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Microstructural refinement through Sc inoculation and its effect on mechanical properties of as-cast 7xxx series Alalloys

P.K. Mandal
Department of Metallurgical & Materials Engineering, Indian Institute of Technology Roorkee (IITR), Roorkee, UK-247667, INDIA

Abstract:- In this study the effect of scandium (Sc) grain refiner on the microstructural and mechanical properties of Al-Zn-Mg-Sc alloys (7xxx series) were investigated. Scandium is most effective to refine Al-Zn-Mg alloys. Scandium is known to be eutectic with aluminium, having a maximum solubility of 0.35 wt% Sc and a eutectic isotherm at 655 ^{o}C. Scandium forms coherent stable Al_3Sc dispersoids with aluminum and has an equilibrium $L1_2$ structure. The present paper investigated on the nucleant effects and solute effects on grain refinement and look intoimportance of dissolved scandium in promoting nucleation of α-Al on nucleant particles. Therefore, the particles of Al_3Sc are crystallized and serve as centers of grain nucleation of aluminium solid solution. Significant improvements in mechanical properties were obtained with the addition of grain refiner combined with T_6 heat treatment. The fracture surface and microstructure evolution was characterized by optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Keywords: grain refinement, nucleant effects, activation energy, artificial ageing, Al-Sc master alloy.

I. INTRODUCTION

The Al-Zn-Mg alloys (7xxx series) have been identified as an important and useful engineering material. It is attracted by its various unique properties, such as age-hardening, strength-to-weight ratio, excellent thermal properties and good mechanical behavior [1]. The common feature of these alloys is high volume fraction of alloying elements, which leads to severe dendrite and grain boundary segregation in the as-cast alloy. The strength of Al-Zn-Mg alloys generally increases with an increase in the amount of Zn+Mg content and Zn:Mg ratios [2]. Adding minor Sc to the present alloys obtain equiaxed structure but the dendrite structure and the second-phase within the grains disappear completely [3]. These grains refining of aluminium alloys can be achieved by melt inoculation with Sc containing master alloy. Numerous potent heterogeneous nuclei of Sc are dispersed in the melt, and a large number of these sites become active during solidification and nucleate the solid. The Al_3Sc phase is a stable heterogeneous particle and can intensively refine aluminum grains when the concentration of Sc in the melt exceeds the critical limit [4]. The aim of this work is to study the effect of scandium inoculation on microstructural refining effect on the hardness as well as on tensile properties of 7xxx series Al-Zn-Mg alloy and how it affect its metallurgical behavior have been discussed [5].

II. MATERIALS AND METHODS

The material used in this study is a conventional 7xxx series of aluminium alloy with Sc grain-refiner. The nominal compositions of the studied alloys were determined by inductively coupled plasma mass spectrometry (ICP-MS) and atomic absorption spectroscopy (AAS) andlisted in Table-1. The starting materials were 99.99%Al,99.95%Zn, 99.5%Mg, and Al-2wt.%Sc master alloys. The alloys were melted in a muffle furnace and then poured into mild steel mould (160×90×24 mm^3). Grain-refiner was added prior to pouring at a temperature near 780 ^{o}C to ensure dissolution of all Al_3Sc particles. After solutionization at 465 ^{o}C for 1 h cast alloys were quenched by cold water, and held at room temperature for one week followed by ageing at 120 ^{o}C (i.e. T_6 condition). The hardness was measured via Vicker's hardness tester with 15 kgfload at different intervals 5, 15, 30, 60, 120, 240, 360, 480, 600 and 720 min. Tensile samples were taken from the solutionized plates. Tensile specimenshaving 5 mm gauge diameter and 25 mm gauge length were tested using Instron tensile machine at a cross head speed of 1 mm/min. prior testing, the specimens were aged at 160 ^{o}C for 2 h for artificial T_6 treatment. The metallographic specimens were examined under optical microscope after etching with a modified Keller's reagent.The as-cast samples were analyzed byelectron probe microanalysis (EPMA) to examined segregation on grain boundary. The ageing kinetics was determined using Vicker's hardness measurement. The activation energy (E_a) was determined usingArrhenius equation. The second-phase precipitation was revealed by field emission scanning electron microscopy (FESEM) with EDX. The fracture surface of tensile specimens was studied by scanning electron microscopy (SEM) analysis. As-cast grain size was measured by linear intercept method.

Table-1: Chemical composition of studied alloys (in wt. %).

