Effect of interrupted ageing treatment on the mechanical properties and intergranular corrosion behavior of Al-Mg-Si alloys

Xuehong Xu\textsuperscript{a,b}, Yunlai Deng\textsuperscript{b,c}, Shuiqing Chi\textsuperscript{c}, Xiaobin Guo\textsuperscript{c,*}

\textsuperscript{a} Light Alloy Research Institute, Central South University, Changsha 410083, PR China
\textsuperscript{b} State Key Laboratory of High Performance Complex Manufacturing, Central South University, Changsha 410083, PR China
\textsuperscript{c} School of Materials Science and Engineering, Central South University, Changsha 410083, PR China

Abstract

In this study, the influence of interrupted ageing treatment on the mechanical properties and intergranular corrosion (IGC) behavior of Al-Mg-Si alloy was investigated. The results show that mechanical properties (strength and elongation) and IGC resistance of the interrupted ageing T6I6 alloys are enhanced simultaneously as compared to the T6 alloys. After treated by the interrupted ageing T6I6, the microstructure of the studied alloy consists of small size and high quantity density $\beta''$ precipitated phase in the Al matrix, discontinuously distributed grain boundary precipitates (GBPs) and narrow precipitation free zones (PFZs). The increment of mechanical properties and the improvement of IGC resistance are attributed to the type microstructures in the Al-Mg-Si alloy.

© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Age-hardenable 6000 series aluminum alloys are being used more and more widely, such as in transportation as a result of the light weight, moderate strength and good forming ability [1–4]. In the ageing treatment process, the needle semi-coherent precipitation phase $\beta''$ in aluminum matrix can effectively prevent the movement of dislocation, so the hardness of Al-Mg-Si alloy will increase [5,6]. The formation of $\beta''$ precipitates mainly depends on the chemical composition of alloys and heat treatment system. And large numbers of researches have been carried out. Previous research [7] found that excessive Si can accelerate ageing hardening rate and increase the peak hardness, while excessive Mg may coarsen the second phase as a result of more dissolved Mg, reducing the strength of AA6101 alloy. Due to lower Cu addition resulting in finer precipitate distribution, the strength and extensibility of the 6xxx alloys are obviously increased [8,9]. Ding et al. [10] found that the introduction of pre-strain before ageing can obviously suppress the bad influence of natural ageing and enhance the hardness of paint baking ageing of 6xxx Al alloys. In recent years, the effect of Zn on Al-Mg-Si alloys has attracted extensive attention, mainly because Zn can play an excellent role in the properties of 6xxx Al alloys [11–14]. Zn can improve the peak ageing hardness of the Al-
Mg-Si alloys ascribed to the increment of the solution clusters at the early ageing stage caused by the addition of Zn [11]. Guo et al. [13] found that the hardness of bake hardening ageing could be improved by adding Zn in Cu-added Al-Mg-Si alloys treated by pre-ageing.

A lot of work [15–26] has been performed to research the corrosion properties of Cu-added Al-Mg-Si alloys. In NaCl solution, the pitting corrosion develops in the Al matrix and produces the corrosion products. With the increment of corrosion products and the increase of chloride ion concentration, pitting corrosion grow up and develop into intergranular corrosion [15]. Bhattamishra et al. [16] found that because Si tends to precipitate along the grain boundary, the intergranular corrosion resistance (IGC) of 6xxx Al alloys is reduced. The corrosion susceptibility of Al-Mg-Si alloys increases with the addition of Cu element [17–20]. Svenningsen et al. [21] have studied the influence of thermal treatment route on the IGC behavior of Al-Mg-Si(Cu) alloys. In the work of Wang [22], it is found that the susceptibility to IGC of Cu-added Al-Mg-Si alloys without strength reduction can be reduced with a two-stage ageing treatment. Zhang et al. [24] found that the air-quenched alloy exhibits better IGC resistance than the water-quenched alloy during immersion in sodium solution. It is claimed that when the Zn content reaches 1%, the IGC of the alloy occurs due to the segregation of Zn on the grain boundary [26].

