

Fine-Grained Cast Aluminium Alloy Produced In Situ By Friction Stir Processing

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Abstract:- Friction stir processing (FSP) is an emerging technique based on the principles of friction stir welding (FSW). Fine-grained Al-Zn-Mg alloy can be fabricated in situ by friction stir processing. Therefore, FSP has been shown to be a feasible technique to produce large-area thin plate of fine-grained 7075 aluminium alloy by running double passes. Thus, friction stirring produces very fine grain microstructure in the stirred region. During FSP alloy possesses enhanced proof strength, ultimate tensile strength and ductility. Therefore, it is possible to further refine and homogenize the cast structure and to produce large volume of materials by applying FSP technique.

Keywords:- fine-grain, 7075 Al-alloy, yield strength, FSP technique.

I. INTRODUCTION

The Al-Zn-Mg alloys have versatile characteristic to phase transition during ageing treatment to make them industrially applicable. One of the strongest in Al-alloys is alloy 7075 series, subjected to solutionizing and subsequent heat treatment. It has a high strength to specific weight ratio, and it is ideal for use in highly stressed components [1-3]. In this phenomenon, friction stir processing (FSP) is an emerging surface-engineering technology, which uses the principles of friction stir welding (FSW) to process materials in a variety of ways besides joining them [4-6]. Mishra et al. developed FSP as a generic tool for microstructural modification based on the basic principles of friction stir welding [7-8]. FSP can locally eliminate casting defects and refine microstructures, thereby improving strength, ductility, resistance to corrosion, formability and other properties [9-10]. FSP is carried out by rotating and plunging a specially designed cylindrical, shouldered tool with a small diameter pin into the plate that is clamped firmly to the bed. Frictional heat causes the metal to soften and allows the tool to traverse along the plate. The FSP generates three distinct microstructural zones: the nugget or stir zone, the thermo mechanically affected zone (TMAZ) and the heat-affected zone (HAZ). The nugget zone, in which occurs dynamic recrystallisation due to severe thermo-mechanical processing resulting in a homogeneous fine equiaxed recrystallized grain structure. The TMAZ adjacent to the nugget is the region where the metal is plastically deformed as well as heated to a temperature, which is not sufficient to cause recrystallisation. The HAZ experiences only heating effect, with no mechanical deformation [11, 4]. Therefore, FSP has been studied not as a modification technique but as a new grain refinement process. Grain refinement is an effective means to improve the mechanical properties, e.g. yield strength, of friction stir processed material. However, in age-hardenable aluminium alloys, precipitation strengthening is the dominant mechanism. During FSW/FSP, coarsening, dissolution of strengthening precipitates, as well as reprecipitation of metastable and/or equilibrium phases are expected [12]. Since, the process eliminates surface microstructural defects, e.g., porosities and cracks, and refines and homogenises the grain size, the processed components become more tolerant to mechanical loading [13, 6]. Sato et al., on the other hand, have proposed that the fine grains arise from recrystallisation within the stir zone [14]. Gil Sevillano et al. showed that rate of generation of grain boundary area per unit volume was maximum for the material deformed under compression than those deformed either by rolling, wire drawing or torsion for the same level of equivalent strain [15]. There are several mechanisms also discussed to strengthening effects depend on their precipitates size, spacing and distribution [16]. In the present experimental works has been evaluated reduction of grain sizes from base (no FSP) to processing materials as different stages by in-situ FSP [17]. Therefore, our assessment has been focused basically reduction of grain sizes through FSP by double passes only. Also, several techniques have been adopted to reveal grain refine mechanisms, which are SEM, TEM, and mechanical testing. The goal of the present task is to analyze the strengthening due to precipitation and grain refinement in age-hardenable 7075 Al alloy.

