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Investigation of Precipitation Behavior and Related Micro structural and Mechanical Properties after FSP of Aluminum Alloy

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ABSTRACT: *The precipitation behaviour and related age-hardening in scandium (Sc) inoculated aluminium alloy (7075 series) aged at 140°C was investigated in detail. The solution treated aluminium alloy was surface layer modified by a novel solid-state technique like DP-FSP then followed by post ageing treatment at 140°C for 2h.During DP-FSP, the work piece was subjected to severe plastic deformation (SPD) by the mechanical stirring action of the rotating tool with 1000 rpm, 70 mm/min traverse speed, and 15 kN axial force. The presence of Al_3Sc and $MgZn_2$ precipitates promotes continuous dynamic recrystallization (DRX) in matrix during FSP. The mechanism for this phenomenon was examined by using OM, FESEM, SEM, and TEM analysis. After processing, the mechanical properties were improved such as 0.2% PS of 104.4 MPa, UTS of 248.5 MPa, elongation of 6.7%, Vicker's hardness of 139.6HV, and strain hardening exponent (n) of 1.60 of the aluminium alloy.*

***Key words**: Sc inoculation, FSP, Al_3Sc precipitates, TEM, mechnaical properties.*

I. INTRODUCTION

The aluminium alloys (7075 series) possess spontaneous age-hardening ability at room temperature as well as at elevated temperatures. The strength of aluminium alloys are mainly due to age-hardening mechanisms. GP zones and ή are the main hardening phases during ageing treatment [1-3]. It is very common in addition of transition elements in this alloy to improve the mechnaical properties and better corrosion resistance preferably for automotive and aerospace applications [4]. Sc is a most precipitation hardening element in aluminium alloys. The minor Sc (0.2wt.%) addition resulted enough Al_3Sc particles dispersion that greatly refine grains, anti-recrystallization effect, and thermally stable [5-8]. Further, Al 7075 alloy has been surface modified by double-pass friction stir processing (DP-FSP), which can locally eliminate casting defects and refine microstructure, thereby improving strength and ductility.With the help of axial applied force, a frictional heat is induced and plastic deformation on the T_4 aluminium plate was modified [9,10]. The microstructure and mechanical properties on working zone can be controlled with the specified tool design and DP-FSP parameters (1000 rpm and traverse speed 70 mm/min). The purpose of this paper is to investigate the precipitation effect on microstructural and mechanical properties of the aluminium alloy after DP-FSP.

II. MATERIALS AND METHODS

Table 1: Nominal composition of experimental 7075 aluminium alloy (wt.%).

7075 Al alloy	Zn	Mg	Sc	Si	Fe	Al	Zn+Mg	Zn/Mg
	6.70	2.80	0.20	0.02	0.04	Bal.	9.50	2.39

Using pure Al, pure Zn (99.99%), pure Mg (99.99%), and Al-2%Sc master alloy as raw materials, and a plate shape casting (size: 200×90×24 mm^3) of mild steel mould was used during melting. The melting temperature was fixed at 780^{o}C in muffle furnace for 3 hours. The low melting point (419.5^{o}C) element like Zn and highly flammable element like Mg had been added carefully during melting, and maximum recovery of Sc. The chemical composition was analyzed by ICP-AES method. The cast plate was sectioned to 150×90×8 mm^3 size and preferred for solution treatment at 465^{o}C for 1h then immediately water quenched to room temperature (T_4). The optical microscopy (LEICA DMI 5000M) was carried out through standard metallographic methods and etched by Keller's reagent (1ml HF, 1.5ml HCl, 2.5ml HNO_3, 95ml H_2O). The TEM microstructure was revealed after electrolytically etching and imaging was carried out by Techai G^2 20S-TWIN at 200 kV. The FESEM microstructure with EDAX analysis was examined by QUANTA 200F, FEG LaB_6, 30kV. Consequently, T_4 plate had been subjected to DP-FSP with predetermined process parameters as shown in Figure 1 and Table 2. The processed plate was machined and sectioned, cleaned and subsequently aged at 140^{o}C for 2h at controlled temperature. The mechanical testing was performed using Instron Testing Machine (UTM, 25 kN, H25 K-S, UK) as per ASTM standard (E8/E8M-11) with 1 mm/min cross head speed at room temperature and results are tabulated in Table 2.

