Microstructure Stability and Creep Strength in a Die-Cast AX52 Magnesium Alloy

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Superior creep strength of a heat resistant AX52 magnesium alloy is ascribed to the grain boundary eutectic Al2Ca phase covering the primary α-Mg grains. The eutectic phase is stable in morphology at temperatures below 473 K, while it collapses during long term exposure at temperatures higher than 473 K. The microstructural change of the alloy during high temperature exposure is characterized by the decrease in the grain boundary coverage by the eutectic phase. The creep strength of the alloy decreases with the decrease in the grain boundary coverage, and the correlation between the creep strength and the grain boundary coverage is discussed.

(Received March 14, 2005; Accepted May 17, 2005; Published August 15, 2005)

Keywords: magnesium alloy, die-cast, creep, microstructure, grain boundary coverage

1. Introduction

The application of lightweight magnesium alloys for the automotive materials has greatly increased in the last decade to improve fuel efficiency through vehicle mass reduction.1–3) The poor creep resistance of the conventional magnesium alloys has hindered the applications for the major powertrain components such as transmission and engine parts, where the operating temperatures can be as high as 450 K.4) Owing to the recent efforts to develop creep resistant magnesium alloys for such applications the cost effective Mg–Al–Ca alloys have been successfully developed.5–9) The compositions of the alloys are located in the range of 4.5–9.0 mass%Al and 0.3–2.3 mass%Ca as reviewed by Luo10) and Pekguleryuz and Kaya.11)

Many of magnesium parts used for the automotive applications are produced by die casting.12) The microstructure of the creep resistant Mg–Al–Ca alloys produced by die casting is usually characterized by the grain boundary eutectic phase covering the primary α-Mg grains.6,7) The field emission scanning electron micrograph of the die-cast AX52 (X representing calcium) alloy obtained in our previous study is shown in Fig. 1.13) The AX52 magnesium alloy is one of the most typical creep resistant Mg–Al–Ca alloys. The superior creep resistance of the AX52 alloy is basically ascribed to the characteristic feature of the eutectic phase, which is expected to resist the plastic flow of the α-Mg grains during the creep deformation.14)

There have been some investigations of the eutectic phases formed in the Mg–Al–Ca die-cast alloys.7,15,16) Our preliminary experiments by the X-ray diffraction and energy dispersive spectrometry indicate that the eutectic phase in the AX52 die-cast alloy shown in Fig. 1 is a C15–Al2Ca phase in the equilibrium state.13) The present work aims two purposes. First, we investigate the stability of the eutectic phase morphology in the AX52 die-cast alloy through the observation of the microstructures of the aged alloys. And second, the correlation between the creep strength and the microstructure is investigated for the AX52 alloy.

2. Experimental

The AX52 alloy with the chemical compositions listed in Table 1 was prepared in a cold chamber die casting machine. The melt temperature was controlled at 993 K with the die temperature maintained at 473 K. The materials were obtained in the form of plates of 150 mm length, 70 mm width and thickness varying in steps from 1 to 3 mm. Specimens for the microstructural observations and creep tests were taken from the 3 mm thickness sections of the castings. The aging treatments were performed in a flow of argon gas at temperatures between 523 and 623 K followed by water quenching.

The microstructure was observed using the field emission scanning electron microscopy (FE-SEM) operated at 5 kV. Specimens for FE-SEM were polished through the standard metallographic procedure and then etched in a solution of 2 ml HNO3 and 98 ml ethyl-alcohol. The tensile creep test was performed under the constant load condition of 80 MPa.
at 473 K on specimens with the gage length of 28 mm and a rectangular cross section of \( 6 \times 3 \) mm. For each specimen, the test temperature was monitored in the two locations of the gage portion to control temperature within 2 K. Tensile displacement was measured using extensometers attached to ridges at both ends of the gage portion, where the displacement of the extensometer heads was continuously recorded by the linear variable differential transformers.

