Heat Treatment and Friction Stir Processing Effects on Mechanical Properties and Microstructural Evolution of Sc Inoculated Al-Zn-Mg Alloys

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Abstract It has studied the influence of different artificial ageing parameters on Vicker’s hardness characteristics and friction stir processing on surface modification of scandium inoculated Al-Zn-Mg alloys. The aluminium alloys were solution treated at 465°C for one hour, quenched in water and artificial ageing at 120°C, 140°C, and 180°C at different time of ageing up to twenty hours. Further, friction stir processing healed the casting porosity and refine cast microstructures. These microstructural changes led to a significant improvement in both strength and ductility the aluminium alloys (7xxx series). Generally, high tool rotation rate is beneficial to break coarse second-phase particles, heal the casting porosity, homogenize and consequently increase strength. Therefore, friction stir processing is adopted to modify the microstructure of cast aluminium alloys to refine grain and enhance mechanical properties.

Keywords: solutionizing, artificial ageing, inoculation, fine microstructures, friction stir processing


1. Introduction

Heat treatment is an important operation in the final fabrication process of any engineering component. The objective of heat treatment is to make the metal better suited, structurally and physically, for some specific application. Similar type of heat treatment, solution heat treatment of aluminium alloys allows the maximum concentration of a hardening solute to dissolve into solution by heating the alloy to a temperature in which single phase will be created. Once the alloy has been held for considerable amount of time to ensure complete solutionizing and homogenous phase, it is quenched rapidly such that the solute atoms do not have enough time to precipitates out of solution. As a result, a super saturated solution exists between the solute atoms in aluminium matrix. In consequence, precipitation strengthening solid solutions involves the formation of finely dispersed precipitates during ageing heat treatment, which may include either natural ageing or artificial ageing. The ageing must be accomplished not only below the equilibrium solvus temperature, but below a metastable miscibility gap called the Guinier-Preston (GP) zone solvus line [1-10]. The Al-Zn-Mg (7xxx series) this kind heat treatable alloy which has been readily ageing response if suitable temperature and time provided to formation stable η phase and its precursors. These alloys benefit from a good precipitation strengthening, through complex decomposition processes involving both stable and metastable phases which can industrially beneficial. In Al-Zn-Mg system, a preferential interaction between small zinc atoms and large magnesium atoms on the aluminium lattice, involving some clustering (in order to reduce lattice strain energy), plays an important role in the decomposition processes during ageing [11,12]. However, this series of alloys are susceptible to stress corrosion cracking (SCC) in the highest strength temper (T6) but at over-aged heat treatment conditions decrease stress corrosion cracking susceptibility and hence reduces the strength [13]. In literature pointed out, precipitation free zone (PFZ) and η (MgZn2) particles are finely dispersed in the grains but also accumulated in the grain boundaries, it’s to be responsible for intergranular corrosion. Hence, the density of fine precipitates which is responsible for the high strength and the stress corrosion cracking resistance of the alloy is generally thought to be controlled by the heat treatment. Furthermore, Al-Zn-Mg alloys are made more harden through inoculation effect of Sc [14,15]. The addition of Sc resulted in Al3Sc dispersoids that greatly refined grains and restrained recrystallization process, which enhanced the strength and corrosion resistance. By forming Al3Sc particles in aluminium alloys, Sc known to reduce hot tearing, shrinkage porosity, to give a more uniform microstructure and can retard the grain growth by Zener-effect [16]. Scandium forms a limited- solubility eutectic diagram with aluminium [in Figure 2] [17]. Inoculation of the melt with Al3Sc (L12-type) phase leads to heterogeneous nucleation of aluminium grains [18]. It is observed that the average amount of Zn and Mg retained in a solid solution is very low within a grain interior. Zn and Mg tend to segregate at grain boundary areas due to partitioning effect during freezing. Since the percentage of
area accounted for by grain boundaries is much higher in the alloys containing Sc, their partitioning effect must be increased with Sc addition. The sequence of precipitation in aluminium alloys strongly depends on the history of the materials, including quenching conditions, natural ageing and further heat treatment. Different processes may be involved: dissolution, coarsening or phase transformation from metastable precipitates to a more stable phase. GP zones formed at low temperature after quenching (typically room temperature); can act as nucleation sites for more stable precipitates. When the temperature is increased from the pre-ageing temperature up to the ageing temperature, dissolution of GP zones occurs. This dissolution is called reversion, can be partial. When the ageing temperature is higher than the reversion temperature, the GP zone dissolution is complete. In spite of this complete reversion, ageing can still result in a fine distribution of η-particles. As η-particles are incoherent with the matrix (and have a relatively high interfacial energy), metastable precipitates (η′), with a higher level of coherency (and lower interfacial energy), may be expected to form preferentially at lower temperatures because of a lower activation barrier for nucleation. The common metastable phases in the 7xxx system are GP zones and η-particles, respectively. According to the binary phase diagram of Al-Zn (in Figure 1), because of existence of solvus solubility, at temperature decreasing, decrease the solubility of solid solution. The decreased solubility of solid solution leads to its saturation and the material becomes thermodynamically unstable and therefore will tend to decompose into two new phases [19]. The effect of the ageing treatment is evaluated by Vicker’s hardness testing and metallography. Hardness measurements can provide a good indication of the material strength and since strength is related to the number, type and spacing of precipitates then hardness measurements can be used to monitor the precipitation process. Vicker’s hardness tests were done with the purpose of analysing the influence of the precipitated phases, formed during the ageing treatment, on the hardness of alloy. Moreover, tensile testing has been carried out to signify the materials mechanical properties to ensure engineering application. The tensile fracture surface examination can be revealed precipitates morphology, size and mode of fracture, alloy chemistry and heat treatment processes.

