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Effect Of Small Addition Of Sc In Artificially Aged Of Cast Al-Zn-Mg Alloy

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ABSTRACT: The Al-Zn-Mg alloys were prepared through cast metallurgy route. Effects of minor scandium (Sc) on the precipitates of an age-hardenable Al-Zn-Mg alloy aged at 80 °C to 180 °C have been studied by optical microscopy (OM), differential thermal analysis (DTA), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis. Results show that the addition of Sc in Al-Zn-Mg alloy is capable of refining cast grains obviously. Interpretation of results is done in terms of essential transformations involving GP zones, the metastable ή hardening precipitates and the stable phase η ($MgZn_2$) respectively. Special focus has been placed on the study of microstructural evolution during initial phases of artificial ageing. Addition of Sc is found to improve the strength appreciably through strengthening mechanisms of precipitation hardening and grain refinement. This is due to the precipitation of secondary Al_3Sc precipitates and noticeable grain refinement. The aim of the study is to elucidate effect of Sc on ageing kinetics of Al-Zn-Mg alloy.

Key words: cast metallurgy, minor scandium, age-hardenable, TEM, grain refinement, Al_3Sc precipitates etc.

1 INTRODUCTION

The age-hardening characteristic of ternary Al-Zn-Mg alloy has received significant research attention because of its technical importance [1-3]. According to Graf and Schmalzried and Gerold, ordered GP zones exist in temperatures ranging between 75 °C to 150 °C. Gjonnes and Simenson proposed that ή forms in the initial stage of ageing of an Al-Zn-Mg alloy at 130 °C to 150 °C [4]. The microstructure primarily comprises GP zones and metastable ή phases in such conditions. Furthermore, T. Ungar et al. [1979] suggested that η phases have existed above about 200 °C [5]. The alloy chemical composition examined with age-hardening curves in the present paper achieved peak hardness level during 140 °C for 6h ageing time. This occurred due to combine effects of total solute contents including Zn/Mg ratio and Sc addition [6, 7]. Therefore, the optical micrograph and the macro hardness testing results are consistent, which indicates that the addition of Sc not only retards the recrystallization, improves recrystallization temperature, and accelerates ageing kinetics, but also refines the recrystallization microstructure [8-11]. In addition, Al_3Sc primary particles can act as nucleus for heterogeneous nucleation, remarkably enhance nucleation ratio and refine the cast grains. Thomas and Nutting [1959-60] and DeArdo and Simenson [1973] asserted that for strengthening, degree of dispersion of both the η and ή phases is more important than precipitate type. In this paper, Sc is added to Al-Zn-Mg alloy and the effects of minor Sc(≈0.27%) on the ageing-hardening characteristic, grain refinement, mechanical properties and microstructures of this alloy have been studied. Several metallurgical characterizations have been done at peak-aged evolution at 140 °C/6 h ageing time of studied aluminium alloy. Ageing kinetics is revealed through Vicker's hardness measurement.

2 EXPERIMENTAL PROCEDURE

The alloy was prepared by cast metallurgy route with high purity aluminium, zinc, magnesium, and master alloy Al-2 wt.% Sc. The electrical resistance muffle furnace was used to melt the alloy at 780 °C. In order to prevent burning of zinc and magnesium during melting of aluminium, both elements were added just before pouring in metallic mould. In addition, the cast alloy was gone through several heat treatments likely to solution treatment (i.e., heated up at 465 °C/1h then immediately water quenched; T_4), naturally aged (in room temperature at a week) and artificially aged (80 °C to 180 °C; T_6), respectively. The chemical composition of alloy was tested through ICP-MS (inductively coupled plasma mass spectroscopy) and revealed (7000 series) Al-5.1Zn-3.1Mg-0.27Sc-0.08Si-0.10Fe (in wt.%) alloy. The composition was selected to existing Zn/Mg ratio to ≈1.64 and total solute content ≈ 8.2 wt.%. The metallographic specimens were examined under optical microscope (LEICA DMI 5000M, Leica Microsystems, Buffalo Grove, IL) after etching using modified Keller's reagent (2.5 ml HNO_3+1.5 ml HCl+1 ml HF+ 175 ml water). The polished samples were subjected to grain size analysis, OM, DTA, SEM, and TEM analyses. The artificial ageing performed in electrical resistance muffle furnace at 80±2 °C, 120±2 °C, 140 ±2 °C, and 180±2 °C, respectively. The ageing kinetics was characterized through Vicker's hardness tester (Model no.: FIE VM50 PC) with 10 kg. load of studied alloy. The each sample dimension was 10×10×10 mm^3 size. The ageing curves were plotted at systemic time intervals likely to 5, 15, 30, 60, 120, 240, 360, 480, 600, 720 and 960 min with 15 sec dwell time. All the ageing specimens characterized within 10 min during hardness measurements. The differential thermal analysis (DTA) measurements were performed in a EXSTAR TG/DTA 6300 equipment with a 10 °C/min heating rate until 600 °C in nitrogen atmosphere on aged (at 140 °C/6 h) alloy. The cast sample was analysed by SEM (scanning electron microscopy) (Model: LEO 435 VP). The TEM (transmission electron microscopy) samples were prepared using twin-jet electro-polishing (solution was 75% CH_3OH and 25% HNO_3) at 12 V and -35 °C. All the imaging was carried out using at Techai G^2 20 S-TWIN at 200 kV. The peak-aged hardness data of different ageing temperatures has been tabulated in Table-1. The mechanical characterization was done through tensile testing of round shape samples (25 mm gauge length and 5 mm in gauge diameter) on T_6 alloy (aged at 160 °C/2h) and the results are shown in Table-2.

