THE MICROSTRUCTURE DEVELOPMENT DURING ISOTHERMAL HEAT TREATMENT STUDY OF AN Al-Mg-Si ALUMINIUM ALLOY

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Abstract

The effects of isothermal heat treatment on the microstructure development of an Al-Mg-Si aluminum alloy have been investigated by means of transmission electron microscopy. From micrographs illustrating the micro structural change of the alloy during isothermal heat treatment; we can observe in the water quenched alloy sample aged for 50 and 75 hours at 350°C, dispersoid particles that were aligned along <100> matrix direction losing their distribution at their early stage of precipitation with an increase in holding times. For the microstructure of the alloy ramp heated and aged at 450°C for different holding times, we can clearly see dispersoid particles in their early stages of formation heterogeneously distributed with different shapes after an increase in holding times. These dispersoid particles exhibited strain field contrast when they are heated at low temperatures, that indicating they are coherent or semi-coherent with the matrix. A loss of coherency was observed for most of the dispersoid particles when they were aged with high holding times.

Keywords: Al-Mg-Si alloy, isothermal heat treatment, precipitate, dispersoid, microstructure

1 Introduction

The 6xxx series aluminum alloys (Al-Mg-Si based) have been the subject of several scientific research works. Their excellent mechanical and electrical properties allowed their use in various sectors such as aerospace, automotive or electrical power transport [1-4]. In addition, with the development of the aluminum alloys industry, the requirements for Al-Mg-Si-Cu alloys with high strength and excellent plasticity have become more urgent. Thus, extensive studies on the design of alloy composition and processing technology for these alloys have been carried out in [5, 6].

The 6xxx series base metals have low alloy content and are easy to form into extrusions, tubing, forgings and other shaped products and then to obtain their maximum mechanical properties through heat treatment and ageing [7, 8]. A rapid heating rate produces large needle-shaped heterogeneously distributed dispersoids, while a slower heating rate produces fine spherical shape dispersoids with a more homogeneous distribution [9]. During homogenization, the β′ phase nucleates first on the matrix along the <100> direction of the matrix. There is a link between precipitation of the dispersoid particles and the β' Mg_2Si phase. Dispersoid particles nucleated

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during the dissolution of β' Mg_2Si. They nucleated upon the interface of the hardening phase β' Mg_2Si in order to decrease their interfacial energy and to get some of the silicon solute after dissolution of the hardening phases [10, 11].
The enhancement of strength properties obtained during the heat treatments is primarily due to the precipitation of metastable phases from the supersaturated solution. The precipitation sequence in Al-Mg-Si alloys is as follows [12-16]:

Needle- shaped GP zones → rod-like β' precipitates → platelets of Mg_2Si

The precipitation reactions in Al–Mg–Si alloys have been the subjects of numerous investigations [17-20] that are concerned with certain ageing conditions and the effects of chemical composition but ageing temperature and time on the precipitation of various intermediate phases' processes during isothermal heat treatments have not been fully understood. Thus, the main objectives of this work are the study and the investigation of the isothermal heat treatments, with different temperatures and holding times, effects on the microstructure evolution of an Al-Mg-Si alloy by means of conventional transmission electron microscopy (TEM).

2 Experimental procedures

2.1 Materials

The Al-Mg-Si alloy samples were provided by the Banbury Laboratories of Alcan International Ltd. They were prepared by direct chill casting process (DC) in a 178 mm diameter moulds and were received in the as cast condition. The chemical composition of the investigated alloys is given in **Table 1**.

2.2 Heat treatment

The alloy samples were ramp heated at a rate of 100 °C /h up to 600°C in an air furnace. After ramp heated, samples were isothermally aged at 350°C and 450°C for several holding times, with a heating rate of 100°C h^{-1} and then water quenched. To follow the nucleation and growth of the dispersoids particles, long heat-treatment times were applied in order to dissolve the coarse particles of types AlFeSi and AlMnSi that were observed in the as-cast alloys.