In addition, friction-stir processing (FSP) is an emerging surface-engineering technology that can locally eliminate casting defects and fine microstructures, thereby improving strength and ductility, increase resistance to corrosion, enhance deformability, and improve other properties [20]. Essentially, FSP is a local thermo-mechanical metal working process that changes the local properties without influencing properties in the remainder of the structure [21]. During FSP tool design and process parameters has been optimised likely, tool rotation rate 1000 rpm and a traverse speed of 70 mm/min, the total FSP length is around 150 mm from the pin entry to the pin exit. A tool with a concave shoulder 20 mm in diameter, and a conical pin 5 mm in root diameter, 3.5 mm in tip diameter, and 5 mm in length is used. During material characterisation several tests were conducted, therefore Vicker’s hardness testing, mechanical testing, electron probe microanalysis (EPMA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis on present experimental alloys. Finally, it can be more emphasised on heat treatment and surface modification aspect through friction stir processing on cast Al-Zn-Mg alloys with Sc inoculation.

2. Experimental Procedures

The four types of aluminium alloys were prepared by cast metallurgy with pure Zn, pure Mg and master alloy Al-2wt.%Sc. Basically, prepared alloys were 7xxx series of Al-Zn-Mg without and with Sc addition. The muffle furnace was used to melt the alloys at 780°C. The melt was cast in an air by a steel mould in plate shape (150×90×8 mm³). The chemical composition of 7xxx series of aluminium alloys were determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) and atomic absorption spectroscopy (AAS) methods and is shown in Table 1. The specimens were subjected to the following heat treatment: solution treatment at 465±5°C for 1 h, followed by quenching in water at room temperature then kept for seven days for natural ageing. Then, artificial ageing performed at 120±2°C, 140±2°C
and 180±2°C respectively. The metallographic specimens were examined under optical microscopy after etching with a modified Keller’s reagent (2.5 ml HNO₃+1.5 ml HCl+1 ml HF+ 95 ml water). The as-cast samples were analyzed by electron probe microanalysis (EPMA) with EDS to examined segregation on grain boundary. Microhardness tests were done with a Vicker’s diamond indenter with a pyramidal shape with 10 kg load at different time intervals 5, 15, 30, 60, 120, 240, 360, 480, 600, 720, 960 and 1200 min. Five measurements were taken randomly for each sample and the average value and the standard deviation were calculated. The hardness values were recorded immediately after ageing and continued for twenty hours at above interval. The tensile tests were carried out by using an Instron testing machine at a cross head speed of 1 mm/min as shown in Table 2. The tensile specimen having 26 mm gauge length, 4 mm width, 2.5 mm thickness and 58 mm length with full processed zone by double passes. During friction stir processing (FSP) as following parameters were controlled to obtain sound defect-free test plates. FSP was conducted at a tool rotation rate 1000 rpm and a traverse speed of 70 mm/min. the total FSP length was around 150 mm from the pin entry to the pin exit. A tool with a concave shoulder 20 mm in diameter, and a conical pin 5 mm in root diameter, 3.5 mm in tip diameter, and 5 mm in length was used. Therefore, in order to obtain full processed zone plates by double passes plates. The field emission scanning electron microscopy (FESEM) was used to analyse the phases, along with energy dispersive spectroscopy to determine their chemical compositions. FESEM was conducted on QUANTA 200F, 30 kV model. Similarly, the fracture surfaces of tensile specimens were studied by scanning electron microscopy (SEM) analysis. And an as-cast grain size was measured by mean linear intercept method. The X-ray diffraction (XRD) method to identified different beneficial phases of aluminium alloys. The differential scanning calorimeter (DSC) measurements were performed in an EXSTAR TG/DTA 6300 equipment with a 10°C/min heating rate until 600°C in nitrogen atmosphere on solution treated and aged at 140°C for 6 h. Specimens for transmission electron microscopy (TEM) with 3 mm diameter were cut from the as-cast 10×10 mm² discs and thickness reduced to 0.1 mm. Then, the TEM samples were prepared 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 G² 20 S-TWIN at 200 kV.