II. EXPERIMENTAL PROCEDURE

Aluminium alloy 7075 of the Al-Zn-Mg system has been studied with the following composition (wt.%): 6.4 Zn; 2.5 Mg; 0.05 Si; 0.10 Fe; balance Al. The dimensions of the plate shape casting are $150 \times 90 \times 8 \text{ mm}^3$. The cast plates were subjected to solutionizing temperature at $465 \text{ }^\circ\text{C}$ for 1 hour then water quenching. This heating process also called T_4 heat-treatment or solutionizing treatment for Al-alloys. An indigenously designed and developed milling machine was used for FSP. A specially designed and developed fixture was used to hold the plate firmly in position during FSP. A mild steel backing plate was used in this process. During FSP, the traveling direction (x-direction) was parallel to the processing direction of the Al-plates. The traverse sides (y-direction) of the plates were clamped using pressing plates to constrain the displacement of the plate during FSP. At axial force (z-direction) 15 KN load was maintained during processing [in Fig.-1]. There are two distinct sides has been classified, namely, retreating side and advancing side. The three different plates were subjected to separate conditions as constant parameters during FSP with double passes as results shown in Table-1. The processing parameters include: 75 mm/min traveling speed, 1025 rpm clockwise rotating speed, and 15 MPa axial pressure, using a tool with 25 mm shoulder diameter and 6 mm pin root diameter with sharp tip with 3.5 mm pin depth for full penetration into the plates. There are procedural phenomena, several stages (from I to V in Table-1) we have characterized tensile properties with standard tensile test samples. The sample dimensions are gauge length 26 mm, gauge width 4 mm, thickness 2.5 mm, with 58 mm full length. Generally, tensile samples was selected through the FSP region and tested using a 25 KN, electromechanically controlled Universal Testing Machine (H25 K-S; Hounsfield Test Equipment, Ltd., Surrey, UK) with 1 mm/min cross head speed at room temperature. 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 $\text{HNO}_3(70\%) + 1.5 \text{ ml HCl}(38\%) + 1 \text{ ml HF}(40\%) + 175 \text{ ml water}$). In Fig.-2 have been displayed macro-images of FSP plates by double passes. TEM analysis has been carried out to study the precipitation morphology, size, and orientation using at Techai $G^2 20 \text{ S-TWIN}$ at 200 kV.