Buha et al. [27–31] found compared to the traditional T6 treatment, an interrupted ageing process called T616 treatment can enhance the mechanical properties of 6xxx Al alloys. This so-called interrupted ageing treatment is as follows: T6 ageing treatment is performed for a short time by quenching the alloy in cold water (room temperature), followed by the long time ageing treatment at lower temperature (25–65 °C) and quenching the alloy in cold water, and then the T6 treatment is continued until the peak ageing is reached [32,33]. Researches [27–33] reported that this interrupted ageing treatment could enhance the mechanical properties for 2xxx, 6xxx and 7xxx Al alloys. However, the influence of interrupted ageing on the corrosion properties of 6xxx Al alloys has barely been investigated.

In this paper, the effect of interrupted ageing T616 process on the mechanical properties and IGC properties of Al-Mg-

| Table 1 – The chemical composition of the studied alloys (wt. %). |
|----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Mg  | Si  | Fe  | Mn  | Cu  | Cr  | Ti  | Zn  | Al  |
| 0.78 | 0.92 | 0.21 | 0.52 | 0.01 | 0.06 | 0.02 | 0.02 | Bal. |

![Fig. 1 – Schematic diagrams of heat treatment route of Al-Mg-Si alloys (a) T6; (b) T616.](image)

Si alloy was investigated. The performance testing methods in this article include the Brinell hardness measurement, tensile tests, intergranular corrosion test and electrochemical measurement. The surface characteristic of the corrosion samples was studied by scanning electron microscopy (SEM), and the microstructure of the studied alloys was researched with transmission electron microscopy (TEM).

### 2. Experiments

The chemical composition of the Al-Mg-Si alloys used in the paper was displayed in Table 1. The ingots were homogenized in an electrical resistance furnace for 16 h at 550 °C to obtain uniform microstructures. The ingots were then kept at 500 °C for 2 h and hot rolled immediately to the thickness of 4 mm. The solution treatment system of the hot rolled sheets was at 550 °C for 1 h and then rapidly quenched in cold water. The ageing heat treatment of all samples were immediately conducted in a FDT500 electrical resistance furnace as follows: (a) T6 treatment at 170 °C for different times; (b) For the T616 treatment, after ageing treatment at 170 °C for 20 min, the alloy is quenched in cold water and then is dwelled at 65 °C for two weeks, followed by continuing ageing at 170 °C. Artificial ageing at 170 °C for different time and ageing at 65 °C were performed in electric resistance furnace, and subsequently quenched in cold water. Schematic diagrams of heat treatment route are displayed in Fig. 1.

The hardness was measured by HBS-62.5 Brinell hardness tester. The loading force is 10 kg, the loading time is 15 s, and the average value is obtained from the 5 test points. The tensile samples were machined in the rolling direction of the sheet. The tensile test was conducted on CSS-44100 test machine at room temperature, and the tensile rate was 2 mm/min. Three samples per set were carried out to ensure the repeatability of the obtained data. After the tensile test, the fracture morphology of the specimens was characterized by ZEISS-EVOM10 SEM.

The intergranular corrosion test (IGC test) was conducted by immersing in an aqueous chloride-peroxide solution which is based on ASTM G110 [34]. Before the IGC test, all the sam-
immersed dried.

Then samples were etched for 1 min with the solution of 50 ml HNO₃ (70%) and 5 ml HF (48%) in 945 ml of deionized water. The etched samples were cleaned using deionized water and air-dried. Then the specimens were submerged into a solution of 5.7% NaCl and 10 ml/L H₂O₂ (30%) at about 30 °C. After being immersed for 12 h, the corroded specimens were cut perpendicular to rolling direction and used to observe the corrosion depth by metallographic examination method as shown in Fig. 2. Then the specimens used for metallographic examination was also conducted by ZEISS-EVOM10 scanning electron microscopy.

The polarization curves of T6 and T616 alloys were tested by MAC90046 system. The test solution of polarization curves was 3.5% NaCl solution. The potential range in the polarization curves test was from −1.5 V to 0.2 V, and the scan rate was 1 mV/s. After polarization curve measurement, the specimens were removed from the solution, cleared using deionized water and air-dried, preparing for characterizing by SEM.

TEM microstructures were examined with a Tecnai G2 F20 and the instrument was operated at 200 kV. The electrolytic polishing was used to prepare thin foils for TEM, and the related solution was 80 vol.% methanol and 20 vol.% nitric acid, the temperature and voltage of electropolishing are −25 °C and 2025 V, respectively.