3. Results and Discussion

Aluminium alloys are generally categorized in two classes: heat-treatable and non-heat-treatable alloys. Following the international AA (Aluminium Association) designation, the heat treatable alloys are the 2xxx series (Al-Cu alloys), the 6xxx series (Al-Mg-Si) and the 7xxx series (Al-Zn-Mg). These are the alloys get their strength from artificial ageing producers and hence from precipitation hardening. At first, addition of Sc to one of the heat-treatable alloys in order to benefit from the added effect of the Al₃Sc-induced hardening seems the most obvious. For instance the ageing temperatures for 2xxx alloys vary between 160 to 190°C, for 6xxx alloys between 160 to 205°C and for 7xxx alloys between 95 to 180°C. All these temperatures are considerably lower than 350°C, at which the Al₃Sc phase in this research is formed. Consequently, adding Sc to non-heat-treatable alloys as for instance the 1xxx series (>99% pure Al), the 3xxx series (Al-Mn) and the 5xxx (Al-Mg) is however promising. These alloys get their strength from strain hardening and from solid solution hardening. Scandium additions to aluminium alloys are promising. Due to the homogenous distribution of nano-sized Al₃Sc precipitates high strengths can be achieved. The formation of the precipitates increases the hardness by several hundreds MPa. Furthermore, these precipitates exert a strong Zener drag on advancing dislocations and grain boundaries, which hinders recrystallization and thereby facilitates the formation of small grained materials. These alloys differentiate themselves from other aluminium alloys by their excellent properties at elevated temperatures. The diffusion of Sc in aluminium is low and consequently the precipitates coarsen slowly. The strengthening qualities of the Al₃Sc phase can be attributed to modulus hardening, coherency hardening and order strengthening. Since coherency strengthening may form a major contribution to the hardness of an alloy (Al-Sc alloy), it is important to keep the size of the precipitates below the critical value at which the first misfit dislocation is formed to relieve the misfits strains. Approximately this will happen the number the lattice planes at the grain boundary multiplied by the lattice misfit is equal to one Burgers vector. For the Al₃Sc interface the critical diameter is: \( d_{\text{crit}} = b/c = 21.0 \) nm. However, this critical value will be higher at elevated temperatures due to larger thermal expansion coefficient of Al and also due to the higher concentration of Sc in solid solution, which will slightly the lattice spacing of the matrix. In a deformed microstructure, the precipitation is less homogeneous. Precipitates will then nucleate preferentially on cell walls and on individual dislocations. The heterogeneous nucleation effects are more prominent when the ageing has been performed at 400°C as compared to ageing at lower temperatures. The increased strength is only one of the useful consequences of the formation of the Al₃Sc phase. Another aspect is the influence the precipitates have on the recrystallization behaviour of the material. It is well known that randomly distributed hinder the motion of advancing grain boundaries. This so-called Zener drag can be expressed in the form: \( p_z = k.f(r) \) where \( k \) is a factor based on the interfacial energy between the dispersoid and the matrix. If at constant volume fraction, the precipitates coarsen, the Zener drag will decrease rapidly [22]. This drag is such that after the aged microstructure is deformed, the temperature required for recrystallization increases. Clearly the presence of the precipitates has prevented the recrystallization of the heavily deformed microstructure completely up to temperatures of 550°C. At these temperatures most of the Sc dissolves in the Al and the remaining precipitates will coarsen rapidly, minimizing the Zener drag on the boundaries. The Zener drag reaches its maximum for very small precipitates. Even if an annealed microstructure is deformed, after which a recrystallization is attempted, the immediate nucleation of precipitates will prevent this. The deformed microstructure
consists of (screw-) dislocations, which arrange themselves in a cell structure with nearly defect free interiors and small orientation differences between the cells. This microstructure is typical for deformed Al specimens and is a consequence of the high stacking fault energy and hence easy with which screw dislocations cross-clip. At the start of the heat treatment, these cells are present but the precipitates are not, but they will nucleate fast enough from the solid solution to preserve the cell structure.