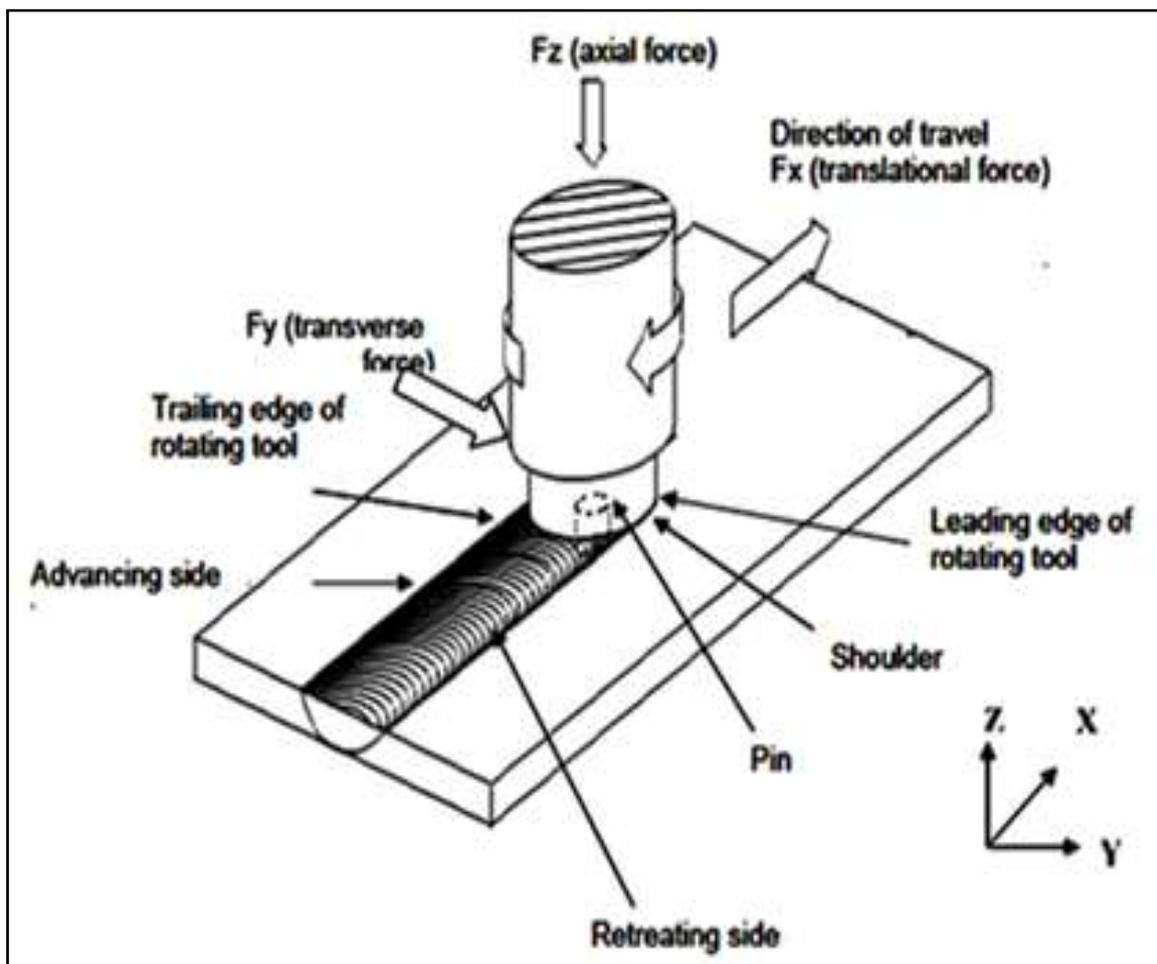


Fig.-1: Schematic set-up of friction stir processing and terminology.