Alloy no.	Zn	Mg	Sc	Si	Fe	Al	Zn+Mg	Zn/Mg ratio
Alloy-1	5.77	3.68	0.00	0.03	0.40	Bal.	9.45	1.57
Alloy-2	5.3	3.0	0.25	0.11	0.10	Bal.	8.30	1.77
Alloy-3	5.65	1.98	0.63	0.26	0.26	Bal.	7.63	2.85

III. RESULTS AND DISCUSSION

The optical micrographs of the studied alloys are shown in Fig.-1. Coarsened as-cast grains, broaden grain boundaries and pitting types segregation can easily be observed in the Alloy-1 without Sc addition (Fig.-1.a). Minor Sc can significantly refine the as-cast grain in the Alloy-2 and Alloy-3 having average grains sizes of 36.9 µm and 22 µm whereas, as-cast Alloy-1 having average grains size of 41 µm. However, dendritic structures are completely disappeared in the Alloy-3 (Fig.-1.c). TheEPMA analysis reveals grain boundary segregation of solute atoms in Alloy-1 (Fig.-1.d). After solution treatment, the Alloy-2 (Fig.-1.e) exhibits obvious equiaxed microstructure and homogenization of solute concentration in the matrix. The several dispersoids of fine second phase particles were observed when the Alloy-3 aged at 160 ^{o}C for 2 h as shown in Fig.-1.f.

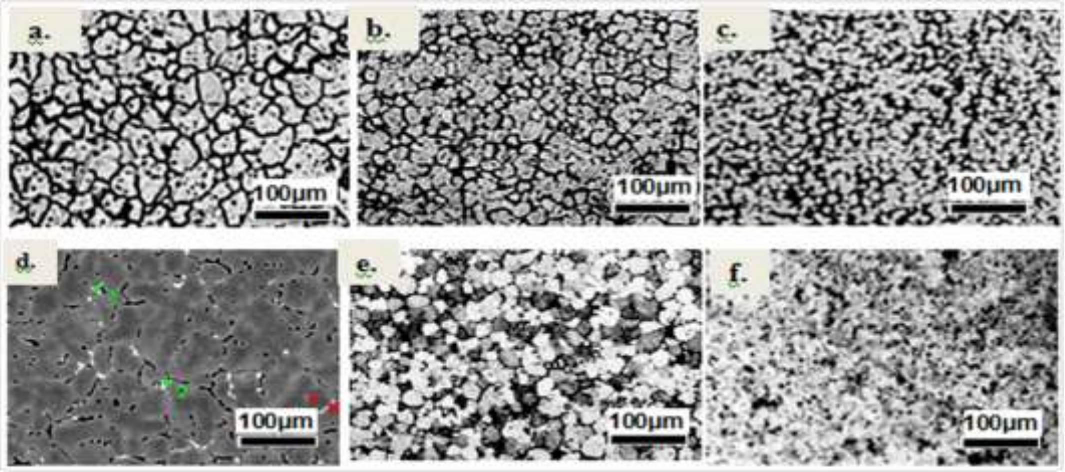


Fig.-1:Optical micrographs of as-cast alloys: (a)Alloy-1, (b)Alloy-2, (c) Alloy-3;(d) EPMA spot analysis of as-castAlloy-1;(e) Optical micrograph of solutionized (at T_4 condition) Alloy-2;(f) Optical micrograph of aged (at T_6 condition) Alloy-3.

The SEM micrograph of the tensile fracture surface of the Alloy-3 (Fig.-2 (a)) represents the evidence of ductile failure and fine precipitates on the grain boundary are indicated by arrows [6]. The TEM micrograph revealed small dispersiods of secondary phase precipitates in the matrix indicated by arrows and strongly pin the dislocation adjacent to the grain boundary as shown in Fig.-2(b). The activation energy (E_a) of 18.77 KJ/mole is required for ή-phase transformation of Alloy-3 for 10 h ageing time [7]. E_a has been calculated by Arrhenius equation by plotting ln(ΔHV) versus 1/T, the slope of the linear regression fitting is shown in Fig.-3(a). The Fig.-3(b) illustrates age-hardening phenomena at 120 ^{o}C for 12 h and the Guinier-Preston (GP) zone formation is dominating rather than Sc grain-refinement.

