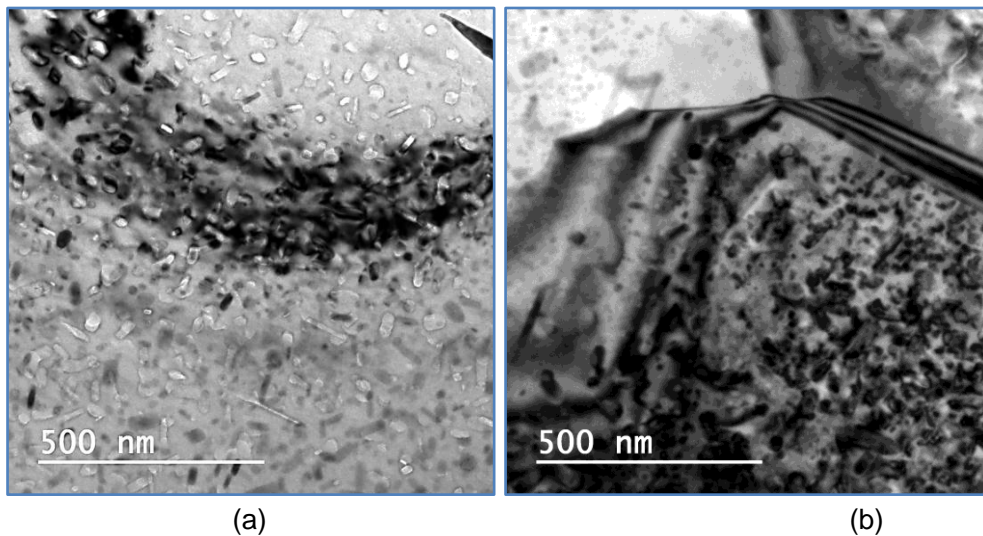


Fig. 8. TEM micrographs collected from SZ of aluminium alloys after T₄+FSP+Aged at 140°C/2h condition: (a) Alloy-1, (b) Alloy-2. (1000 rpm and 70 mm/min).

Fig. 8(b) shows TEM micrograph exhibited different shapes and morphology of precipitates after Sc addition of Al₃Sc precipitates. Fine and coarse mixture type coherent spherical Al₃Sc particles appear in Alloy-2 after T₄+FSP+Aged at 140°C for 2h. These particles had strong interaction with the dislocations and the grain boundaries during FSP. Some of precipitates are big size ~120-200 nm. This is because

agglomeration tendency among the coarse precipitates of high solute atoms and high density of SA particles facilitated during FSP. Appearance of long dislocation line introduced into the FSP alloy. Since aluminium has high stacking fault energy, so the presence of dislocations suggest that recrystallization was not complete and that it had a more dynamic nature than static character. The refinement of microstructure during FSP is an inherent feature of the process. It is usually considered as a result of dynamic "phase-precipitation around the Al₃Sc particles to reduce the oversaturated degree of solute atoms in the matrix [32]-[34]. Hence, the effect of precipitate strengthening has not obvious and the strength of Alloy-2 is lower than that of Alloy-1 as shown in Fig. 10.