During precipitation hardening is the interaction of a dislocation with a field of obstacles. The obstacles of primary interest here are precipitates defined broadly to include GP zones and metastable second phases as well as stable phases. Consider a row of such obstacles and a dislocation moving on a slip plane. For slip to occur, the dislocation must either move around the particles or through the particles. An active dislocation will select from the various paths available to it the path where the least energy is expanded. The dislocation may avoid the particles or obstacles by leaving the slip plane in the vicinity of each particle, or it may avoid the particles by the Orowan mechanism. In this mechanism, the dislocation bends between the particles leaving a dislocation ring about each particle. In either case, energy must be supplied to increase the total length of dislocation line; the stress required is, neglecting the numerical factor, roughly \( (Gb)/L \) where \( G \) is the shear modulus, \( b \) is the Burgers vector, and \( L \) is the spacing between obstacles. The ageing sequence at temperatures below 190°C is usually given as \( \alpha \) solid solution\( \rightarrow \)GP zones\( \rightarrow \eta \rightarrow \eta' \), where the plate-like \( \eta \) transition phase and the lath or rod shaped \( \eta'(\text{MgZn}_2) \) equilibrium phase are considered hexagonal. According to Graf and Schmalzried and Gerold suggested that the GP zones become ordered with Zn and Mg atoms on alternate \{100\} planes within the zones. The difference in the growth kinetics of the two types of particles, suggests that the particles aged at a higher temperature reach a metastable equilibrium concentration of solute rapidly and undergo coarsening (\( r \propto t^{1/3} \)), whereas the lower ageing temperature produces particles which do not reach such a solute concentration and follow a different growth law (\( r \propto t^{1/6} \)) [23]. The stress corrosion cracking (SCC) in Al-Zn-Mg alloys are complex and a great number of parameters are involved. Cracking is generally initiated on the surface of the component when small surface imperfections generate a fissure that will propagate through the component. The precipitation hardening process is directly responsible for stress corrosion cracking susceptibility in high strength aluminium alloys. These materials are immune to stress corrosion cracking in the as-quenched state where grain-boundary precipitation is prevented, but they usually become susceptible with increasing precipitation hardening reaching a maximum before peak strength. Over-aged heat treatments decrease stress corrosion cracking susceptibility and hence reduces the strength. Furthermore, the density of the fine precipitates which is responsible for the high strength and the stress corrosion cracking resistance of the alloy is generally thought to be controlled by the heat treatment. According to Ward and Lorimer the Zn content is lower in the precipitate-free zones (PFZ) or remains at the level of the grain interior. At temperature during heat treatment a Zn ‘profile’, i.e. a lower Zn content appears at the grain boundary or in its vicinity the stress corrosion sensitivity of the aluminium alloy decreases [24,25,26].

