EFFECT OF THE AGEING TEMPERATURE, AGEING TIME AND QUENCHING TEMPERATURE ON PRECIPITATION KINETICS IN Al-Mg-Si ALLOY

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Abstract
In the present paper, we have studied the effect of ageing temperature, ageing time and quenching temperature on structural, microstructure and mechanical properties of Al-Mg-Si alloy. The ageing temperature used in this work was 300 °C pending time 24 and 48 h. The structural properties were investigated using X-ray diffraction; the microstructural evolution was investigated using optical, scanning and transmission electron microscopies and microhardness measurement for the mechanical properties. After various states of ageing, the Al-Mg-Si alloy shows significant changes in the microstructure and microhardness values. After ageing, the microstructure of the matrix consisted of a two solid solution of $\alpha$-Al and $\beta$-Mg$_2$Si phases precipitation. After two-step heat treatment (quenching and ageing), the microhardness value were decreased with comparing original sample, the alloy reveals high $\beta$ discontinuous precipitates. After ageing at 300 °C of original sample, corresponding to the minimum value of microhardness, the volume fraction of the continuous precipitate becomes smaller. We found be that the best results have been obtained with the ageing at 300 °C pendinf time 48 hours in all steps.

Keywords: Al-Mg-Si alloy, Ageing temperature, Phases precipitates, Microhardness

1 Introduction
The 6000 series Al-Mg-Si alloys have a widespread application, especially in the building, aircraft and automotive industry due to their excellent properties. The 6000 alloys contain magnesium and silicon as major addition elements. They have good extrudability and hardening characteristics as well as excellent corrosion, surface, and welding properties. In this aluminium alloys besides the intentional additions, transition metals such as Fe, Mg and Cu are always present. Even not large amount of these impurities causes the formation of a new phase component. The exact composition of the alloy and the casting condition will directly influence the selection and volume fraction of intermetallic phases [1–4].

The precipitation sequence in Al-Mg-Si1–4 alloys is atomic clusters $\rightarrow$GP. Zones $\rightarrow$ metastable $\beta''$ precipitates $\rightarrow$ metastable $\beta'$ precipitates $\rightarrow$ stable $\beta$ phase [5–8]. This precipitation sequence was observed for most investigated Al-Mg-Si alloys within a relatively wide temperature range. Previous investigation indicated that equilibrium Si phase is formed at the end of the precipitation sequence if the atomic ratio of Mg to Si is less than 2, and this phase can nucleate...
along the stable $\beta$ particles \[9,10\]. However, Except for the equilibrium phase $\beta$, with composition Mg2Si, and Si, all phases are metastable. Historically, all the precipitates were assumed to have the Mg2Si composition.

The heat treatment of a metal part is to subject it to structural changes through predetermined cycles of heating and cooling of metal and their alloys in the solid state for the purpose of changing their structure and consequently their properties. Oladele and Omotoyinbo \[11\] they defined the common heat treatment operation for aluminum alloys is called ageing. This is the method of precipitating from solid solution a metastable phase that leads to change in properties of the alloy \[11\].

In this paper, we have studied the effect of ageing temperature, ageing time and quenching temperature on structural, microstructure and mechanical properties of Al-Mg-Si alloy. The homogenization of the Al-Mg-Si alloy was performed at 400 °C for 2 hours and quenched in water. However, The ageing temperature used in this work was 300 °C pending time 24 and 48 h.

2 Experimental material and conditions

The material used in this investigation was an Al-Mg-Si (6000) aluminium alloy. Its chemical composition is listed in Table 1. Samples in the form of bars having a diameter of dimensions of (1×1×1) cm$^3$ in size for Al 6000 alloy were used for XRD, SEM and the microhardness measurements. The solution heat treatment program used for the samples is shown in Table 2. Five different samples were investigated with temperature treatment. The first corresponds to original sample (A). The samples (B and C) were aged at 300 °C for 24 and 48 hours of quenched Al-Mg-Si alloy at 400 °C for 2 hours, respectively. Finally, in the last process way samples (D and E) an ageing at 300 °C for 24 and 48 hours of original sample, respectively (see Table 2).

Crystallographic and phase structures of the thin films were determined by X-ray diffraction (XRD, Bruker AXS-8D) with CuK$\alpha$ radiation ($\lambda = 0.15406$ nm) in the scanning range was between $\theta = 10^\circ$ and $90^\circ$. For microscopic studies, specimens were polished and etched with a concentrated solution of 10 % NaOH in the water at room temperature during (30 –120 s). A scanning electron microscopy (SEM, JSM–6700F) equipped with EDX was used to examine both morphology and elemental analysis of the samples, and the microhardness variation was measured on samples using HVM-2000 hardness, at a load of 125 g applied for 30 seconds.