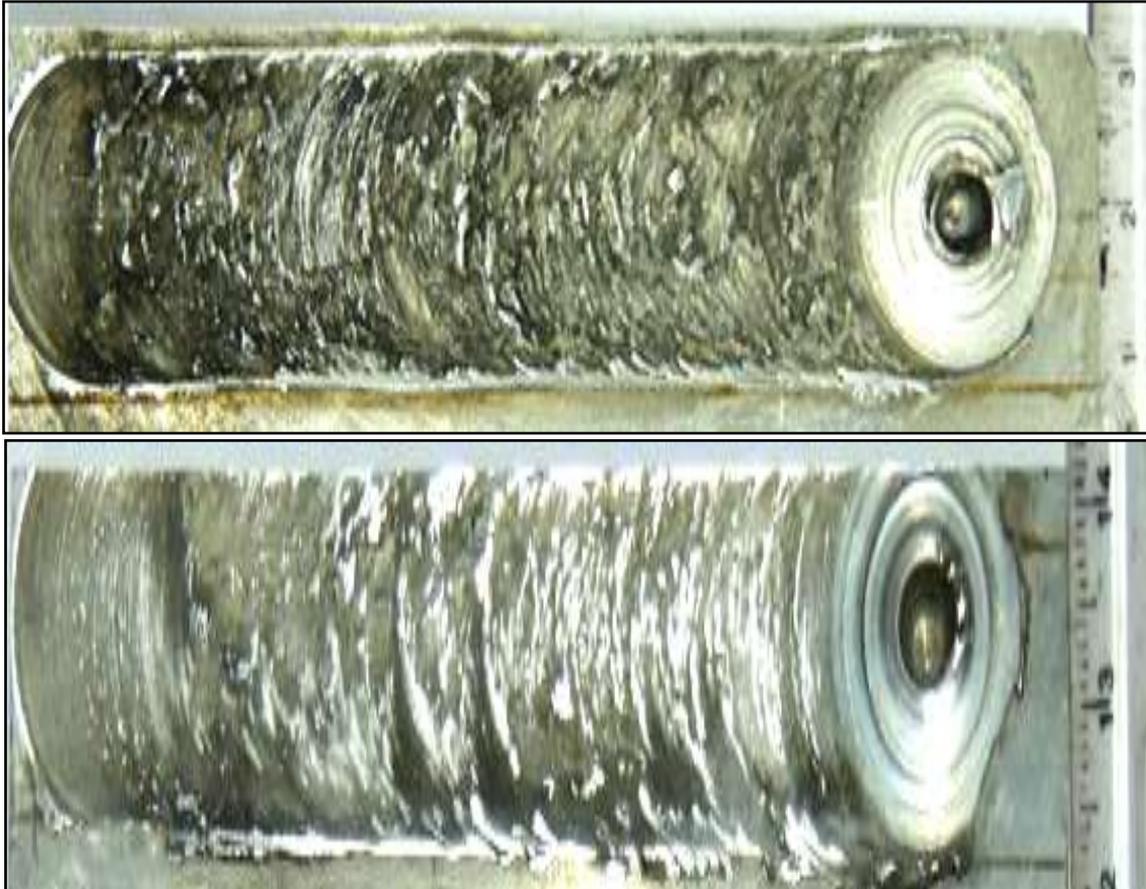


Fig.-2: Macro-images FSP of Al plates performed by double passes (1025 rpm, 75 mm/min).

III. RESULTS AND DISCUSSION

This experimental alloy principally utilized precipitation hardening to attain high strength. Precipitation in the Al-Zn-Mg alloy system has been well documented and can be summarized as: (Supersaturated solid solution) → Guinier-Preston (GP) zones → η' (MgZn₂) → η (MgZn₂), while η' is monoclinic and semicoherent, η is hexagonal and incoherent [18,6]. Al-Zn-Mg alloys are strengthening by GP zones and η' precipitates in the T₆ (ageing treatment for Al alloys) condition when aged below their GP zone solvus [19]. The GP zones solvus is approximately 170 °C for the present alloy composition which is above the ageing temperature (140±2) °C in present alloy. While, friction stir welding (FSW) is a solid state of joining technology. During the welding process temperature cannot reach the melting point, so there are not some porosities and shrinkage cracks inside the materials. Friction stir processing (FSP), using same methodology as FSW, produces the fine recrystallized grains in the stir zone due to dynamic recrystallization from severe plastic deformation, and further affects the mechanical properties [20, 12]. Recently, much attention has been paid to FSP that is known as a surface modification technique. This has led to several applications for microstructural modification in metallic materials, including superplasticity, surface composite, homogenization of nanophase aluminum alloys and metal matrix composites, and microstructural refinement of cast aluminum alloys. There are several strengthening mechanisms can be summarized during processing as well as precipitation hardening nature in studies alloy is present discussing matter as follows [21, 22, 16].

The strengthening also depends upon the concentration of solute (differences in size, modulus and valance) and solute solution which is typically represented as, $\Delta\tau_{ss} = AC_o^{2/3}$ (1)
Where $\Delta\tau_{ss}$ is increase in yield stress (0.2% proof stress) due to solid solution strengthening, A is constant and C_o is concentration of the solute atom in weight percent.

The contribution of yield strength of fine grain strengthening can be expressed by the Hall-Patch relationship: $\sigma_y = \sigma_o + \frac{k}{\sqrt{d}}$(2)

Where σ_y is the increase in yield strength due to fine grain strengthening, σ_o is the friction stress, k is a constant and is the grain size. The precipitation strengthening in the base metal is due to the presence of plate-shaped metastable precipitates. The strengthening effect is described by: $\Delta\sigma = \frac{0.85Gb}{2\pi\sqrt{(1-\nu)}} \frac{C}{D(1-\frac{\pi Ct}{2D})} \ln(\frac{2D}{\pi r_o})$(3)

Where $\Delta\sigma$ is the increase in yield strength due to precipitation strengthening ν is the poisson's ratio (0.3 for Al) and G is the shear modulus (26 GPa for Al).

$$C = \sqrt{(fA) + (2/\pi - \pi/2A) fA} \dots\dots\dots(4)$$

Where f is the volume fraction of precipitates, D is the diameter of the precipitates, t is the thickness of the precipitates and r_o is the dislocation core radius (6×10^{-10} m).