In heat treatable alloys, precipitation hardening is the dominant mechanism responsible for strength and local strength variations. Thus, the friction stir processed materials all the known mechanisms of strengthening of polycrystalline alloys can play a role. The mechanisms for increasing the critical resolved shear stress (CRSS) of the slip planes are: precipitation strengthening, solid solution strengthening and dislocation strengthening. The response to stress of the polycrystal will depend on the CRSS and factors such as: local grain size, if the grain size is sufficiently small this will lead to grain boundary strengthening and crystallographic orientations of grains with respect to each other, therefore, the crystallographic texture [27]. The as-cast alloys chemical composition are determined by ICP-AES and AAS methods, is shown in Table 1. The Zn and Mg are main constituents of 7xxx series of Al-Zn-Mg alloys. The effect of Zn and Mg on the strength of age-hardened Al-Zn-Mg alloys in mainly function of \( Zn + Mg \) contents. The Zn: Mg ratio controls the Zn-bearing constituents. With a ratio over 2, \( \text{MgZn}_2 (\eta) \) is formed to main hardening in among the all phases in age-hardening phenomena [28]. As impurities Fe and Si appear to be mainly responsible for affecting adversely the ageing response and on fracture toughness [29]. The optical micrographs of the studied alloys are shown in Figure 3. Coarsened as-cast grains, dendritic structures, segregation and eutectic formation on grain boundary observed in the Alloy-1 and Alloy-2 without Sc addition [Figure 3(a-b)]. While, minor Sc can significantly refine the as-cast grain in the Alloy-3 to Alloy-4. In the Alloy-3 minor Sc (0.2%) addition cannot refine dendritic structure but for Alloy-4 dendritic structure completely disappeared to obtain fine average grains sizes of 40.3 μm and 21.7 μm, respectively [Figure 3 (c, d)]. Similarly, as-cast grain size measured to Alloy-1 and Alloy-2 as 50.3 μm and 41.2 μm respectively. The EPMA spot analysis reveals grain boundary segregation of solute atoms in the as-cast Alloy-2 and Alloy-4 [Figure 3 (e, f)]. The common feature of Al-Zn-Mg alloys is high volume fraction of alloying elements, which leads to severe dendrite and grain boundary segregation in the grain boundary regions. The EPMA analysis indicated the high solute content in grain boundary regions in as-cast Alloy-2 and Alloy-4 with EDS analysis. The EPMA analysis (Model no. CAMECA SX100) has been shown in as-cast condition for Alloy-2 (in average 10 points analysis recorded: Zn-2.55 wt.%, Mg-1.95 wt.%, Si-0.03 wt.%, Fe-0.05 wt.% rest Al) and for Alloy-4 (in average 10 points analysis recorded: Zn-11.88 wt.%, Mg-2.12 wt.%, Sc-19.17 wt.%, Si-0.02 wt.%, Fe-0.15 wt.%, rest Al). After solution treatment, the alloys [Figure 4(a-d)] exhibits obvious equiaxed recrystallized microstructure, homogenization of solute concentration and eliminated eutectic phases in the grain boundaries region. The purpose of solution treatment (so called \( T_4 \) condition) is promote the coarse equilibrium phases dissolved in order to form supersaturated solid solution. Due to decrease of solute supersaturation of the matrix, it would reduce the capabilities of precipitation hardening and lower strength
of the aluminium alloys [30]. The ageing kinetics has been characterized by Vicker’s hardness measurement and activation energy. The activation energies for solute diffusion obtained from the evolution of the Vicker’s microhardness. The activation energy ($E_a$) has been calculated by Arrhenius equation by plotting $\ln(\Delta H/V)\) versus $1/T$, the slope of the linear regression fitting indicate activation energy for Alloy-1 and Alloy-4. The values of activation energies of studied alloys has been found out 9.79 kJ/mol (at 10 h ageing time) and 23.73 kJ/mol (at 10 h ageing time), respectively. The different values of the activation energies indicate that the diffusing species are not same for all alloys [32]. Similarly, the DSC analysis implies precipitation and dissolution of metastable and stable phases in present alloys at heating rate 10°C/min in nitrogen atmosphere. The detailed analysis of DSC data indicates that compositional variations of studied alloying elements have significantly effects the on the formation and dissolution of GP zones, $\eta$, and $\eta$ T phases [33]. The purpose of solution treatment (i.e., $T_4$ condition) is dissolution of solute elements that will later stage cause of age hardening, spherodization of undissolved constituents and homogenisation of solute concentrations in the materials. Subsequently, quenching is used to retain solute elements in a supersaturated solid solution (SSS) and also create a supersaturated of vacancies that enhance the diffusion and the dispersion of precipitates. Therefore, the size distribution, average size, number density and volume fraction of the Al$_3$Sc particles were determined as a function of the solution treatment temperature and time. Starting from the grain size of the cast aluminium alloys, additions of Sc also increase the resistance to recrystallization during hot working and introduce additional strengthening through the formation of the fine coherent Al$_3$Sc particles from the solid solution. Because the low equilibrium solubility of Sc in the aluminium matrix, these particles precipitates from a supersaturated solid solution during first heat treatment after casting and cannot be dissolved after that, while the strengthening effect depends on their size and number density. Therefore, it is necessary to control precipitation of the Al$_3$Sc particles, in order to achieve the best balance of mechanical properties. In Figure 5 as solutionizing curve shows Alloy-4 (Sc content 0.83%) best hardness profile through the solution heat treatment. Because high Sc content (hypereutectic content, >0.55 Sc, shown in Figure 2) implies greater anti-recrystallization effect and coherent Al$_3$Sc particles main cause of high hardness profile among the present alloys. Similarly, Alloy-1 shows second highest hardness due to higher Zn+Mg contents to effect of solid solution hardening in this heat-treatment phenomenon. The Alloy-2 and Alloy-3 shows moderate hardness profile in this heat treatment phenomena. The artificial ageing has been carried out to all four present alloys to following temperatures at 120°C, 140°C, and 180°C respectively [Figure 6, (a-c)]. During artificial ageing treatment is to make strengthening phases precipitate from the supersaturated solid solution. The precipitation sequence of Al-Zn-Mg alloy is $\alpha_{ssn}$ (supersaturated solid solution) $\rightarrow$ GP zones $\rightarrow$ metastable $\eta$ $\rightarrow$ stable $\eta$. In these three types of precipitates, GP zones and $\eta$ metastable phase have strengthening effect on alloy. In the initial stages of ageing treatment, only GP zones and a few $\eta$ phases precipitates inside the grains. The strengthening is associated with the shearing GP zones and $\eta$ phases by dislocation during deformation. With the longer ageing time, the quantity of GP zones increases, and the strength of alloy increases gradually. In the initial stages of ageing treatment, only GP zones and a few $\eta$ phases precipitates inside the grains. The strengthening is associated with the shearing GP zones and $\eta$ phases by dislocation during deformation. With the longer ageing time, the quantity of GP zones increases, and the strength of alloy increases gradually. When the GP zones transform to metastable $\eta$ precipitates, the strengthening effect increase with the increase of volume fraction and size. As continuing to extend ageing time, $\eta$ phase inside the grains are coarsened gradually, and a part of $\eta$ phases transform to $\eta$ phases. This $\eta$ phase is incoherent with the matrix and lighter strength to called over-ageing stage. Based on the results and discussion, it can be concluded that strengthening effect of the alloy is associated with the fine strengthening caused by minor additions of Sc, the subgrain strengthening and precipitation strengthening of Al$_3$Sc particles and $\eta$ precipitates.