2.3 TEM microscopy

Electron microscopy examination was carried out with an EM 300 electron microscope at 100 keV. A liquid nitrogen-cooled decontaminator, an eccentric goniometer and double tilt holder were used in order to prevent the contamination after extended observation of an area of the thin foil.

2.4 Metallographic preparation of Thin foil

Thin foils for TEM were prepared by spark machining to form discs 3 mm in diameter. The discs were subsequently grounded with fine silicon-carbide emery paper to about 200 μm thick. Final thinning was by jet polishing using a Struers Tenupol Unit with a solution of 33% HNO_3 in Analar grade methanol at -10 to -15 volts and a temperature of -20 to -30°C. When the electropolishing was completed the specimens were removed from the solution as quickly as possible and washed with Analar methanol. The specimens were dried between filter papers and then stored in a specimen grid box under vacuum.

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Table 1 Chemical compositions of the investigated alloy

Chemical compositions of the investigated alloy (mass %)						
Si	**Fe**	**Cu**	**Mn**	**Mg**	**Cr**	**Al**
1.30	0.23	0.004	0.65	0.79	0.001	bal

3 Results and discussions

3.1 The as cast alloy studied

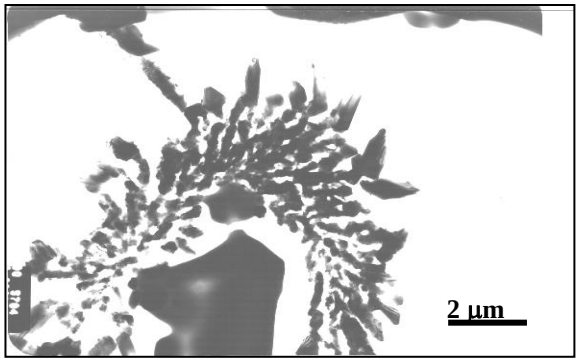

Fig. 1 Transmission electron micrograph of as-cast alloy showing coarse particles with the plate like and "Chinese script" morphology

Fig. 1 shows the microstructure of the as cast alloy studied. It illustrates that the particles have different shapes and sizes and randomly distributed.

3.2 Microstructural developments during isothermal heat treatment

Samples were ramp heated and isothermally aged at different temperatures and different holding times then water quenched in order to follow the nucleation and growth of the dispersoid particles. **Fig. 2** shows the transmission electron micrographs illustrating the micro structural change of the water quenched alloy during isothermal ageing at 350°C. Distribution of small dispersoid particles aligned along <100> matrix direction in the water quenched samples aged for 50 and 75 hours at 350°C are observed in **Fig. 2(b)** and **Fig. 2(c)**. These particles were observed losing their distribution along <100> matrix directions at their early stage of precipitation, **Fig. 2(d)**, with an increase in holding time. The observed distribution of dispersoid particles along the same orientation as β' Mg_2Si indicated that there is some link between the nucleation of dispersoids and β' Mg_2Si phase. All our statements are in good agreements with those obtained by H. Farh et al [10], K. Strobel et al [20] and L. Lodgaard [21] who have reported that these particles are acting as nucleation sites for Mg_2Si.

Fig. 3 shows the microstructure of the alloy ramp heated and aged at 450°C for different holding times : (a)15 hours, (b) 48 hours, (c)100 hours and (d)120 hours respectively. The alignment of dispersoid particles in their early stages of formation, **Fig. 3(a)**, and the heterogeneous distribution of dispersoid particles with different shapes after an increase in holding time can be clearly seen. The size of the Mn-bearing dispersoid particles of type α-Al (Mn, Fe) Si was about 25 to 350 nm. These dispersoid particles exhibited strain field contrast when they are heated at low temperatures, **Fig. 3(a)** and **Fig. 3(b),** indicating that they are coherent or semi-coherent with the matrix. A loss