3 RESULTS AND DISCUSSION

An experimental analysis of high strength Al-Zn-Mg-Sc alloy precipitation processes is presented in this paper due to its attractive mechanical properties. Artificial ageing curves were

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constructed using Vicker's hardness measurement for this alloy on the basis of 7000 series with duplex treatments (i.e., natural ageing and artificial ageing) widely used in industries. Industrial 7000 series heat treatments are commonly founded on duplex ageing schedules involving initial low temperature ageing after solution treatment. Then, a second slightly elevated temperature ageing is done. The two step ageing treatment results in a noticeably high strength as compared to the single ageing treatment because of a finer dispersion of GP zones [12]. It is also mentioned that the after Sc inoculated in aluminium alloy refine grains as well as accelerated ageing kinetics promptly. This article offers enhanced knowledge of ageing kinetics and control of significant features influencing balance properties in 7000 series of aluminium alloy. For a particular peak-ageing treatment at 140 ^{o}C/6h with Zn/Mg ≈1.64 ratio and Sc inoculation is mainly discussing matter. The precipitation sequence in 7000 series is usually exhibited first stage precipitates phase GP zones formation at 80 ^{o}C to 120 ^{o}C. The second stage precipitates phases are ή-$MgZn_2$ formation at 130 ^{o}C to 150 ^{o}C. It has second stage product of peak-aged formation. Consequently, the third stage precipitates phases are η-$MgZn_2$ formation at 150 ^{o}C to 200 ^{o}C. However, at higher temperatures, the yield strength of 7000–T_6 rapidly decreases due to dissolution or transformation of the metastable phases (ή) into the equilibrium η phase. Furthermore, in Al-Zn-Mg alloy with relatively high Mg contents may be formed complex cubic precipitates T ($Mg_{32}Al_{49}$ Zn_{49}), also [13]. It is believed that the probability of the transformation occurring will depend upon the activation energy required for the transformation in the crystal structure. Fig.-1 optical micrograph shows the effect of Sc inoculation in equiaxed fine grains of cast aluminium alloy. The grain size estimated 30±5 µm in T_6 alloy. It has categorically mentioned to solution treated (465 ^{o}C/1h), quickly water quenched then ageing at elevated temperatures.

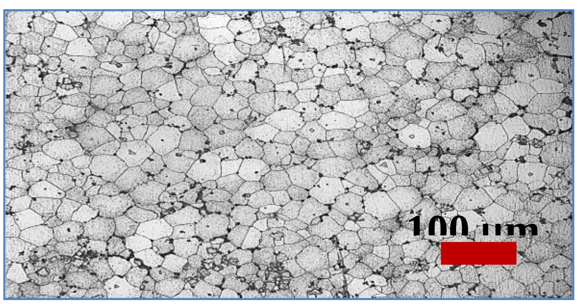

Fig.-1: *Illustration of optical micrograph of studied alloy (aged at 140 ^{o}C/6h).*