### 3. Results

#### 3.1. Mechanical properties

Age hardening curves of the studied alloys treated by T6 and T616 treatment were shown in Fig. 3. It can be found that the hardness of T6 and T616 alloys increases rapidly from 52 to 71 HBW after being aged at 170 °C for 20 min. Then the hardness of the T6 treated sample increases gradually until reaches to the peak hardness. For the T616 treated alloy, the hardness increased rapidly from 71 to 91 HBW after being secondary aged at 65 °C for 2 weeks, and then the hardness also increases gradually until the peak-aged condition is achieved. Both the T6 and T616 samples reached the peak hardness at 170 °C for 8 h, and the values is 109 and 118 HBW of T6 and T616 samples, respectively. Obviously, the T616 sample gets higher peak ageing hardness, hinting the interrupted ageing T616 treatment increases the age hardening behavior of Al-Mg-Si alloy.

The tensile test results of T6 and T616 samples were shown in Fig. 4. The results displayed that the ultimate tensile...
strength (UTS), yield strength (YS) and elongation (El) of the T6 sample at peak ageing are 325 MPa, 269 MPa and 19.9%, respectively. While the T6I6 sample at peak ageing has higher strength and better ductility with the related mechanical property parameters of UTS, YS and El are 349 MPa, 289 MPa and 23.6%, respectively. As compared with T6 sample, the UTS, YS and El of T6I6 treated sample increased by 24 MPa, 20 MPa and 19%, respectively, which indicated that the mechanical properties of the studied alloy treated with interrupted ageing process were better than conventional T6 treatment.

Fig. 5 shows the fracture morphology of the aged alloy at peak ageing. The results show that there are numerous dimples of different depths and morphology in both T6 and T6I6 samples. The fracture surface is regarded as ductile fracture, and the more and deeper dimples are, the better the fracture toughness is. Therefore, both T6 and T6I6 samples have higher elongation. On the other hand, as can be seen clearly from the SEM pictures, the dimples in the T6I6 sample are more and deeper than that in the T6 sample. The dimple area fraction of the fracture morphology in the alloys at peak ageing using ImageJ software is displayed in Table 2. The area fraction of the transgranular fracture ($A_{\text{T}}$) shows that the T6I6 sample (as shown in Fig. 5b) has the bigger $A_{\text{T}}$ than the T6 sample (as shown in Fig. 5a). It demonstrates that the interrupted ageing T6I6 has a favorable influence on the improvement of the plasticity of Al-Mg-Si alloy. This is accordance with the tensile results.

Fig. 5 – Fracture surfaces of the aged alloy at the peak ageing: (a) T6; (b) T6I6.

Fig. 6 – The cross-sectional corrosion morphologies of the specimens: (a) T6; (b) T6I6.

3.2. The intergranular corrosion behaviour

Fig. 6 shows the optical micrographs of cross-sectional corrosion morphologies in the T6 and T6I6 samples. The intergranular corrosion test results in the peak aged states reveal that both the T6 ageing and interrupted ageing T6I6 samples display IGC susceptibility, but shown local corrosion as
shown in Fig. 6a and b. In contrast, pitting corrosion and intergranular corrosion are developed in the T616 sample (Fig. 6b).

Additionally, the maximum depth of corrosion in the T6 sample (Fig. 6a) and the T616 sample (Fig. 6b) are 109.18 μm and 77.32 μm, respectively, indicating that the T6 sample is more susceptible to IGC. The Corrosion depth distribution of the specimens is shown as Fig. 7. It shows that the T6 specimen is corroded more than the T616 specimen. Besides, there are

Fig. 8 – SEM micrographs of T6 and T616 Al-Mg-Si alloy surfaces after IGC test: (a)-(b) general micrographs; (c)-(d) typical corroded grain boundaries, (e) EDS analysis of particle 1in.(c), (f) EDS analysis of particle 2 in.(c), (e) EDS analysis of particle 1in.(c), (g) EDS analysis of particle 3 in.(c).
several corrosion depths more than 58 µm in T6 specimen, but only one corrosion depth more than 58 µm in T6I6 specimen. The average of the corrosion depths of T6 and T6I6 specimen is 64.58 µm and 41.26 µm, respectively. Therefore, the T6 specimen has the higher IGC susceptibility than T6I6 specimen. In other words, it reveals that, in contrast to the T6 alloy, the resistance to IGC of the Al-Mg-Si alloy treated by the interrupted ageing T6I6 can be improved with the increment both in strength and ductility.