$$\text{The increase in strength due to work hardening is represented as, } \Delta\tau_D = \frac{BGb}{L} \dots\dots\dots(5)$$

where $\Delta\tau_D$ is increase in strength due to dislocation strengthening, B the constant= 0.2 for FCC material, G is the shear modulus = 26 GPa, b is the burgers vector = 2.84 Å for Al, and L is the interparticle spacing. The stacking fault energies of the matrix and precipitate differ and the motion of dislocation are impeded because the separation of the partial dislocations depends on the phase in which the dislocations reside. The increase in the strength of Al-Zn-Mg alloys can be attributed to stacking fault strengthening and can be approximated by the following expression for strong particles: $\Delta\tau_{sf} = (\frac{Kf}{\tau_{sf}})^{1/2} (\frac{\Delta\gamma}{b}) (\tau_p)^{-1/2} \dots\dots\dots(6)$

$\Delta\tau_{sf}$ is contribution to yield strength due to stacking fault strengthening, $\gamma_{sf} = \frac{\gamma_{sfm} - \gamma_{sfp}}{2}$ and γ_{sfm} is the stacking fault energy; γ_{sfp} is the stacking fault energy of η precipitates. There exists a difference in the elastic moduli between that of the matrix and the η precipitate. The modulus strengthening can be expressed as:

$$0.9(r_p f)^{1/2} (\frac{r}{b}) (\frac{\Delta G}{G}) (2b) \ln(\frac{2r_p}{f^{0.5} b}) \dots\dots\dots(7)$$

The total strength expected due to the combination of these various contributing mechanisms can be expressed as,

$$\sigma_{tot} = \sigma_{gb} + M\{(\Delta\tau_{ss}^2 + \Delta\tau_D^2 + \Delta\tau_{r ods}^2 + \Delta\tau_{sf}^2 + \Delta\tau_{mod}^2)^{1/2}\} \dots\dots\dots(8)$$

Where σ_{tot} the total yield strength of the matrix and M is Taylor factor ~3. In the several literature has to pointed out, the thermo-mechanical treatment (combination deformation and heat treatment) increases the yield stress and ultimate tensile stress of 7075 Al alloy. The briefly high strength of the fine grain 7075 Al alloy may be attributed to: i)-solid solution strengthening; ii)-grain refinement strengthening; iii)-dislocation strengthening and iv) precipitation strengthening. The precipitation strengthening results from the precipitates ability to impede dislocation motion by forcing dislocations to either cut through or circumvents the fine precipitates [23]. It is also noteworthy that, the formation of a fine grain structure suggests a very high nucleation rate during dynamic recrystallization. In other words, the deformed microstructure creates a high density of local components with highly stored energy of deformation, which can act as nucleation sites for new grains. It is believed that a complex stress state and strain components with very large-strain gradients were produced in the processed material around the pin tool during FSP. Furthermore, large amount of dislocations were introduced to accommodate the strain incompatibility. The complex state of stress and strain, dislocation configurations and high density of geometrically necessary dislocations are all beneficial in promoting copious nucleation during dynamic recrystallisation, and eventually contribute to the small grain structure [24].