3. Results and Discussion

3.1 Microstructure stability

The FE-SEM micrographs of the AX52 die-cast alloys aged at 573 K for 1 and 10 h are shown in Figs. 2(a) and (b), respectively. The eutectic phase partly collapses at grain boundaries by the aging treatment as shown by arrows in Fig. 2(a). The collapse of the grain boundary eutectic phase becomes marked after longer time exposure at high temperatures as shown in Fig. 2(b), whereas the average size of the \( \alpha \)-Mg grains remains unchanged at around 5 \( \mu \)m. It is concluded that the microstructural change by the aging treatment is characterized by the collapse of the grain boundary eutectic phase, i.e., the decrease in the grain boundary coverage by the eutectic phase.

The grain boundary coverage by the eutectic phase, \( \chi \), for the aged alloys is summarized in the contour map in Fig. 3, where the vertical and horizontal axes are aging temperature and time, respectively. The value of \( \chi \) was determined using the following equation:

\[
\chi = \frac{l_{\text{eut}}}{l_{\text{tot}}}.
\]  

where \( l_{\text{eut}} \) is the grain boundary length covered by the eutectic phase and \( l_{\text{tot}} \) is the total grain boundary length in the magnified FE-SEM micrographs. It is noted that the measurements of \( l_{\text{eut}} \) and \( l_{\text{tot}} \) were performed for more than 30 grains to determine the value of \( \chi \) for each specimen.

The grain boundary coverage of the as die-cast alloy is 0.98 and monotonically decreases as the aging temperature increases and the aging time is lengthened. The value of \( \chi \) is reduced to 0.49 by the 100 h exposure at 573 K, while it is assumed to remain values higher than 0.90 even by the aging treatments for 1000 h at temperatures below 473 K. It is clarified that the eutectic phase of the AX52 die-cast alloy is stable in morphology at temperatures below 473 K.

3.2 Creep strength and microstructure

Creep tests were performed for the AX52 die-cast alloys aged at 573 K to evaluate the effect of the eutectic phase morphology on the creep strength. The creep rate-time curves at 473 K–80 MPa for the alloys aged for 3, 30 and 100 h are shown in Fig. 4 together with the curve for the as die-cast alloy. The creep rate-time curves of the aged alloys exhibit three stages; a transient creep stage, a minimum creep rate stage and finally an accelerating stage, similarly to the as die-cast alloy. It is found that the aging treatment usually results in the increased creep rate and decreased rupture life of the AX52 die-cast alloy. The minimum creep rate and rupture life data for the as die-cast and aged alloys are summarized in Table 2. The rupture life of the alloy aged at 573 K for 100 h is 23 times as short as that of the as die-cast alloy.

The minimum creep rates of the as die-cast and aged alloys are summarized against the grain boundary coverage in Fig. 5. It is obvious that the decrease in the grain boundary coverage usually results in the increased creep rate. A dramatic increase in the creep rate is observed when the value of \( \chi \) decreases from 0.98 to 0.80. On the contrary, the increase in the creep rate with decreasing the grain boundary coverage becomes less pronounced for the values of \( \chi \) below...
The minimum creep rate of the alloy aged at 573 K for 100 h is about eight times of that of the as die-cast alloy. The creep weakening caused by the decrease in the grain boundary coverage by a second phase has been also reported for the thixocast magnesium alloys, nickel-based alloys and austenitic steels.

Why does the decrease in the grain boundary coverage cause the decreased creep strength for the AX52 die-cast alloy? It is well established that the creep rate, \( \dot{\varepsilon} \), for the single phase alloys is inversely proportional to the grain diameter, \( d \), typically smaller than 100 \( \mu \text{m} \) at any creep conditions of temperature and stress where dislocation creep takes place. Note that dislocation climb has been identified as a rate controlling process of the AX52 die-cast alloy. The phenomenological relation between \( \dot{\varepsilon} \) and \( d \) is described as follows:

\[
\dot{\varepsilon} = A d^{-1},
\]

where \( A \) is a constant depending on the alloy composition and creep condition. The equation (2) simultaneously means that the creep rate is proportional to the area of grain boundaries, where dislocation sinks during creep deformation to reduce the dislocation density inside grains.