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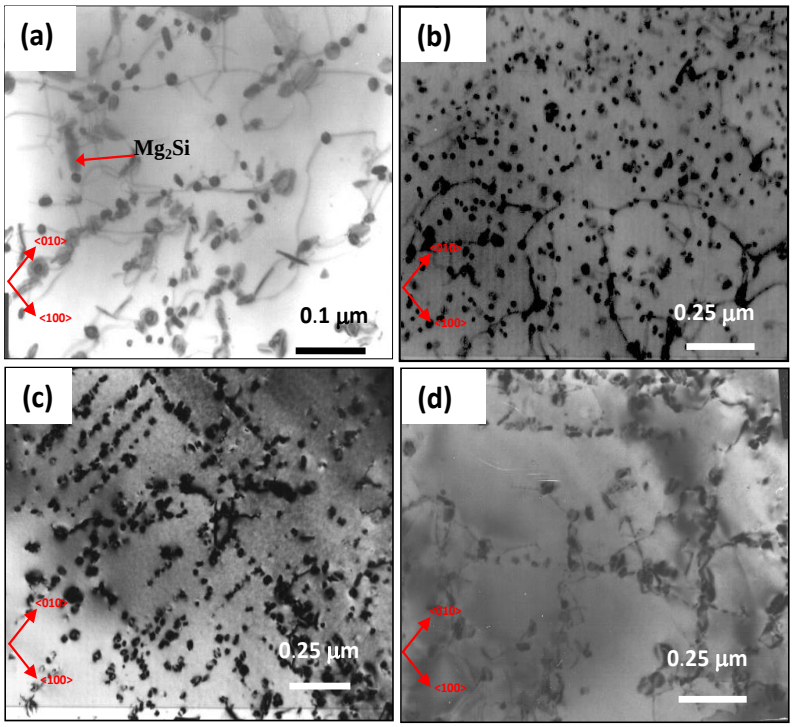

Fig. 2 Transmission electron micrographs of alloy sample ramp heated at 100°C h^{-1} to 350°C and isothermally aged for : **(a)** 14 hours, **(b)** 50 hours , **(c)** 75 and **(d)** 120 hours

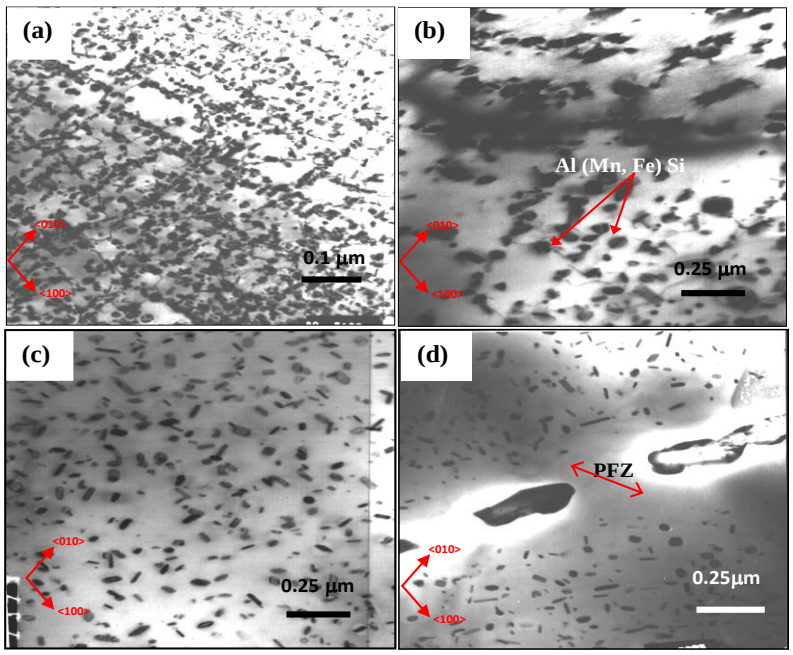

Fig. 3 Transmission electron micrographs of alloy sample ramp heated at 100°C h^{-1} to 450°C and isothermally aged for (a) 15 hours, (b) 48 hours, (c) 100 hours and (d) 120 hours