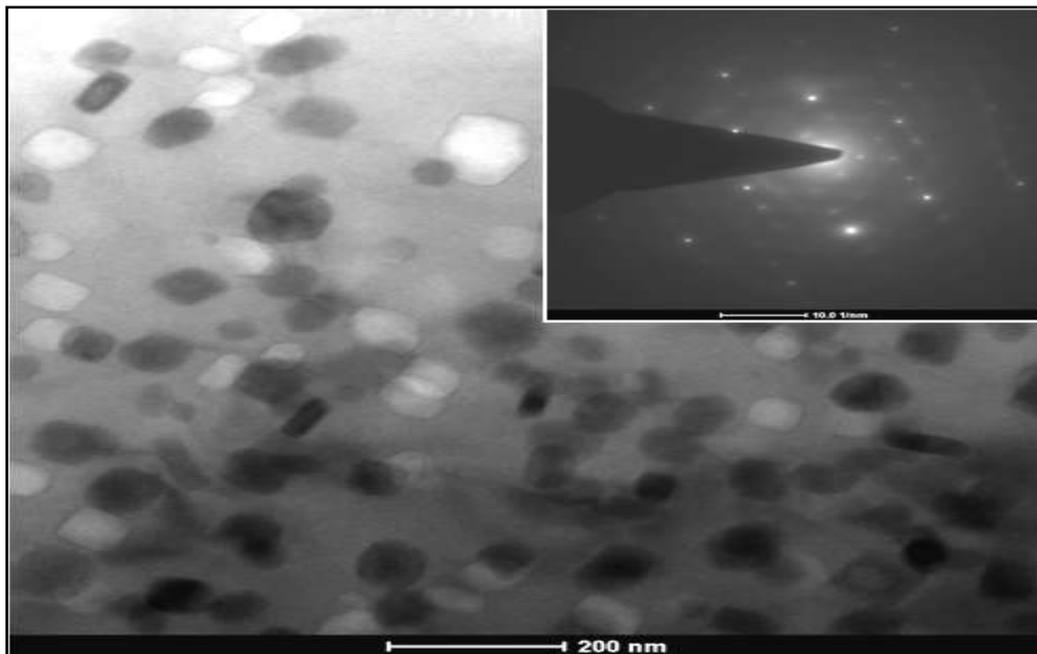


Fig.-3: TEM micrograph with inside diffraction pattern of studied in as-cast alloy.

TEM (in Fig.-3) observations in the as-cast alloy indicate the presence of GP zones formation in matrix with dark-field image in selected area diffraction spotted fine precipitates but not uniform throughout in matrix. In Fig.-4(a-c) depicts three types of optical micrographs, in (a) no FSP, clearly visible cast grain inhomogenities with grain boundary segregation. In (b), after solutionized plus FSP, reveal the tensile characterization has been shown optimum properties due to supersaturation with age-hardening factors, and dynamic recrystallisation to fine grains effect are dominant. In (c), here has been shown three different colour arrows (i) red arrow- for stir zone or nugget zone grain size, 2- 5 μm ; (ii) black arrow- for TMAZ grain size, 7-10 μm ; (iii) blue arrow- for HAZ grain size, 10 -15 μm . whereas, in as-cast or no FSP grain size has 25-30 μm . In Table-1 (Stage-V), low strength and ductility attributed to the loss of metastable age-hardening precipitates (η') and the formation equilibrium (η) precipitates during FSP, which reduced precipitation-hardening contribution [25, 12].