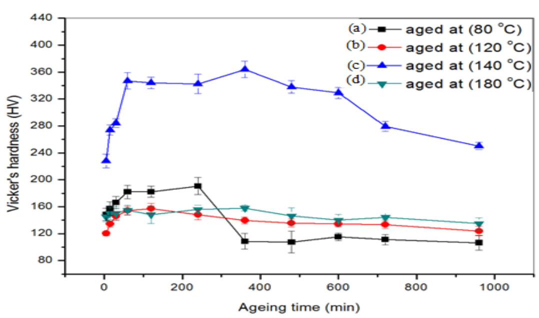

Fig.-2(a-d): *Illustration of ageing curves at different temperatures upto 16h ageing time of studied alloy.*

On the other hand, DTA and TEM studies were revealed on the alloy throughout the ageing process (at 140 ^{o}C/6h) to fully understand the precipitation events occurring. Fig.-2(a) shows that at 80 ^{o}C ageing phenomena, highest hardness is exhibited at 4h ageing time to 190.8±12.6 HV due to coherent aggregates of GP zones formation with Al_3Sc particles. These Al_3Sc particles may not directly affect as seeds for nucleation of new particles. However, Zn-rich VRC (vacancy rich cluster) may influence GP zone formation during quenching process following solution treatment. Main fact is that vacancy-solute interaction with total solute contents mainly cause of high hardness such as lower ageing temperature. Thus, after peak hardness attended this curve tends to constant lower hardness may due to coherency loss of hardening particles at this ageing regime. Fig.-2(b) shows at 120 ^{o}C ageing phenomena, when highest hardness exhibited at 2h ageing time to 157.4±7.6 HV due to loss of hardening effects for comparing with lower ageing at 80 ^{o}C. GP zones with Al_3Sc particles coherency main factors of higher hardness at this ageing regime. Fig.-2(c) shows at 140 ^{o}C ageing phenomena, when highest hardness exhibited at 6h ageing time to 364.0±12 HV due to formation of metastable ή-$MgZn_2$ phases with Al_3Sc particles synergic effects main cause of highest hardening effects at this ageing regime. Maximum hardness is dependent on the coherency of the precipitates with the matrix, size and distribution, and the vicinity of particles. Also, coherency precipitates generate a higher strain field in the matrix resulting in increased hardness. This ageing regime exhibited highest hardening effects because greater volume fraction of precipitates contributes to a higher elastic modulus. It is concluded that at this ageing regime mostly considerable phases are ή plus Al_3Sc particles which precipitated homogeneously. In a result, ageing process has accelerated and ageing effect is improved. Fig.-2(d) shows at 180 ^{o}C ageing phenomena, when highest hardness exhibited at 6h ageing time to 158.0±4.2 HV due to coherency loss of main hardening particles η-$MgZn_2$ with Al_3Sc. Age-hardening effect is part of the transformation process generally involving heterogeneous nucleation at sites of earlier products. Fine and uniform precipitate dispersions are a result of this process.

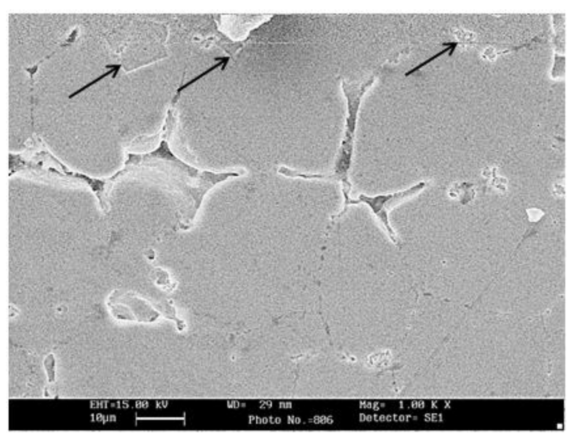

Fig.-3: *The SEM micrograph of T_6 alloy (aged at 140 ^{o}C/6h).*

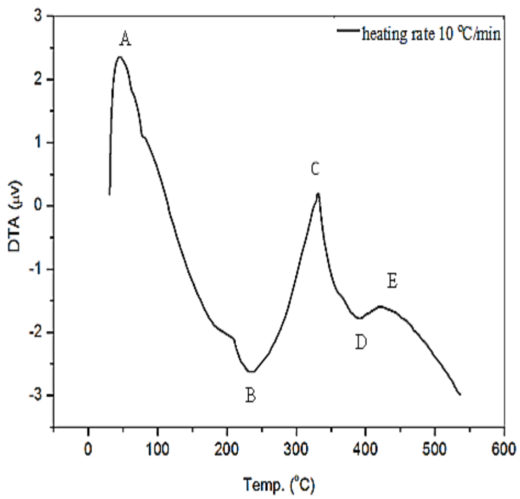

Fig.-4: *The graphical representation of DTA curve of T_6 alloy (aged at 140 ^oC/6h).*