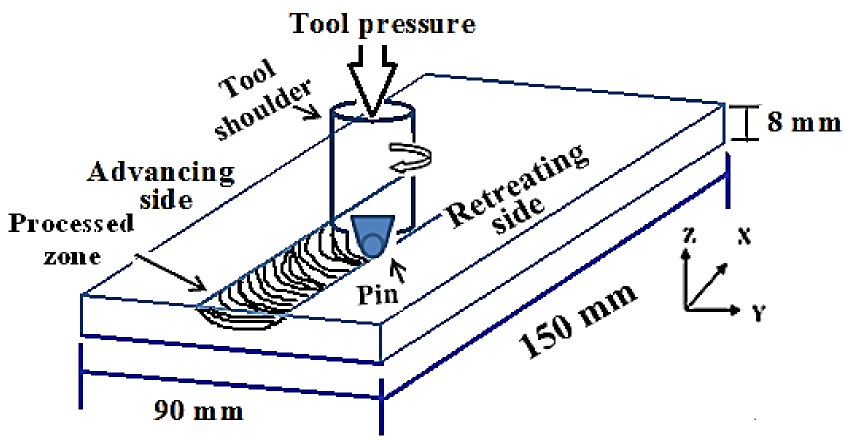
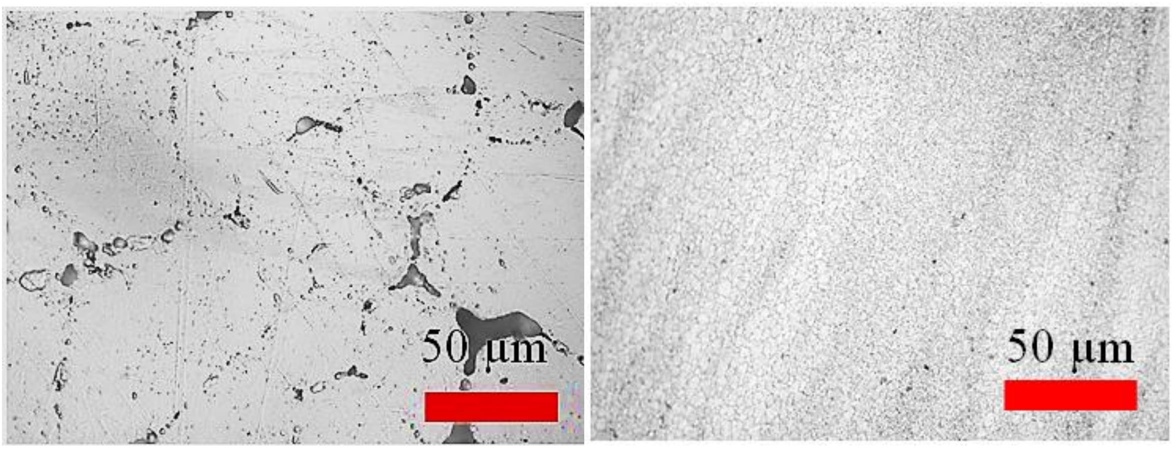


Figure 1: The schematic illustration of DP-FSP set-up.