In Figure 6(a) shows Alloy-4 at 120°C ageing phenomena highest hardening effect due to GP zones formation and high density, fine dispersion of Al$_3$Sc precipitates. Similarly, Alloy-1 shows second highest hardening effect due to high solute contents. And Alloy-2, Alloy-3 shows moderate hardening effect at two hours ageing time. In Figure 6(b) shows Alloy-1 at 140°C ageing phenomena highest hardening effect due to high solute contents to formation of GP-II zones (form above 120°C) and transform to $\eta$ phases. Others three alloys in this regime shows lowest ageing response due to transform of $\eta$ phases, which is noncoherent with the matrix and resistance effect to dislocation movement is decreased, so strength of alloys decreases. In Figure 6(c) shows Alloy-1 at 180°C ageing phenomena highest hardening effect high solute contents and $\eta$ phases transformation. Others three alloys indicate less ageing response in this regime. This suggests that a phase transformation to $\eta$ occurs during this period in the overaged stage. The $\eta$ precipitates are incoherent in the matrix. Hence, the longer interparticle spacing and the coarse size of the $\eta$ precipitates in the grain result in more reduction in strength. Similarly, FESEM with EDS analysis of as-cast Alloy-2 and Alloy-3 exhibits grain boundary segregation of impurity elements [Figure 7 (a-b)]. In EDS analysis has been shown in both alloys in as-cast condition grain boundary segregation of solute elements of impurities Fe surplus in Alloy-2 and Sc existence in Alloy-3. In Alloy-2 high Fe content, effects lower strength and ductility in as-cast condition which has been shown in Table 2. In TEM analysis revealed in as-cast alloys in Figure 8 (a, b), for Alloy-2 dark and white spots indicated GP zones formation and for Alloy-3 precipitates and dislocation interaction observe in present TEM micrograph [33]. In Figure 9 illustrates friction stir processing set-up to cast 7xxx series of aluminium alloys under different heat treatment conditions were subjected to double-passes friction stir processing (FSP). Friction-stir processing has been developed as an effective grain refinement technique based on the principle of friction stir welding. It is well documented that the intense plastic
deformation and temperature rise during FSP result in the generation of dynamic recrystallization, producing fine and equiaxed grains in the stirred zone. Essentially, FSP is a local thermo-mechanical metal working process that changes the local properties without influencing properties in the remainder of the structure. During FSP, a specially designed cylindrical tool is plunged into the plate causing intense plastic deformation through stirring action, yielding a defect free, and dynamically recrystallized, fine grain microstructure. Since solid-state friction stir processing does not result in solute loss by evaporation and segregation by solidification, solute elements are homogeneously distributed in the processing zone [34].

