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Effect of scandium on ageing kinetics in cast Al-Zn-Mg alloys

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The effect of Sc addition in cast Al-Zn-Mg alloys with precipitation strengthening was studied different ageing conditions. Alloy (1–4) systems, the ageing at 140 °C for 16 h shows the moderate hardness value as compared to the solution treated samples. Further, the ageing at 120 °C the high hardness (360 HV) values for the Alloy-1system and the same was estimated (320HV) for the Alloy-1 for the 180 °C for 16 h. DSC curve for the all alloy system ageing at 140 °C for 16 h more endothermic peaks for the Alloy (3–4) system due to the $L1_2$-Al_3Sc precipitate as compared to the Alloy (1–2) system exothermic peaks. Moreover, the TEM analysis confirmed the formation the $L1_2$-Al_3Sc precipitate particles for the solution treated and aged sample. The high strength has been achieved through scandium addition in cast Al-Zn-Mg alloys through FSP (friction stir processing) by double passes. The scandium additive can retain the recrystallization behavior of Al-Zn-Mg alloys and to the increase of recrystallization temperature due to the pinning effect of fine dispersion $L1_2$-Al_3Sc precipitate. It confirmed from the optical microstructures and tensile test for the T4 and T6 condition sample. The mechanical properties have been revealed through FSP and post ageing treatment at 140 °C for 16 h. The most important results due the formation of GP zones, ή precipitates, $L1_2$-Al_3Sc precipitate and to improve the mechanical properties.

1. Introduction

High strength 7XXX series of Al-Zn-Mg alloys are widely used for modern engineering applications such as excellent structure components, automotive industries and aerospace/aeronautical industry. In the recent years, Al-Zn-Mg alloys have received a great attention due to its high strength-to-weight ratio, good corrosion resistance, good machinability, weldability, relatively low cost and its high thermal conductivity in the due to its high strength to weight ratio. The major drawback of the Al-Zn-Mg alloys decrease of strength at elevated temperatures is accounted to be behind this incompatibility. The precipitation hardening phenomenon plays a pivotal role in to improve the strength and mechanical properties at elevated temperatures [1–3]. The purpose of natural ageing is to develop a structure of GP zones which can then act as efficient nuclei for the hardening phase ή during the artificial ageing [4,5]. Many researches [6,7] are reported that the precipitation ($MgZn_2$) or ($Al_2Zn_3Mg_3$) during ageing (i.e., with an increase in the ageing time and/or temperature) at temperature below about 200 °C.The interfacial energy between the coherent GP zones and the matrix is low, so that zones of a very small size can form. [8,9]. GPI zones are coherent with the Al matrix, with internal ordering of Zn and Al/Mg from room temperature to 150 °C. GPII zones are zinc-rich layers were found after quenching from temperatures above 450 °C and ageing at 70 °C [10–12]. In that case, it is assumed that concentration fluctuations rich in Zn and Mg are left behind by GP zones which can play an important role in the formation of the ή-phase [13–15]. Generally either GPI or both GPI and GPII zones can form as precursors to the metastable η-phase. The mechanisms of GPI zone formation is linked to the relative solute-vacancy interaction. The metastable ή and T´ phase are semi coherent with the matrix and have higher interfacial energies and it requires larger critical nucleus sizes to form and grow. In the alloys with Zn/Mg ratios between about 2:1 to 3:1 the η-phase is the only stable temperature and T-phase at the higher than 200 °C. The ή and η phases are formed during ageing of the alloys having high Zn:Mg ratios (generally, > 2), while T and T´ phases are formed in the alloys with moderate to low Zn:Mg ratios (generally, < 2) [16,17]. The η-precipitates are incoherent with the matrix. At least 11 different crystallographic orientation relationships have been reported in the literature [18,19]. Furthermore, the effect of ageing is amplified by the simultaneous addition of scandium as the precipitates of the $L1_2$ phase Al_3Sc are supposed to have an even stronger anti-recrystallization effect [20,21]. The Al-Zn-Mg-Sc quaternary alloys allow precipitation hardening in a duplex manner during the solutionizing and artificial ageing treatments because of the slow precipitation rate of scandium comparing with those of Zn and Mg. The one is due to the precipitation of the metastable phases of Al_3Sc in solutionizing and the other GP zones and /or η precipitates during ageing treatment [22,23]. On the other hand, Al-Zn-Mg is aged to gain its high strength, heterogeneous precipitation

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occurs on the grain boundaries, making the alloy into brittle manner for grain boundary segregation [24]. The presence of a deformed microstructure may affect the nature and kinetics of the precipitation, and the presence of precipitation may interfere with the recovery and recrystallization processes [25]. Therefore, the SPD (severe plastic deformation) technique is generally applied for obtaining ultrafine grained microstructure in the studied alloys. Friction stir processing (FSP) is the most often used method among SPD procedures since it results in homogeneous ultrafine grained microstructure without contamination and changing the dimensions of the bulk material [26–29]. The stir zone grains suggest effective strains together with a microstructural evolution that occurs by a combination of hot working and a dynamic recovery or recrystallization [30–34]. Although these alloys rarely reported that the age hardening behavior, DSC analysis, mechanical properties and frictions stir processing and the precipitates microstructures with high dislocation density and ultrafine grains have been investigated under deformation of the alloys.