<table>
<thead>
<tr>
<th>Alloy (wt. %)</th>
<th>Al</th>
<th>Si</th>
<th>Mg</th>
<th>Fe</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>98.5</td>
<td>0.59</td>
<td>0.564</td>
<td>0.184</td>
<td>0.015</td>
</tr>
</tbody>
</table>

| Table 1 The chemical composition of aluminum 6000 alloy, values are in wt. %.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Solution heat treatment temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>400 quenched</td>
</tr>
<tr>
<td>A</td>
<td>–</td>
</tr>
<tr>
<td>B</td>
<td>2 h</td>
</tr>
<tr>
<td>C</td>
<td>2 h</td>
</tr>
<tr>
<td>D</td>
<td>–</td>
</tr>
<tr>
<td>E</td>
<td>–</td>
</tr>
</tbody>
</table>

Table 2 Durations (h) and temperatures (°C) of the quenching and ageing treatments
Results and discussion

The XRD spectrum of an original sample of Al 6000 alloy, shown in Fig. 1, presents diffraction peaks representing two solid solutions: the \( \alpha \) (Al-rich) solid solution and apparently \( \beta \) (Mg-Si-rich) solid solution, as confirmed in Fig. 2b. According to previous characterizations of Al-Mg-Si [5,6,9], these peaks correspond to \( \alpha \)-Al and \( \beta \)-Mg-Si phases precipitation.

The nature of the precipitates that formed in the matrix as fine and coarsened particles was examined by EDS analyses. In Fig. 2a, the precipitates are identified as particle 1 and particle 2 in the pre-solution treated alloy [12]. The average grain intercept length in the solutionised condition was found to be \( \sim 4 \) μm. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) analysis showed that the matrix consisted of a two solid solution of Al, Si and Mg, whereas both particles 1 and 2 consist of Al, Fe and Cu as given in Fig. 2b.

The Fig. 3 shows the X-ray diffraction of aged at 300 °C as quenching of Al alloy 6000 at 400 °C. The ageing was obtained at two different times (24 and 48 h). As can be seen in Fig. 3, five diffraction peaks were observed at \( 2\theta = 38.5^\circ, 44.7^\circ, 65.1^\circ, 78.1^\circ \) and \( 82.3^\circ \) which can be attributed respectively to (111), (200), (220), (311) planes of \( \alpha \) phase and (222) plane of \( \beta \) phase, respectively. As can seen that the variation of peaks intensity change in the \( \alpha \) phase and \( \beta \) phase.

It is clear that both regions have the same crystalline structure but different lattice parameter. It is evident that \( \beta \) phase has a composition different from that of \( \alpha \) phase. It is clear that its intensity becomes \( \beta \) phase precipitated during this treatment (see Fig. 3).
The XRD analysis results of Al 6000 alloy of (a): aged at 300 °C for 24 hours as quenching at 400 °C, (b): aged at 300 °C for 48 hours as quenching at 400 °C.

Fig. 4 shows the microstructural evolution of the aluminium 6000 alloy processed of ageing at 300 °C for 48 hours of original sample (Fig. 4a) and ageing at 300 °C for 48 hours of quenched at 400 °C (Fig. 4b). We have observed that the precipitations are continuous and discontinuous of without and with quenching temperature. The ageing at 300 °C of original sample produces a continuous precipitation characterized by finer precipitates inside of grains with their length estimated at 1 μm (Fig. 4a). However, the precipitates are fragmented with their length estimated at 10 μm for ageing at 300 °C of quenched sample (Fig. 4b), it should also be noted that the volume fraction of this precipitate is higher than in the case of the original processed samples was 4 μm (see Fig. 2a). This is due to the partial dissolution and transformation of the particles as a result of shearing. A similar observation was reported for the 6082 alloys in [13]. By contrast, the results reported in [14–16] show only partial fragmentation of the β’ precipitates.

Fig. 4 Microstructures observations of Al6000 alloy with aged at 300 °C for 48 hours: (a): ageing at 300 °C of original and (b): ageing at 300 °C of quenched at 400 °C 2 hours.

The microhardness variation curves of Al alloy 6000 at various treatments such as original sample, the quenching samples at a temperature of 400 °C during 2 hours, the ageing at 300 °C for 24 and 48 hours of quenched Al alloy 6000 and the ageing at 300 °C of original sample are presented in Fig. 5. As can be seen, the microhardness increases after the quenching alloys. The alloy becomes soft with prolonging of ageing and its mechanical properties are reduced by the appearance of equilibrium precipitate β. It has been found that a discontinuous precipitation can cause major changes in the microstructure and properties of solid alloys.
Conclusions
In summary, the ageing effect at 300 °C pending time 24 and 48 h before and after quenching at a temperature of 400 °C of Al-Mg-Si alloy on the structural, microstructural and mechanical properties were investigated. We found that the Al-Mg-Si alloy shows significant changes in the microstructure and microhardness values. The microstructure observations after ageing shows that the matrix consisted of a two solid solution of $\alpha$–Al and $\beta$–Mg$_2$Si phases precipitation. With two-steps of heat treatment (quenching and ageing), the microhardness value found decreases with comparing original sample, showing that the alloy reveals high $\beta$ discontinuous precipitates. After ageing at 300 °C of original sample, corresponding to the minimum value of microhardness, the volume fraction of the continuous precipitate becomes smaller. On the other hand, the TTT curves for the discontinuous and continuous precipitation reaction in this alloy have been suggested. The best results were obtained with the ageing at 300 °C pending time 48 hours in all steps.

References

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