Fig. 8a and b shows general micrographs of the T6 and T6I6 alloys surfaces, elucidating local corrosion on the surfaces of the tested samples. We can observe that the maximum depth of corrosion of the T6 state specimens is deeper than that of T6I6 state samples. Besides, Fig. 8c and d show the corroded grain boundaries of the red dot areas in Fig. 8a and b at increased magnifications. As displayed in Fig. 8a and b, the corroded grain display narrow and dark lines on the alloy surfaces, implying anodic dissolution of alloy matrix [35]. Additionally, the particles at the corroded grain boundaries in Fig. 8c are shown in Fig. 8e–g based on the EDS analysis. From the EDS analysis, we can observe that the white particles (point 1 in Fig. 8c) on corroded grain boundaries in Fig. 8e consist of Al, Si, Mn and Fe, indicating the white particles are Al(FeMn)Si phases. As to the black near the crack (point 2 in Fig. 8c) shown in Fig. 5f, the signals of Al, Mg and Si are detected, demonstrating the black particles are Mg-Si precipitated phases. The matrix particle (point 3 in Fig. 8c) shown in Fig. 8g contains Al and Mg elements. The type of element of the corresponding 1, 2 and 3 particles in Fig. 8d is the same as that in Fig. 8c, although the element content is slightly different, so the EDS diagrams is not shown here. It hints that IGC is caused by the combination of Al(FeMn)Si particles and Mg-Si precipitated phases. The effect of Mg-Si precipitated phases on the Al alloy has been discussed in detail in the literature of [36].

Table 3 exhibits the area fraction of the precipitate particles of SEM micrographs in the Al-Mg-Si alloy surfaces after 12 h immersion in testing solution at the peak condition by using Image) software. It displays that the T6 sample has the similar area fraction of the precipitate particles with the T6I6 sample, in which the area fraction of the precipitate particles of the T6I6 sample and the T6 sample is 1.74% and 1.89%, respectively.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Size of the second phase particles (µm)</th>
<th>Area fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Length (L)</td>
<td>Interparticle spacing (A)</td>
</tr>
<tr>
<td>T6</td>
<td>3.77</td>
<td>14.20</td>
</tr>
<tr>
<td>T6I6</td>
<td>4.56</td>
<td>15.12</td>
</tr>
</tbody>
</table>

Fig. 9 – The potentiodynamic polarization curves in 3.5% NaCl solution for T6 and T6I6 alloys.

Table 4 – Electrochemical parameters of the aged alloys tested in 3.5% NaCl solution.

<table>
<thead>
<tr>
<th>Ageing temper</th>
<th>Icorr (A/cm²)</th>
<th>Ecorr (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T6</td>
<td>3.535 x 10⁻⁷</td>
<td>−0.7618</td>
</tr>
<tr>
<td>T6I6</td>
<td>1.135 x 10⁻⁷</td>
<td>−0.7190</td>
</tr>
</tbody>
</table>

3.3. The electrochemical corrosion behavior

The polarization curves of T6 and T6I6 alloys in 3.5% NaCl solution are displayed in Fig. 9, and the corresponding parameters derived from polarization curves are displayed in Table 4. One breakdown potential about −0.7798 V is observed on the polarization curve of the T6 sample. Meanwhile, there is also one breakdown potential about −0.7278 V on the polarization curve of T6I6 sample. The breakdown potential relates to the dissolution of the passive film on the alloys surface. Corrosion will occur when the passivation film dissolves. Additionally, the corrosion potential (Ecorr) suggests the degree of electrochemical corrosion of the material, and the more negative Ecorr is, the more serious the corrosion is. The corrosion current density (Icorr) reveals the corrosion rate, and the greater Icorr is, the faster the corrosion is. The electrochemical analysis results (Table 4) show that the Ecorr and Icorr of T6 sample are −0.7618 V and 3.535 x 10⁻⁷ A/cm², respectively, and the corresponding value of T6I6 sample are −0.7190 V and 1.135 x 10⁻⁷ A/cm², respectively. It reveals the T6 sample exhibits lower Ecorr and higher Icorr than T6I6 sample, implying that the T6 sample is more susceptible to corrosion than T6I6 sample. In other words, the resistance to corrosion of the studied Al alloy is improved by interrupted ageing T6I6. These results are consistent with the intergranular corrosion results.