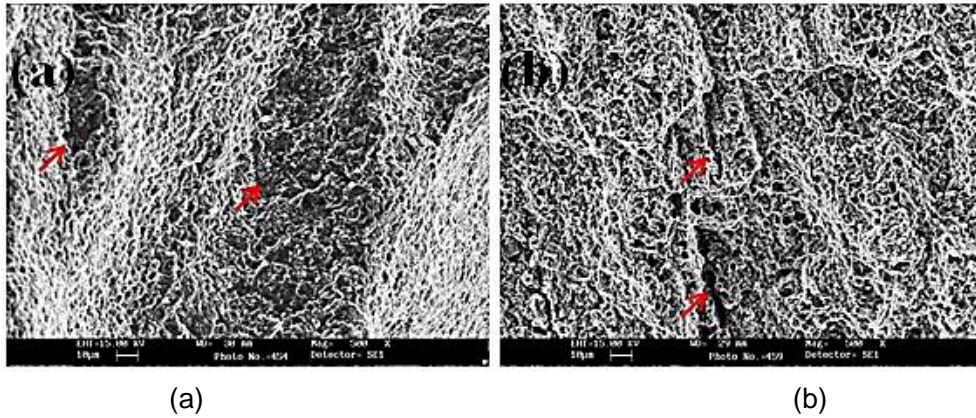


Fig. 9. SEM tensile fractographs at T₄+FSP+Aged at 140°C/2h condition: (a) Alloy-1, (b) Alloy-2. (1000 rpm and 70 mm/min).

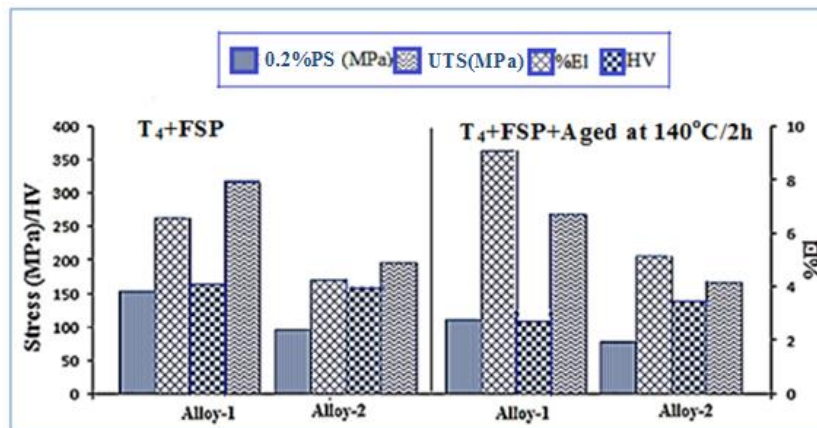


Fig. 10. Illustration of bar diagrams of mechanical properties at different conditions of AZn-Mg alloy after double passes FSP. (1000 rpm and 70 mm/min).

In Fig. 9 shows tensile fractographs exhibited ductile manner of entire fracture surface for both the alloys at the present condition. In Fig. 9(a) shows several black holes/cracks (indicated by red arrows) seems stress raiser points created by Zn vaporization during FSP (causes for high heat input; where, heat input = rpm/traverse speed = 1000/70 = 14.39). Fig. 9(b) shows several crack lines (indicated by red arrows) which may have generated along the Al₃Sc chunky particles or Zn vaporization points, also, accelerated after post ageing treatment at 140°C for 2h. Formation of Al₃Sc chunky particles (for 0.87 wt.% Sc content) facilitated due to high angle grain boundaries (generally 80-90%) during FSPed. It is observed that the mechanical

- 5) The tensile fractographs revealed Al_3Sc chunky particles, the source of crack formation which might be the reason for low tensile properties. Even after FSP these particles were found to be source of crack initiation.
- 6) The tensile properties have been evaluated for T_4 FSP and T_4 FSP+Aged at $140^\circ C$ for 2h conditions. The tensile properties of Alloy-1 decreased marginally such as ultimate tensile strength of 14.77%, 0.2% proof strength of 26.74%, hardness of 30.42%, but elongation increase of 37.88% after T_4 FSP+Aged at $140^\circ C$ for 2h. Similarly, the tensile properties of Alloy-2 increased marginally such as ultimate tensile strength of 5%, 0.2% proof strength of 15%, elongation of 19%, but decreased hardness of 6.3%, after T_4 FSP+Aged at $140^\circ C$ for 2h. Reason for increased strength during post ageing treatment effects of fine nucleation of $MgZn$ homogeneous precipitates and fine grains, fragmentation of Al_3Sc dispersoids through Orowan mechanism.

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