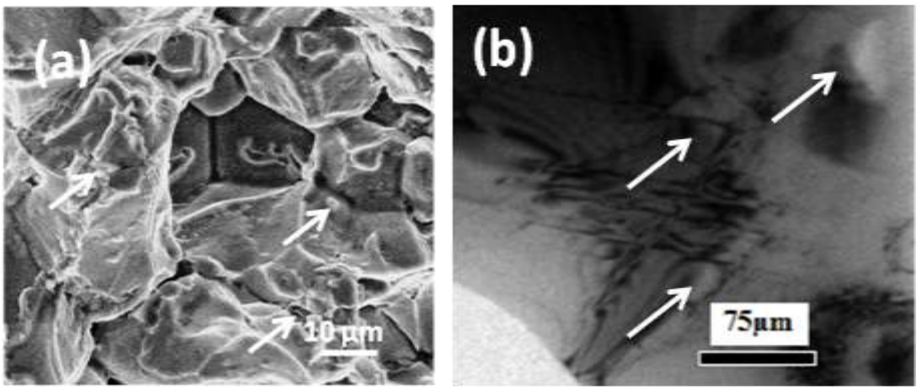
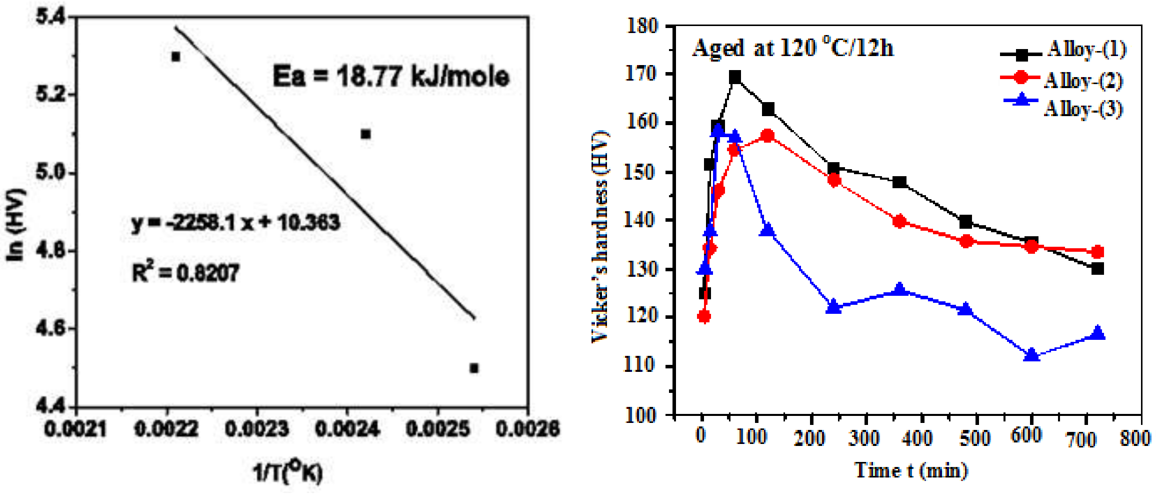


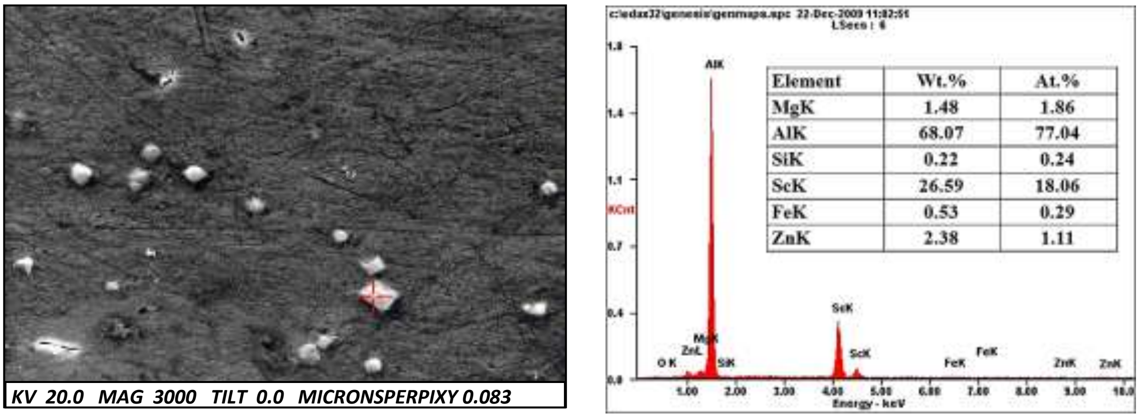
Fig.-2: (a) SEM-Tensile fractograph (T_6 treatment) of Alloy-3; (b) TEM micrograph of as-cast Alloy-2.