Fig. 10 shows the SEM micrographs of T6 and T6I6 specimens after the electrochemical corrosion tests in a 3.5% NaCl solution. It is clear that the surface of T6 sample is almost completely corroded. In contrast, the corrosion on the T6I6 sample surface is slighter and the corrosion is observed only on a few surfaces of the sample.
Fig. 10 – SEM micrographs of T6 (a and c) and T6I6 (b and d) after electrochemical corrosion test of the alloys: (a)-(b) general view; (c)-(d) SEM observation of a pit; (e) EDS analysis of particle 1 in. (c), (f) EDS analysis of particle 2 in. (d).

The SEM observations of the red dot areas in Fig. 10a and b at increased magnifications are exhibited in Fig. 10c and d, respectively. The corrosion propagation not only develops under the passive film but also spreads in depth. X-ray energy dispersive spectroscopy (EDS) analysis was carried out on the un-corroded surface and the residuum of the corroded pits as displayed in Fig. 10e and f. As is shown in Fig. 10e and f, after completing the electrochemical corrosion test, the chemical composition of the un-corroded matrix (point 1 in Fig. 10c) in Fig. 10e are composed of Al, Mg and Si. However, only Al and Mn element are determined and few Mg and Si is detected on the residuum of the corroded pits (point 2 in Fig. 10c) in Fig. 10f, revealing that most or all of the Mg-Si precipitates and Al(FeMn)Si phases in the corroded matrix have been etched out. As mentioned above, the corrosion of the T6 sample is more serious that of T6I6 sample in a 3.5% NaCl solution, suggesting the higher corrosion susceptibility of T6 sample. That is, the resistance to corrosion of studied Al alloy is increased with interrupted ageing T6I6 treatment, which is agreed with the IGC results.

3.4. Microstructure

The precipitation behavior in the grains and on grain boundaries is studied so as to expose the age strengthening response and the IGC mechanism of the T6 and T6I6 samples. Fig. 11 shows the TEM micrographs of T6 and T6I6 alloys. The images were all obtained in the <001> zone axis of the Al matrix, which can be verified by the selected area electron diffraction (SADP) images. For T6 alloy, after peak ageing at 170 °C, both spherical and needle-like precipitated phases are formed in the alloys (Fig. 11a). Most of the precipitated phases are considered to be β′′ phase. Both the GP zones and β′ precipitated phases play an important role in strengthening, but semi-coherent and needle-shaped β′ precipitates are considered to be more effective in preventing dislocation movement than the fully
coherent and spherical GP zones. As compared to T6 alloy, the precipitates in the T6I6 alloy are smaller in size and greater in number by consuming more solution atoms from the Al matrix (Fig. 11b), and therefore the particles is more densely dispersed and the distance between particles is smaller [28].

The quantitative TEM measurements of \(\beta''\) precipitates in the Al matrix of the alloys at peak ageing using ImageJ software are displayed in Fig. 12 and Table 5. From the length distribution of \(\beta''\) precipitates in the alloys (as shown in Fig. 12), it is clear that the number fraction of \(\beta''\) precipitates with length 10–20 nm and 20–40 nm in the T6 sample is about 0.203 and 0.602, respectively, and the number fraction of \(\beta''\) precipitates with length 10–20 nm and 20–40 nm in the T6I6 sample is around 0.584 and 0.404, respectively. That is, almost all (about 0.988) \(\beta''\) precipitates are less than 40 nm in length. However, for the T6 sample, the number fraction of \(\beta''\) precipitates with

| Table 5 – Quantitative TEM measurement of \(\beta''\) precipitated phases in the Al matrix of the alloys at peak ageing. |
|---|---|---|---|
| Alloys | Length (nm) Max | Min | Average |
| | Cross section (nm²) |  |
| T6 | 131.7 | 10.4 | 32.5 | 16.0 |
| T6I6 | 62.1 | 7.122 | 19.8 | 9.8 |

Fig. 11 – TEM micrographs of the precipitated phases in the Al matrix of the specimens at peak ageing: (a) T6, (b) T6I6; grain boundary precipitation of the specimens at peak ageing: (c) T6, (d) T6I6.