Therefore, after longer ageing time with elevated temperature the equilibrium phase has gradually loss their coherency. In addition, all peak-aged hardness values are shown in Table-1. Fig.-3 SEM micrograph shows that cuboid particles of Al_3Sc which marked by arrows. Also, indicated grain boundary thickening owing to as-cast inhomogeneous segregation of eutectic phases. To understand how the alloy behaves during ageing treatment at 140 ^oC/6h, it is necessary to examine the ageing sequence that can occur during 10 ^oC/min heating cycle during DTA. The studied alloy has gone through solution treatment, water quenching, aged then opted for DTA analysis. Fig.-4 DTA curve exhibits that three distinct exothermic peaks (A, C, E) and two endothermic peaks (B, D), respectively. This shows that three exothermic reactions and two endothermic reactions occur between nearly 50 ^oC to 415 ^oC and nearly 235 ^oC to 390 ^oC, respectively. At 50 ^oC, an exothermic peak A is first observed that corresponding to the GP zones plus Al_3Sc particles during early stage of ageing precipitation. At around 330 ^oC, an exothermic peak C is second observed that corresponding ή-phases plus Al_3Sc particles anti-recrystallization effects confirmed. At around 420 ^oC, an exothermic peak E is third observed that corresponding η-phases plus Al_3Sc particles tends to dissolution effects confirmed.

(a)

(b)