The decrease in the grain boundary coverage causes the increased grain boundary areas which are not covered by the eutectic phase for the AX52 alloy. The ratio of the minimum creep rate of the aged alloy to that of the as die-cast alloy, \( \varepsilon_{\text{min}} / \varepsilon_{\text{min,0}} \), is plotted against the ratio of the grain boundary areas not covered by the eutectic phase, \( (1 - \chi) / (1 - \chi_0) \), in Fig. 6. Here, \( \varepsilon_{\text{min,0}} \) and \( \chi_0 \) are the minimum creep rate and the grain boundary coverage for the as die-cast alloy, respectively. It is found that the ratio of the minimum creep rate increases linearly with the value of \( (1 - \chi) / (1 - \chi_0) \). The slope of the line is not equal to unity but is very close to 0.6. The underestimation of the creep weakening by the decrease in the grain boundary coverage may be resulted from the enhancement of solid solution strengthening of the primary \( \alpha \text{-Mg} \) grains by the decomposition of the grain boundary eutectic phase during aging treatments. The solute concen-

![Fig. 4 Creep rate-time curves at 473 K–80 MPa for the AX52 die-cast alloys aged at 573 K for 3, 30 and 100 h, together with that for the as die-cast alloy.](image)

![Table 2 Tensile creep data for the AX52 alloys at 473 K under 80 MPa.](table)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Aging condition</th>
<th>Minimum creep rate, ( \varepsilon_{\text{min}} / \varepsilon_{\text{min,0}}, )</th>
<th>Rupture life, ( t_{\text{rup}}/h )</th>
</tr>
</thead>
<tbody>
<tr>
<td>AX52</td>
<td>as die-cast</td>
<td>9.1 \times 10^{-4}</td>
<td>59.0</td>
</tr>
<tr>
<td></td>
<td>573 K/1 h</td>
<td>2.6 \times 10^{-3}</td>
<td>12.9</td>
</tr>
<tr>
<td></td>
<td>573 K/3 h</td>
<td>3.2 \times 10^{-3}</td>
<td>18.4</td>
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<tr>
<td></td>
<td>573 K/10 h</td>
<td>3.8 \times 10^{-3}</td>
<td>10.3</td>
</tr>
<tr>
<td></td>
<td>573 K/30 h</td>
<td>4.5 \times 10^{-3}</td>
<td>6.91</td>
</tr>
<tr>
<td></td>
<td>573 K/100 h</td>
<td>7.1 \times 10^{-3}</td>
<td>2.62</td>
</tr>
</tbody>
</table>

![Fig. 5 Correlation between the minimum creep rate at 473 K–80 MPa and the grain boundary coverage for the AX52 die-cast alloys aged at 573 K, together with the plot for the as die-cast alloy represented by a solid symbol.](image)

![Fig. 6 Correlation between the ratio of the minimum creep rate at 473 K–80 MPa, \( \varepsilon_{\text{min}} / \varepsilon_{\text{min,0}}, \) and that of the grain boundary areas not covered by the eutectic phase, \( (1 - \chi) / (1 - \chi_0) \), for the AX52 die-cast alloys aged at 573 K. It is noted that \( \varepsilon_{\text{min}} \) and \( \chi_0 \) are the minimum creep rate and the grain boundary coverage for the as die-cast alloy.](image)
etration of Al and Ca inside the primary α-Mg grains should be clarified in order to understand the detailed creep strength for the alloy.

4. Conclusions

The stability of the grain boundary eutectic phase morphology and the correlation with the creep strength were investigated for the creep resistant AX52 die-cast alloy. The results are summarized as follows:

(1) The grain boundary eutectic phase is stable in morphology at temperatures below 473 K, while it collapses after longer time exposure at higher temperatures above 473 K. The microstructural change during high temperature exposure is characterized by the decrease in the grain boundary coverage by the eutectic phase.

(2) The creep strength decreases caused by the decrease in the grain boundary coverage by the eutectic phase. The minimum creep rate is about eight times larger when the grain boundary coverage decreases from 0.98 to 0.49.

Acknowledgments

The authors gratefully acknowledge Dr. Koichi Ohori of Mitsubishi Aluminum Co. for providing the die-cast alloy specimens used in this work.

REFERENCES

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