(a) (b)

Fig.-3: (a) Activation energy curve of Alloy-3; (b) Ageing curve at 120°C of Alloy-1, Alloy-2 and Alloy-3.

In Fig.4, FESEM analysis indicates Sc cuboids in the matrix in Alloy-3 as reflected in EDS analysis. In Al-Zn-Mg alloys, the following precipitation sequence is oftenly attributed: Supersaturated solid solution→GP zones→ή→η (Zn_2Mg). The maximum hardness is normally achieved when the ή precipitates are predominant. Age-hardening is a function of total Zn and Mg content and Zn:Mg ratio. If Zn:Mg ratio is greater than 2 then η(Mg_2Zn) phases formed. Subsequently, if Zn: Mg ratio is less than 2 then T($Al_2Mg_3Zn_3$) phases formed [8].



Element	Wt.%	At.%
MgK	1.48	1.86
AlK	68.07	77.04
SiK	0.22	0.24
ScK	26.59	18.06
FeK	0.53	0.29
ZnK	2.38	1.11

Fig.-4: FESEM photograph with EDS analysis of as-cast Alloy-3.

Generally, common precipitation hardening temperatures are in the range 70-200°C, and a two-step precipitation heat treatment within this temperature regime is often used. Several researchers are pointed out that, there is no effect of Sc on the precipitation behavior of ή, and yet others report that Sc additions significantly speed up the precipitation of ή. Meanwhile, Sc addition leads to coarser ή precipitates and it is claimed that this gives an improved ductility of alloy [9].However, it is also reported that the mechanisms proposed for grain refinement are based on heterogeneous nucleation of aluminium grains on inoculants particles Al_3Sc, grain growth retardation by solute additions and edge-to-edge matching of the planes in the inoculants particle and nucleating aluminium grain. Therefore, scandium additionto aluminum is an efficient way to achieve grain refinement during solidification of the molten metal and also during subsequent mechanical and thermal processing stages. It is reported that the crystallographic similarity is a main cause of the dramatic modifying effect of scandium. In this work, it is found that the Al_3Sc dispersiods are very effective in preventing recrystallization up to temperatures of ~350-400°C. A more important contribution from the dispersoids is their ability to stabilize the grain/subgrain structure of an alloy through Zener-drag action [10], and thus improve the mechanical properties of the alloy. The tensile properties ofAlloy-1,Alloy-2 and Alloy-3are illustrated in Table-2. The tensile strength is increasing monotonically with the addition of Sc in case of as-cast alloys and gives ~39% improvement due to fine grain strengthening, substructure strengthening and dispersion strengthening by Al_3Sc. However, in case of T_6 condition ~34% improvement except in Alloy-2 is observed. In Alloy-2, there is no improvement due to precipitation coarsening effect.

Table-2: Tensile properties of as-cast and after ageing at 160 °C for 2 h of studied alloys.

Alloy no.	As-cast condition			T_6 condition		
	$\sigma_{0.2}$ (MPa)	σ_u (MPa)	δ(%)	$\sigma_{0.2}$ (MPa)	σ_u (MPa)	δ(%)
Alloy-1	84.0	210.8	7.37	130.8	290.6	3.92
Alloy-2	112.1	249.4	2.28	111.8	243.3	2.22
Alloy-3	253.0	292.2	4.96	360.0	391.4	4.3

IV. CONCLUSIONS

(1) In Al-Zn-Mg alloys containing Sc, the primary Al_3Sc particles precipitated from the melt during solidification is the most effective grain refiner for α-Al matrix, the secondary Al_3Sc precipitated during ageing can effectively restrain recrystallization during solid solution treatment.

(2) The tensile strength of as-cast alloys with the addition of Sc is enhanced and gives a maximum improvement of ~39% while in case of T_6 condition only ~34% improvement is achieved. The elongation decreases in both the cases due to hardening effect.

(3)The activation energy (E_a) has been found out of 18.77 KJ/mole for Al-Zn-Mg-Sc alloy at 10 h ageing time.

(4)The addition of Sc improves the mechanical properties of the alloys. The strengthening mechanisms such as fine grain strengthening, substructure strengthening and dispersion strengthening by Al_3Sc are found to be mainly responsible.

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