The main objective of the present study is to investigate the effect scandium content on the microstructure and mechanical properties of Al-Zn-Mg alloys. Four different Al-Zn-Mg-Sc alloys with high Zn + Mg contents (8, 9 & 10 wt%) were prepared by melting and casting under ambient condition in electrical resistance muffle furnace followed by aging treatments, DSC analysis, microstructure analysis, friction stir processing and mechanical properties such as tensile test and vickers hardness measurements.

2. Materials and methods details

2.1. Materials

The materials used for the work includes; pure Aluminium, Scandium, Magnesium and Zinc.

2.2. Methods

The 7XXX series of aluminium alloys were prepared by melting and casting under ambient condition in electrical resistance muffle furnace. The preheating/melting temperature was set at 780 °C. Initially pure aluminium was kept in graphite crucible in muffle furnace to preheating at above set temperature. Initially stage, melt the pure aluminium (as liquefied condition at 780 °C) then addition of pure scandium 2 wt%. and stir the molten metal in the crucible for half-an-hour to dissolve with molten aluminium. Second stage, after half an hour the crucible was taken out from the furnace. Finally, the pure Mg and pure Zn were added sequentially in liquid molten aluminium metal. The molten metal was poured in a metallic mould at dimension $160 \times 90 \times 24$ mm^3. The composition of cast alloys was determined by ICP-AES (inductively coupled plasma atomic emission spectroscopy) and OES (optical emission spectrometry) methods are shown in Table 1.

2.3. Ageing treatment

There are various heat treatments conditions have been carried out such as solution treatment, water quenching and ageing treatments. The heat treatment was carried out in tubular furnace (± 1 °C). The cast alloys (dimension of $20 \times 20 \times 10$ mm^3) was given a solution treatment at 465 °C (± 5 °C) for 1 h followed by quenching in water. The solution treatment was carried out at different temperatures 120 °C, 140 °C and 180 °C, respectively. Moreover, the ageing time was varied to 5, 15, 30, 60, 120, 240, 360, 480, 600, 720 and 960 min (16 h) at each ageing temperature.

Table 1
Chemical compositions of the studied alloys (all in wt%).

Alloys	Chemical composition						Zn + Mg	Zn/Mg Ratio
	Zn	Mg	Sc	Si	Fe	Al		
1	6.50	2.90	–	0.01	0.05	Balance	9.40	2.24
2	6.65	2.90	–	0.03	0.01	Balance	9.55	2.29
3	6.60	3.81	0.39	0.01	0.02	Balance	10.41	1.73
4	5.93	2.90	0.45	0.10	0.19	Balance	8.83	2.04

2.4. Microstructures analysis

Microstructures study was carried out through OM (optical microscopy)and TEM (transmission electron microscopy) analysis. The sample for the optical microstructures (OM) were polished with different grade of silicon carbide emery paper and followed by cloth polishing (Al_2O_3 powder particles size 0.5–1.0 µm)and diamond paste polishing. The polished specimens were etched with modified Keller's reagent to reveal microstructure using LEICA DMI 5000 M (Leica Microsystems, Buffalo Grove, IL). Similarly, the TEM analysis has been carried out as-cast and aged (i.e., aged at 140 °C for 6 h) alloys. The TEM slice with 3 mm diameter was cut from the sample 10×10 mm^2 discs and thickness reduced to 0.1 mm by disc polisher. Then, the TEM samples were polished using twin-jet electro-polishing (solution was 75% CH_3OH and 25% HNO_3). TEM was carried out using at Techai G^2 20 S-TWIN at 200 kV. The activation energy (Q) was determined using Arrhenius equation. DSC (Differential scanning calorimetry) was carried out as aged alloys (i.e., aged at 140 °C for 6 h) by DSC (EXSTAR TG/DTA 6300) method, using a nitrogen atmosphere and a constant heating rate of 10, 20, 30 °C/min, respectively.

2.5. Hardness measurement

The mechanical properties were performed through the Vicker's hardness measurements(FIE VM 50 PC) machine with 10 kg load with 15 s dwell time. The average hardness 10 measurements values were taken for each condition.

2.6. Tensile strength test

The tensile test was performed in instron (universal testing machine) UTM(25 KN, H25 K-S, Hounsfield Test equipment Ltd., Surrey, UK). The tensile samples (Dimension are full length 58, gauge length 26, width 4) had collected from stir zone, and tested with cross head speed 1 mm/min at room temperature.

2.7. Friction stir processing

The cast alloys were performed by using FSP (friction stir processing) by double passes and all parameters were predetermined during metal processing. In Fig. 1(a-c), the FSP set-up of vertical milling machine is shown and used plate dimension $150 \times 90 \times 8$ mm^3. The tilt angle of the rotating tool with respect to Z-axis of the FSP machine was 2.5 (degree). The experimental plates were subjected to separate conditions as constant parameters during FSP with double passes. The processing parameters included: 75 mm/min traverse speed, 1025 rpm clockwise tool rotating speed, and 15 kN axial force, using a tool with 25 mm shoulder diameter and 5 mm pin root diameter with sharp tip with 3 mm pin depth for full penetration into the plates.

3. Results and discussion

3.1. Ageing behaviors and hardness

Fig. 1a shows the different alloys with Vickers hardness as function of solution treatment time. It can be notice that the Alloy-4 (maximum hardness of ~140 HV) is achieved at 460 °C after an ageing time of 32 h as compared to the other alloys system. Fig. 1b-d shows the cast Al-Zn-Mg alloys as a function of ageing temperature and time. In the (Fig. 1b-