Fig. 12 – The length distribution of \(\beta''\) precipitates in two peak aged alloys.
length less than 40 nm only is 0.805 and length greater than 40 nm accounts for about 0.195. In addition, Table 5 shows the maximum length of $\beta^\prime$ precipitates in the T6 and T6I6 samples is around 131.7 nm and 62.1 nm, respectively, and the average length of $\beta^\prime$ precipitates in the T6 and T6I6 samples is about 32.5 nm and 19.8 nm, respectively. Furthermore, the T6 sample has bigger cross area of $\beta^\prime$ precipitates than the interrupted ageing T6I6 sample, in which the cross areas of the T6 and T6I6 samples are 16.0 nm$^2$ and 9.8 nm$^2$, respectively. Therefore, $\beta^\prime$ precipitates in the Al matrix in the T6I6 sample are smaller in size and more evenly distributed compared to the T6 sample.

GP zones are regarded as the precursors of $\beta^\prime$ precipitates in 6xxx Al alloy. During ageing at $170^\circ$ for 20 min, a great quantity of small clusters including Si-enriched, Mg-enriched and Mg-Si co-clusters are formed. At lower temperatures ($65^\circ$), most of these clusters gradually become stable GP zones by consuming the solution atoms from the Al matrix. The more stable the GP region is, the easier it is to transform into $\beta^\prime$ phase during the higher temperature ageing ($170^\circ$) [28,29]. The number of GP zones is more than that formed during T6 treatment, and then they act as precursors to nucleate and grow to $\beta^\prime$ precipitates when ageing at higher temperature $170^\circ$. Besides, due to the dwell effect at low temperature ($65^\circ$) of T6I6 sample, the growth of $\beta^\prime$ precipitates is delayed so that the precipitated phase particles are smaller. In this way, the precipitates in the T6I6 alloy are smaller in size and greater in number. This is responsible for the increment in the strength.

The size of precipitated phases along the grain boundaries is larger than those in the grains. For the T6 sample, the distribution of the second phases on the grain boundaries is discontinuous (Fig. 11c), and the width of the precipitation free zone (PFZ) is 160 nm. The precipitations on the grain boundaries of the T6I6 sample are smaller and also distributed discontinuously, and the width of the precipitation free zone (PFZ) is 120 nm (Fig. 11d). The electrochemical micro couples between the matrix, precipitation free zones and grain boundary precipitates (GPBs) are the main cause of the IGC susceptibility of the 6xxx Al alloy [22]. As compared with the T6 sample, firstly, the size of the precipitates is smaller but the number density is within grains in the T6I6 alloy, resulting in smaller difference of corrosion potential between the matrix and PFZs. Secondly, the smaller precipitates along the grain boundaries makes an increase in the inter-particle spacing between precipitates, leading to the slower propagation. Furthermore, the PFZs of the T6I6 alloy are narrower, so the channel for anodic dissolution is narrower, which makes the corrosion propagation slower. In short, the resistance to IGC of studied Al alloy is improved by T6I6 ageing.

4. Discussion

The strength of 6xxx Al alloy is influenced by various parameters of the precipitated phases, such as the morphology, type, distribution, density and size [30]. The TEM results (Fig. 11a and b) showed that the type of precipitates of the peak-aged T6 and T6I6 samples do not change, but the density of precipitates are greatly increased and the size are greatly reduced by interrupted ageing. During ageing at $170^\circ$ for 20 min, a lot of fine clusters including Si-enriched, Mg-enriched and Mg-Si co-clusters are formed. At lower temperatures ($65^\circ$), most of these clusters gradually become stable GP zones by consuming the solution atoms from the Al matrix. The more stable the GP region is, the easier it is to transform into $\beta^\prime$ phase during the higher temperature ageing ($170^\circ$) [28,29]. The number of GP zones, especially stable GP zones, is more than that formed during T6 ageing, and more GP zones act as precursors to nucleate and grow to $\beta^\prime$ precipitates when re-ageing at $170^\circ$, leading to the increment in number of T6I6 sample. For $<100>_{\text{Al}}^\prime$ needle-like $\beta^\prime$ precipitated phases with diameter D and length L (l>D), the strengthening increment could be expressed as