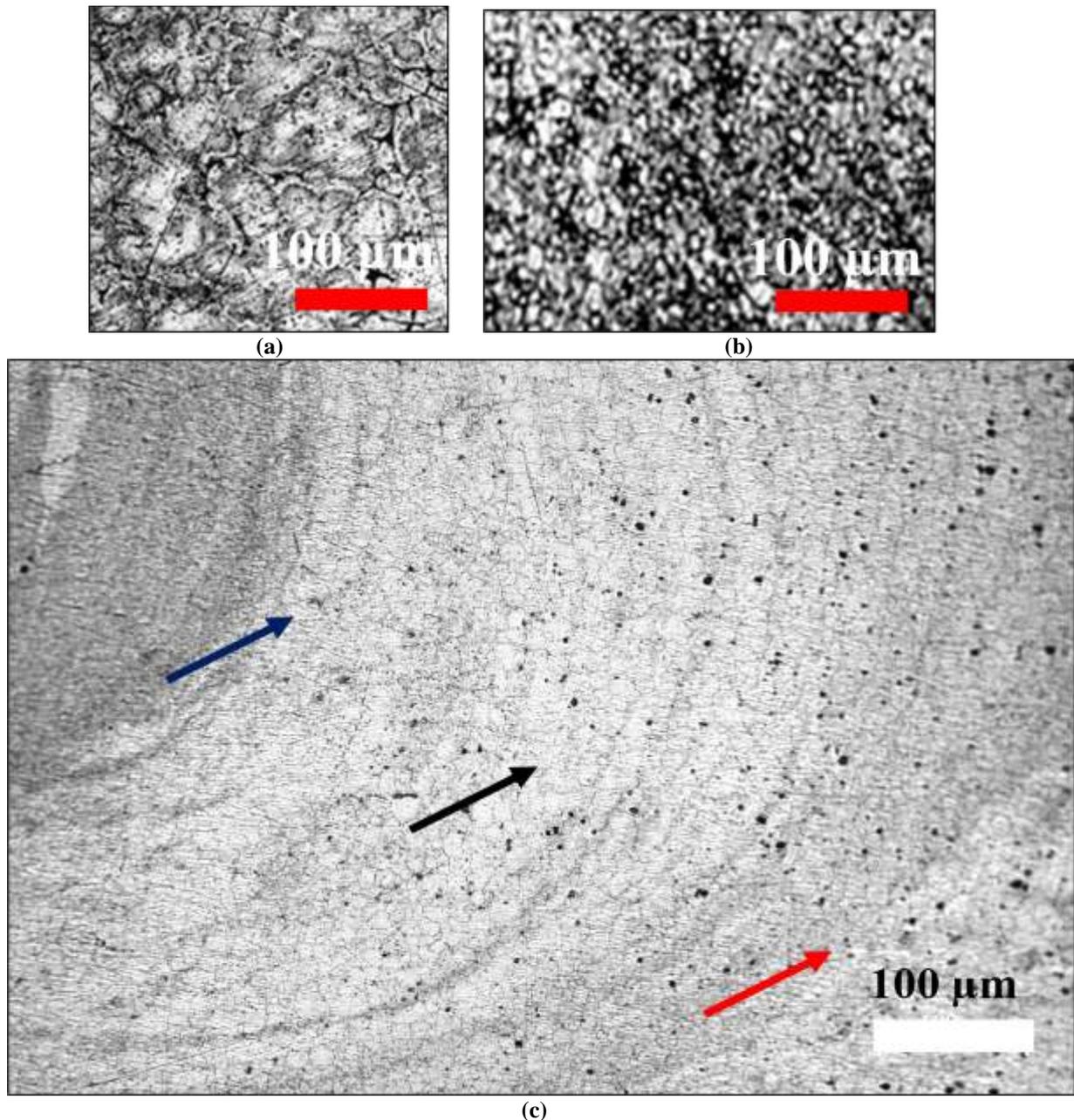


Fig.-4: Illustration of optical micrographs during FSP (a) without FSP; (b) T_4 + FSP; (c) T_4 + FSP + aged at 140 °C/2h (different arrows indicated different processing zones).

The fracture surface of the tensile specimen revealed sharp cutting edges in individual grains, which indicated after post ageing treatment became brittleness or overaged. Due to overaged condition materials became incoherent equilibrium (η) phases to drop ductility and strength (in Table-1, stage-V). Furthermore,

overageing materials resulted in particle coarsening, particularly at grain boundaries. Because, at FSP with post-heating specimen often exposed faster precipitates nucleation (i.e., depends nature of material, time, and temperature) with facilities of high angle grain boundaries are main factors.