Thus, FSP creates a fine-grained microstructure with dispersively distributed particles and predetermining high-angle grain boundaries, features that are important for enhanced superplastic properties [35]. The tensile specimens are selected from the stir zone as per tensile specification (gauge length 26 mm, thickness 2.5 mm, width 4 mm and total length 58 mm) and tensile test results as different heat treated conditions are tabulated in Table 2. In Figure 10(a-d) shows friction stir processed Alloy-2 in optical micrographs from different regions identified grain refinement as comparison to unprocessed zones.

Figure 3. All optical micrographs are as-cast condition: (a) Alloy-1, (b) Alloy-2, (c) Alloy-3, (d) Alloy-4; EPMA micrographs are as-cast condition: (e) Alloy-2, (f) Alloy-4
Figure 4. All optical micrographs are T4 condition: (a) Alloy-1, (b) Alloy-2, (c) Alloy-3, (d) Alloy-4

Figure 5. Effect of solutionizing [465°C/1h then immediately water quenched (WQ)] curves on Vicker’s hardness of present alloys
Table 1. Chemical composition of prepared aluminium alloys (in wt. %)

<table>
<thead>
<tr>
<th>Alloy nos.</th>
<th>Zn (wt%)</th>
<th>Mg (wt%)</th>
<th>Sc (wt%)</th>
<th>Si (wt%)</th>
<th>Fe (wt%)</th>
<th>Al (wt%)</th>
<th>Zn+Mg</th>
<th>Zn/Mg ratio</th>
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<td>Alloy-1</td>
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<td>0.04</td>
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<tr>
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<td>6.40</td>
<td>2.50</td>
<td>-</td>
<td>0.05</td>
<td>0.10</td>
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<td>8.90</td>
<td>2.56</td>
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<tr>
<td>Alloy-3</td>
<td>6.70</td>
<td>2.80</td>
<td>0.20</td>
<td>0.02</td>
<td>0.04</td>
<td>Balance</td>
<td>9.50</td>
<td>2.40</td>
</tr>
<tr>
<td>Alloy-4</td>
<td>7.10</td>
<td>3.20</td>
<td>0.83</td>
<td>0.01</td>
<td>0.03</td>
<td>Balance</td>
<td>10.30</td>
<td>2.22</td>
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Table 2. Results of tensile properties (FSP proceed by double passes and full processed zone)

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<th>Alloy no.</th>
<th>As-cast condition</th>
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<th>Sol. + FSP</th>
<th>Sol. + FSP + Ageing at 140°C for 2 h</th>
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<tr>
<td></td>
<td>σ₀.₂ (MPa)</td>
<td>σ₀ (MPa)</td>
<td>δ (%)</td>
<td>σ₀.₂ (MPa)</td>
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<td>49.1</td>
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<td>Alloy-4</td>
<td>89.9</td>
<td>179.8</td>
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<td>115.2</td>
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</table>

*N.B: Solutionized at 465°C/1 h then WQ and yield strength denoted at 0.2% offset from stress-strain curve.