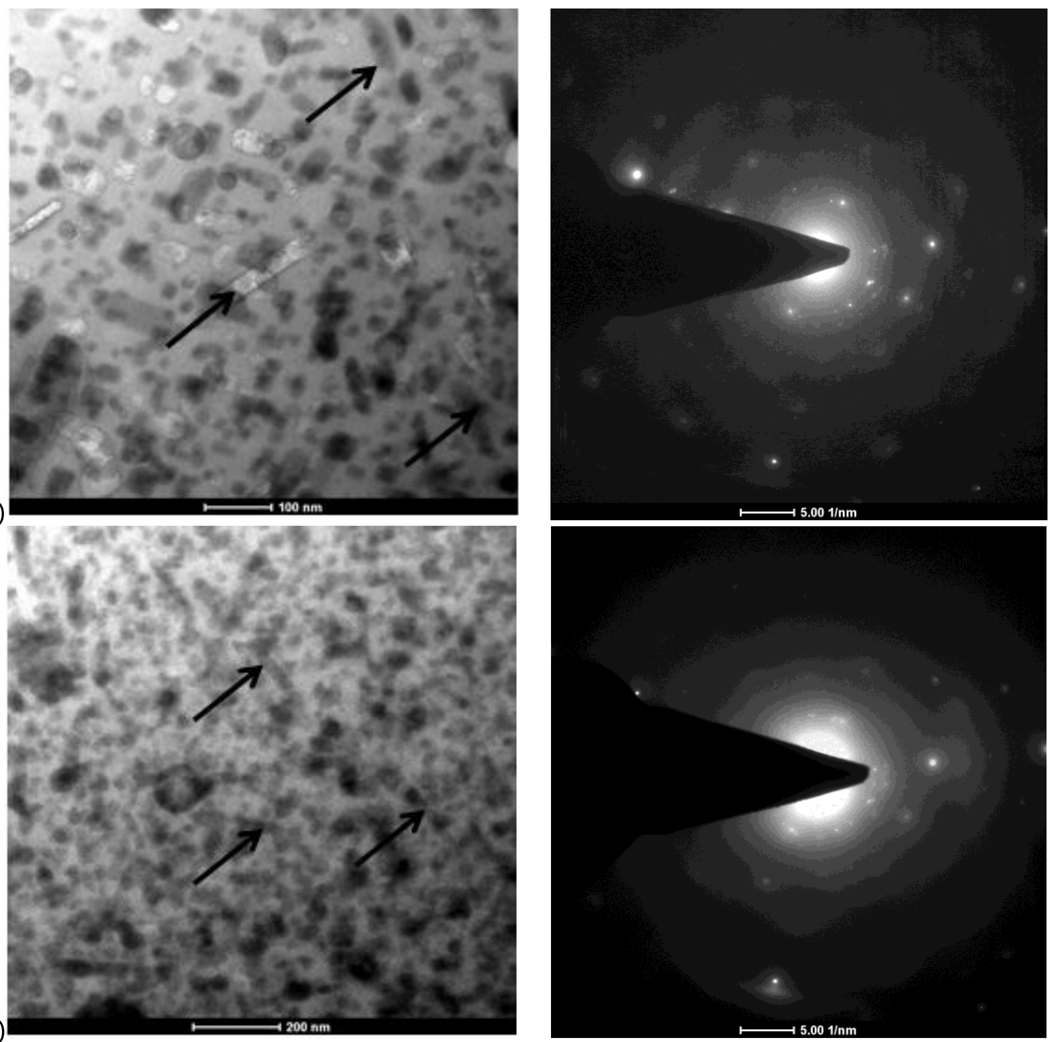

Fig.-5(a-b): *Illustration of TEM micrographs with CCD (charge- coupled device) pattern at different magnifications of T_6 alloy (aged at 140 ^oC/6h) of studied alloy.*

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Consequently, at 242 ^{o}C, an endothermic peak B is first observed that corresponding to the GP transformation that corresponding to ή-phases plus Al_3Sc particles dissolution effects confirmed. At 392 ^{o}C, an endothermic peak D is second observed that corresponding to the η and Al_3Sc hardening precipitates dissolution during thermal heating cycles. Fig.-5(a-b) shows TEM analysis with inset CCD pattern at different magnifications after ageing at 140 ^{o}C/6h in aluminium alloy, many fine precipitates (ή) and some larger particles distribute in the matrix. But some of bigger particles (mark by arrows) perhaps due to fact that the fine Al_3Sc agglomeration with main hardening phases at this ageing temperature which do not dissolve even after solution treatment and finely distributed in matrix. In CCD patterns indicated uniform distribution of fine submicroscopic precipitates with mixer of larger precipitates which has agglomeration of hardening phases with Al_3Sc particles inside circles. In Table-2 shows mechanical properties evaluated under aged at 160 ^{o}C/2 h (T_6) condition of studied alloy. It can be seen that the ageing strengthening effect of alloy is marginally high of 0.2% proof stress, UTS and ductility exhibited 301.8 MPa, 377.4 MPa, ductility 3.04 % and average hardness 138.6 HV, respectively. The maximum strength achieved due to uniform distribution of hardening precipitates with Al_3Sc is coherently dispersed throughout in matrix of the alloy which has hinted in DTA and TEM analyses.

Table-1: *Hardness attained after selected ageing treatments of studied alloy.*

Solution treatment	Ageing- 1st step	Ageing- 2st step	Peak aged hardness HV±SD (10kg.)/in hr.
465 ^{o}C/1h + water quenching	hold 7 days in RT	80 ^{o}C/16h	190.8±12.6/4h
as above	as above	120 ^{o}C/ 16h	157.4±7.6/2h
as above	as above	140 ^{o}C/16h	364.0±12.0/6h
as above	as above	180 ^{o}C/16h	158.0±4.2/6h

Table-2: *Results of mechanical properties at T_6 condition of studied alloy.*

Alloy	Aged at 160 ^{o}C/2h			
	$\sigma_{0.2}$ (MPa)	σ_u (MPa)	δ (%)	HV(10kg.)
	301.8	377.4	3.04	138.6

4 CONCLUSIONS

i) The duplex ageing treatment is responsible for high hardness to fine dispersion of GP zones with ή precipitates with combined effects of Al_3Sc particles in early stage of ageing.
ii) Experimental results show a significant hardening effects of Sc addition in Al-Zn-Mg alloy at 140 ^{o}C/16h ageing treatment.
iii) The net hardening effects derive from solute effects, precipitation hardening and dispersion hardening of Sc inoculated aluminium alloy.
iv) The characterization of DTA, SEM and TEM revealed several hardening phases of η, ή, T(Mg_{32} Al_{49} Zn_{49}) and Al_3Sc particles.
v) The high mechanical properties of as-cast aluminium alloy has mainly associated with the fine grain strengthening caused by the addition of Sc and chemical hardening and precipitation strengthening of Al_3Sc particles and η precipitates during T_6 ageing.

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