$$t_p = \frac{G b}{2\pi(1-v)} \left[ \frac{1}{(1.075 \sqrt{\frac{0.4334}{f}} \sqrt{1.732D})} \ln \frac{\sqrt{1.732D}}{r_0} \right]$$

where $t_p$ is the critical resolved shear stress (CRSS) increment attributed to particle strengthening, G the shear modulus of the precipitated phase particles in the Al matrix, b the magnitude of the Burgers vector of the dislocations on the slip planes, v the Poisson’s ratio, f the volume fraction of precipitated phase particles, and $r_0$ the cut-off radius of dislocations. Due to the dwell effect at low temperature ($65^\circ$) of T6I6 sample, the growth of matrix precipitates is delayed so that the precipitated phase particles within the grain are smaller. In this way, the precipitates in the T6I6 alloy are smaller in size and greater in number. According to Eq. (1), the precipitates in the T6I6 sample are more densely dispersed and closely spaced, which is more effective to prevent the dislocation movement. This is considered to be mainly responsible for the improvement in the strength of Al-Mg-Si alloy.

Vrataiva and Cvijovic [37] set up a ductile-fracture model displaying the relationship between the fracture toughness and the volume fraction of the coarse intermetallic (IM) particles. It is assumed that when the opening at the crack tip reaches the width of the intact ligament that separates the cracked particles, the unstable crack continues to propagate, the plane-strain fracture toughness $K_{\text{lc}}$ was calculated for a lot of Al alloys, and the equation was expressed as follows:

$$K_{\text{lc}} = 2 \cdot \sigma_y \cdot E \cdot \frac{2}{3} \cdot \frac{1}{L} \cdot \left( f_0 \cdot \frac{1}{f} \cdot A_{\text{IP}} \cdot \frac{1}{1.5} \right) \cdot \exp \left( \frac{A_{\text{AP}}}{1.5} \cdot \frac{1}{\lambda} \cdot \frac{N \cdot W_{\text{PFZ}}}{\lambda} \right)$$

Where $\sigma_y$ and $E$ are the yield strength and Young’s modulus, respectively. L and $f_0$ are the mean linear size and volume fraction of coarse IM particles, respectively. $\lambda$ is the interparticle spacing of coarse IM particles. $A_{\text{IP}}$ is the area fraction of large voiding, $A_{\text{AT}}$ the area fraction of the transgranular fracture, $W_{\text{PFZ}}$ the width of the precipitation free zones. When the fraction of the fractured particles is low, m is assumed to be 0.3. When the fraction is high, m is equal to 0.5. N is the number of precipitation free zones between coarse IM particles with the distance greater than $\lambda$, and for simplicity, $N=3$. The relevant parameter values are shown in Table 6.

The results show that the T6I6 sample has higher $K_{\text{lc}}$ than the T6 sample, indicating the better plane-strain frac-
ture toughness. Corresponding to the elongation results of the T6 and T616 sample are 19.9% and 23.6%, respectively. From the literature [38], the relationship between toughness $\psi$ and elongation can be expressed as:

$$\psi = 3.848EI$$

Thus, the $\psi$ of the T6 and T616 sample is 76.5752 MJ/m$^3$ and 90.8128 MJ/m$^3$, respectively, implying the better fracture toughness of T616 sample than than the T6 sample, which is in good accordance with the $K_{IC}$.