Figure 6. Variation in the Vicker’s hardness with ageing time t for the alloys aged at: (a) 120°C, (b) 140°C, and (c) 180°C
Figure 7. FESEM micrographs and EDS analysis of as-cast condition: (a) Alloy-2; (b) Alloy-3

Figure 8. TEM analysis in as-cast condition of: (a) Alloy-2; (b) Alloy-3
Figure 9. The schematic diagram of friction stir process (FSP) set-up

Figure 10. Optical micrographs of Alloy-2 (solutionized+FSPed): (a) parent metal, (b) process and unprocessed zone (c) nugget zone, (d) recrystallized zone. (1000 rpm and 70 mm/min)
In nugget zone (in Figure 10(c)) shows very fine grain due to intense plastic deformation and temperature rise during FSP result in the generation of dynamic recrystallization, producing fine and equiaxed grains in this zone. Similarly, in Figure 11 (a-d) shows friction stir processed Alloy-4 in optical micrographs from different regions identified grain refinement as comparison to unprocessed zones. In nugget zone (in Figure 11(c)) shows fine grain refinement (100-200 nm size) due to frictional heat and dynamic recrystallization and Al₃Sc dispersoids balance effects. Since Alloy-4 has very high Sc content (0.83%), so dispersive effect accelerated the increases strength. The tensile test has been performed as different heat treatment conditions as results indicate after FSPed 0.2% proof strength and ultimate tensile strength are increased gradually but specimens with solution heat treated and friction stir processed more enhanced strength and ductility in all four alloys. Due to homogenisation supersaturated solid solution and Al₃Sc dispersoids (in cases of Sc added alloys) are more pronounce to fine grain refinement during precipitates dispersion of friction stir regions. Similarly, specimens next step to ageing at 140°C for 2 h shows ultimate tensile stress drop slightly for Alloy-1 and Alloy-2 but ductility increased marginally due to η phase pronounced to formation in 2 h ageing time. However, in Alloy-3 (0.2%Sc content) shows better strength and ductility due to Al₃Sc dispersoids and during stirring intense heat generation to anti-recrystallization effects to obtained balance properties. For Alloy-4 (0.83%Sc content) shows maximum strength and ductility due to formation of high density of Al₃Sc particles and MgZn₂ (7.10%Zn content) precipitates enhanced tensile properties and elimination of porosity of cast structure after FSPed [in Table 2].

4. Conclusions

1. The 7xxx series of alloys are heat-treatable Al-Zn-Mg alloy. Most ternary alloys are age hardenable.
They develop their strength by solution treatment followed by ageing.

2. To addition Sc in Al-Zn-Mg is strong grain-refiner. Eliminated grain boundary segregation, dendritic structure and retarding recrystallization in the as-cast aluminium alloy.

3. In addition Sc to Al-Zn-Mg alloy accelerated ageing kinetics and concentrated GP zones formation.

4. The addition of Sc improves the mechanical properties of the alloys. The reason is the precipitation of secondary Al₃Sc particles and the remarkable refinement of the grains, namely, precipitation strengthening and fine-grain strengthening.

5. The higher ductility is due to the elimination of porosity and the breakup of coarse second phase particles. The tensile strength after FSP has been improved due to grain refinement and homogenisation of precipitates particles of present aluminium alloys.

6. However, the frictional heat and severe plastic deformation and the residual stresses are prime mechanisms to enhanced strength of present aluminium alloys.

7. In the as-cast state the strength of the supersaturated solid solution increases with increasing Zn and Mg content due to solid solution hardening. However, after FSP the work hardening and Hall-Patch hardening due to the decreasing grain size competes with softening due to the decomposition of a supersaturated solid solution. In the net effect, the FSP results in a softening of aluminium alloys.

8. Dispersoids like Al₃Sc are effective means of inhibiting the grain growth during FSP.

References