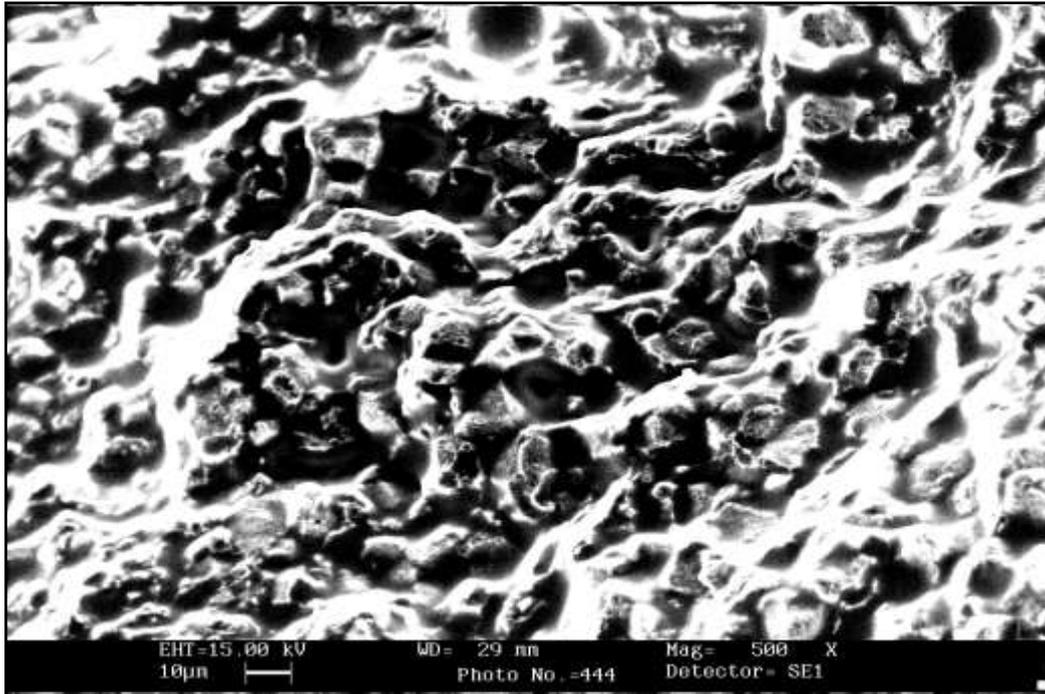


Fig.-5: SEM analysis of tensile fractograph after (T₄+FSP+Aged at 140 °C /2h) processing.

Table-1: Results of tensile properties are tabulated through FSP direction of studies alloy compared with the unprocessed materials.

FSP by double passes at different conditions		Tensile properties		
		$\sigma_{0.2}$ (MPa)	σ_u (MPa)	δ (%)
I	AC	66.3	106.2	2.2
II	AC + FSP	200.6	236.2	6.8
III	T ₄ condition	49.3	87.3	2.6
IV	T ₄ + FSP	165.0	275.4	9.0
V	T ₄ + FSP + Aged at 140 °C /2h	130.8	211.0	5.5

Note: (I)AC = as-cast; (III)T₄ = Solutionized at 465 °C/1 h then WQ; yield strength denoted at 0.2% offset from stress-strain curve.

IV. CONCLUSIONS

1. The Al-Zn-Mg alloy is an age-hardenable as well as high strength 7075 Al alloy.
2. The FSP is the effective surface modification technique to produce fine-grained by double passes only.
3. Grain size in the FSP specimen is about 15 times less than that of the as-cast one. This fine equiaxed microstructure, according to the Hall-Patch relationship, results in a higher strength. Also, refined microstructure resulted from FSP leads to improvement of crack growth resistance in the matrix during the tensile test, and therefore increases the elongation.
4. Fine equiaxed grains in the stir zone implies that dynamic recrystallization has taken place during FSP due to plastic deformation.
5. The optical microstructure features indicate that during FSP, very small grains were formed around the pin tool by dynamic recrystallization at elevated temperatures. The grain boundaries evolved by absorption of numerous dislocations provided by the surrounding structure, and additional dislocations were generated by subsequent plastic deformation in the larger grains, which accommodate strains preferentially.
6. The different processing zones exhibited (Table-1 at Stage-V) a non-uniform grain sizes. It is also noted that nugget region grain sizes from 2-5 μm , TMAZ region grain sizes from 7-10 μm , and HAZ region grain sizes from 10-15 μm .

7. Thus, FSP has potential of grain refining as small as 2-5 μm in stir zone.
8. In addition, during strengthening mechanisms with FSP and post-ageing (140 $^{\circ}\text{C}/2$ h) did not improve the strength. This result indicates that the matrix lost the supersaturation after ageing treatment.
9. In stage-V (Table-1), low strength and ductility attributed to the loss of metastable age-hardening precipitates (η') and the formation equilibrium (η) precipitates during FSP, which reduced precipitation-hardening contribution.

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