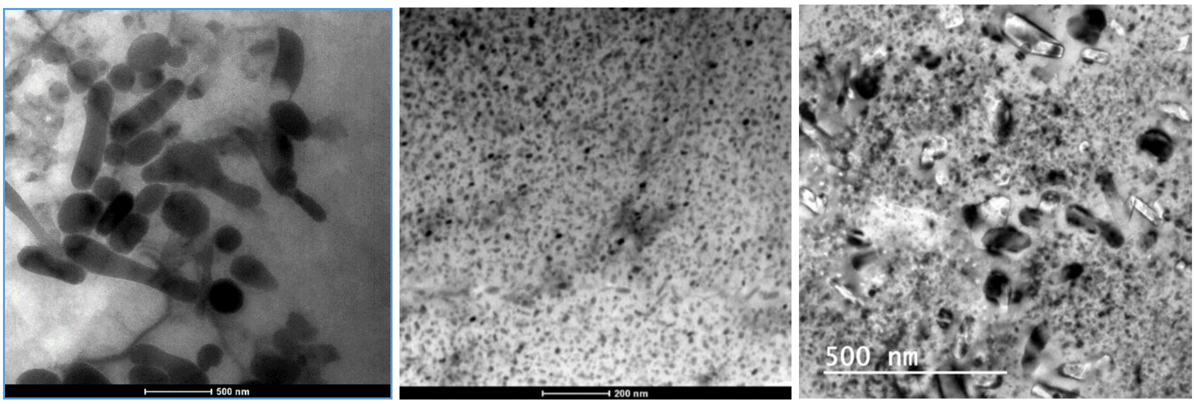
III. RESULTS AND DISCUSSION



(a) **(b)**

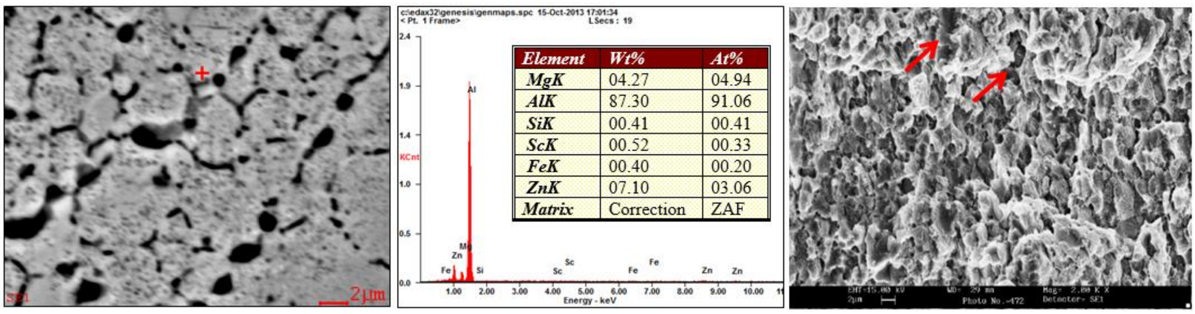
Figure 2: Optical microstructures of aluminium alloy: (a) T_4 condition, (b) SZ (T_4+DP-FSP+Aged at 140^{o}C for 2h condition)

Figure 2(a) shows optical microstructure of T_4 aluminium alloy exhibited homogeneous with some grain boundary segregations and relatively coarser grains (average grain size, 152.34±8.93 µm) achieved due to minor Sc (0.20%) addition in matrix. Figure 2(b) shows optical microstructure of SZ (stir zone) is characterized by the fine and recrystallized grains (4.85±1.94 µm) caused by the SPD and thermal exposure during DP-FSP [11, 12]. Figure 3(a) shows TEM microstructure of cast aluminium alloy exhibited inhomogeneous, coarse-grained dendritic structures, and unrefined structure (0.20%Sc) in the matrix. Figure 3(b) shows TEM microstructure of T_6 (aged at 140°C for 6h) aluminium alloy exhibited very fine and uniform precipitates throughout matrix, reasons for ageing treatment at 140°C and 6h with minor Sc addition. The alloy can be designated as having typically very fine precipitates microstructure due to GP zones, ή, and Al_3Sc particles are the most hardening phases. A high number of density, uniform distribution and size of precipitates are responsible for high hardness (172HV) of this alloy [13]. Figure 3(c) shows TEM microstructure of SZ exhibited uniform distribution of very fine precipitates along with some coarse precipitates of agglomeration of η plus Al_3Sc particles after DP-FSP plus post ageing treatment at 140°C for 2h in matrix. Figure 4(a) shows FESEM microstructure with EDAX analysis of aluminium alloy exhibited several black holes on the grain boundaries due to high tool rotational speed (1000 rpm), traverse speed (70 mm/min), Zn vaporization effects and high heat input (2.15 kJ/mm) of DP-FSP in matrix [14]. On the other hand, edge of spot of black hole shows moderate Zn content (7.10%), Mg content (4.27%), and high Sc content (0.52%) after EDAX analysis in matrix. Figure 4(b) shows SEM tensile fractography after DP-FSP plus aged at 140°C for 2h exhibited overall ductile mode of fracture with several dip holes due to Zn vaporization effect indicated by red arrows. Table 2 shows results of mechanical properties with specified process parameters of DP-FSP of aluminium alloy.