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of coherency was observed for most of the dispersoid particles when they were aged at high temperature and holding times, **Fig. 3(c)**. **Fig. 3(d)** illustrates PFZ′s (Precipitate Free Zones) and coarse second phase particles lying on the grain boundary and dispersoid particles inside the grain the precipitates are heterogeneously distributed and have an indistinct morphology. Most of them have a needle-like morphology, **Fig. 3(c)**. Some of the coarse particles on the grain boundaries that were formed during casting remain after these heat treatments. These results are in accordance with those obtained by H. Farh et al [1], L. Lodgaard [21] and R. Hu et al [22] who have investigate the nucleation mechanism, the precipitation behavior and the formation of dispersoid particles in Al-Mg-Si-Mn alloys. In our case EDX (Energy Dispersive X-ray) microanalysis revealed that the Dispersoid particles contain mainly Al, Mn, Fe and Si, **Fig. 4**.

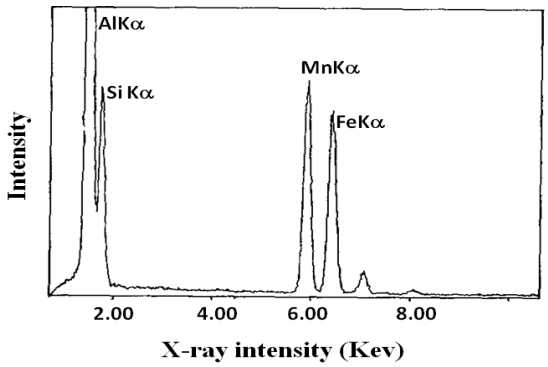

Fig. 4 X-ray spectrum of a dispersed particles observed in alloy sample ramp heated at 100°C h^{-1} to 450°C and water quenched. The main elements are: Al, Si, Mn and Fe

3.3 Treatment costs

To reduce heat processing costs, we must seek the optimum time for each specific cycle to the heat treatment total cycle. It was noted that the time to isothermal ageing process may be substantially reduced and this because the properties remain unchanged for long time intervals of treatment. The work can be performed to generate data that can be used to determine the shortest (optimum) for each processing cycle property and at all levels.

Table 2 Electric energy consumption in solution heat treatment [23]

Ageing process	Electric energy consumption (kWh/t)
4 h at 150°C	658.7
4 h at 160°C	678.8
4 h at 180°C	720.6
7 h at 150°C	1019.5
7 h at 160°C	1048.7
7 h at 180°C	1108.4
7h at 200°C	1170.3
15h at 200°C	2252.1
30 h at 200°C	4280.4

Electric energy consumption can be reduced by finding optimum time for each heat treatment cycle. **Table 2** gives electric energy consumption in kWh/t for many ranges of temperatures and times.

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4 Conclusions

Based on the analysis of experimental test results presented in this paper, it can be concluded that:

1. By increasing ageing temperature and/or holding time we have observed dispersoid particles losing their initial distribution along <100> matrix direction and also their coherency for most of them with the matrix.
2. The corresponding TEM microstructures illustrate the GP zone (needle-shaped precipitates) that are characteristic of:
   - The under ageing structure with very small needles, ~ 0.01μm in length, along <100> matrix directions **Fig. 2(a)**.
   - The peak ageing structure with slightly larger needles **Fig. 3(c)** which can indicate the peak hardness.
   - Finally the over ageing structure with further coarsened needles **Fig. 3(d)**.
3. Combination of heating , the temperature to which the alloy is heated , the holding time at this temperature and the quenching rate define the properties of the obtained material
4. A very long time of isothermal heat treatment process gives excellent results but it is certainly not economically suitable because of a large consumption of electrical energy. In this case the search for optimal treatment times is necessary.

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