As shown in Figs. 6 and 7, the T6 sample exhibits higher IGC susceptibility than T616 sample. According to the concept of the electrochemical micro couples, the change in IGC susceptibility of the 6xxx Al alloy must be correspond to the influence of the different heat treatment on the continuity of the electrochemical micro couples at the grain boundaries. Based on the IGC test by the ASTM G110, the corrosion of the T6 sample is IGC corrosion, however, the corrosion of the T616 sample is composed of IGC corrosion and pitting, demonstrating the higher IGC resistance of the T616 sample. This can be further proved by polarization curve test results (as shown in Fig. 9 and Table 4). The corrosion potential ($E_{corr}$) reveals the electrochemical activity of corrosion of the material, and the corrosion current density ($I_{corr}$) suggests the corrosion rate. The electrochemical analysis results (Fig. 9 and Table 4) show that the T6 sample exhibits lower corrosion potential and higher corrosion current density than T616 sample, demonstrating that the T6 sample is more susceptible to intergranular corrosion (IGC) than the T616 sample. In other words, the resistance to corrosion of studied alloy is increased by interrupted ageing T616. As reported, the susceptibility to IGC of the 6xxx Al alloy is responsible for the difference of corrosion potential between the matrix, PFZs and GBPs. Because PFZs are solute-depleted zones, the corrosion potential of PFZs is more negative in contrast to the Al matrix. Besides, the corrosion potential of the Mg-Si particle is more positive than that of PFZs [15,17]. It is reported that dissolution of the PFZs is ascribed to the potential difference between the Al matrix, GBPs, and PFZs. For the studied alloy, the corrosion potentials of the Al matrix in the T6 and T616 sample are different because the amount of Mg-Si precipitates is different, as revealed in Figs. 11 and 12 and Table 5. It is found that the precipitates within the grain in the T616 alloy are smaller in size and greater in number by consuming more solution atoms from the Al matrix [28], resulting in smaller difference of the potential between the matrix and PFZs. Additionally, the smaller precipitated phases on the grain boundaries makes the interspacing bigger (as shown in Fig. 13), leading to the slower propagation. Furthermore, the difference resistance to IGC is also as a result of the difference in PFZs. IGC easily occurred when the PFZs is dissolved in the T6 sample, because the PFZs of T6 sample are wider than that of T616 sample (as shown in Fig. 13). Therefore, the channel for anodic dissolution in the T6 sample is wider, which makes the corrosion propagation quicker. In other words, the IGC resistance of Al-Mg-Si alloy is improved by T616.

### Table 6 - The relevant parameter values in the T6 and T616 samples.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>$\sigma_t$ (MPa)</th>
<th>E (GPa)</th>
<th>L (µm)</th>
<th>$f_v$ (vol %)</th>
<th>$A_{KF}$</th>
<th>$A_{KT}$</th>
<th>$W_{PFZ}$ (µm)</th>
<th>$\lambda$ (µm)</th>
<th>$K_{IC}$ (MPa m$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T6</td>
<td>269</td>
<td>69</td>
<td>3.77</td>
<td>1.89</td>
<td>0.077</td>
<td>0.328</td>
<td>0.16</td>
<td>14.20</td>
<td>33.20</td>
</tr>
<tr>
<td>T616</td>
<td>289</td>
<td>69</td>
<td>4.56</td>
<td>1.74</td>
<td>0.084</td>
<td>0.397</td>
<td>0.12</td>
<td>15.12</td>
<td>39.83</td>
</tr>
</tbody>
</table>

5. Conclusions

In this work, the influence of interrupted ageing on the mechanical properties and IGC behavior of Al-Mg-Si alloy was investigated. The effective conclusions are as follows:

1) The strength, elongation and the IGC resistance of Al-Mg-Si alloy can be enhanced simultaneously by an interrupted ageing treatment, which contains pre-ageing at the conventional T6 ageing temperature (170 °C) for a shorter time (20 min) by followed a long period ageing at the lower temperature (65 °C) for two weeks, and then re-ageing at the same T6 ageing temperature until peak hardness.

2) After the peak ageing, the microstructural characteristics of the interrupted ageing T616 Al-Mg-Si alloy consists of small size and high number density $\beta''$ precipitated phases in the Al.
matrix, which is considered to be directly responsible for the improvement in the strength and elongation.

3) There are two reasons for the increment of IGC resistance of the interrupted ageing T616 Al-Mg-Si alloy: (i) the precipitated phases within the grain in the T616 alloy are smaller in size and greater in number by consuming more solution atoms from the Al matrix, resulting in smaller potential difference between the matrix and PFZs; (ii) due to the smaller and more dispersed precipitates along the grain boundaries and the narrower PFZs, the corrosion propagation becomes slower.

Acknowledgments

This work was supported by the National Key R&D Program of China (project No 2016YFB0300901), and the National Science Foundation of China (Project No. 51705539). The authors would like to take this opportunity to express their appreciation.

REFERENCES