(a) (b) (c)

Figure 3: (a) TEM micrographs of aluminium alloy: (a) as-cast condition; T_6 condition; (c) SZ (T_4+DP-FSP+Aged at 140°C for 2h condition).



(a) (b)

Figure 4: (a) FESEM micrograph with EDAX analysis of aluminium alloy, (b) SEM tensile fractography. (T_4+DP-FSP+Aged at 140°C for 2h condition)

Table 2: Results of mechanical properties of aluminium alloy after T_4+DP-FSP+140°C for 2h.

7075 Al alloy	Tool rotational speed (rpm)	Traverse speed (mm/min)	Axial force (kN)	0.2%PS (MPa)	UTS (MPa)	%El	VHN (10kg.)	n value
	1000	70	15	104.4	248.5	6.7	139.6	1.60

The ageing process in aluminium alloy (7075 series) is complex and the decomposition of supersaturated solid solution achieved after water quenching and subsequent natural ageing phenomenon which enhances minor Sc addition. Among this metastable ή phase density may be accelerated due to minor Sc addition after 140^{o}C for 6h ageing treatment. The high hardness of 172HV (10kg. load) was achieved due to optimum density of fine precipitates in TEM micrograph (Figure 3.b). In addition, FSP was developed as a generic tool for surface modification based on the basic principles of FSW (friction stir welding). FSP causes intense plastic deformation and elevated temperature (450^{o}C) in SZ due to dynamic recrystallization [15, 16]. The SZ is the central zone in between TMAZ (thermo-mechanically affected zone) and HAZ (heat affected zone), which has been created during DP-FSP as shown in Figure 1. The optical microstructure of T_4 condition achieved average grain size of 152.34±8.93 µm rather after DP-FSP in SZ showing average grain size of 4.85±1.94 µm due to elimination of porosity, break-up cast dendritic structure, homogenization of coarse second phase particles, and intense plastic deformation with raising temperature (450^{o}C) causes grain refinement in matrix (Figure 2.a-b) [17-19]. The TEM microstructure of aluminium alloy exhibited fine precipitates of ή phases (average size of 11.97±2.60 nm) and existence of Al_3Sc particles (average size of 87.10±22.61 nm) with some black holes (aspect ratio of 1.51±0.464) formation due to Zn vaporization defects in matrix (Figure 3.c) [20-24]. The mechanical properties likely hardness and tensile properties are enhanced as139.6HV, UTS (ultimate tensile strength) of 248.5 MPa, elongation of 6.7% with high strain hardening (n) of 1.60. The above properties are improved due to fine dispersion of hardening phases (ή, Al_3Sc) after DP+FSP plus post ageing treatment at 140^{o}C for 2h of aluminium alloy (Table 2) [25,26].

IV. CONCLUSIONS

(1) The ageing process 7075 aluminium alloy is complex and the decomposition of supersaturated solid solution achieved after water quenching and subsequent natural ageing phenomenon which enhances minor Sc addition.
(2) The aluminium alloy (7075 series) usually contains minor Sc (0.20%) which form extremely fine precipitates of ή phases (11.97±2.60 nm) and existence of Al_3Sc particles (87.10± 22.61 nm) after T_4 and DP-FSP plus post ageing treatment condition.
(3) The TEM and FESEM investigates fine precipitates and several black holes on the grain boundaries after DP-FSP condition.
(4) The SEM tensile fractography revealed several black holes of Zn vaporization effects created due to high heat input (2.15 kJ/mm) and high tool rotational speed after DP-FSP condition.
(5) The mechanical properties have been evaluated as 0.2%PS of 104.4 MPa, UTS of 248.5 MPa, elongation of 6.7%, hardness along the SZ of 139.6HV and high strain hardening (n) value of 1.60 indicates good toughness after T_4 and DP-FSP plus post ageing at 140^{o}C for 2h.

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