Creep studies of coarse-grained AZ91D magnesium castings

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Abstract

Creep properties of specimens taken from the core of AZ91D magnesium alloy (9% Al–1% Zn) ingots were investigated in the temperature range 120–180°C and stress range 40–115 MPa. Creep tests were performed under constant and varying loads, solution treated creep specimens were also tested. Observed creep rates are about three orders of magnitude lower than those of pure magnesium and elongations to fracture are at least twice those of pure magnesium. No steady-state stage is observed, however a minimum creep rate is reached after approximately two thirds of the creep life of the specimens. The stress exponent is \( n = 11 \) and is independent of temperature and stress. The activation energy decreases with increasing temperature, from 220 to 94 kJ mol\(^{-1}\). Fractography studies show that the fracture is intergranular. © 1998 Elsevier Science S.A. All rights reserved.

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1. Introduction

The fabrication of Mg–Al–Zn alloys began in Germany during the first world war, but these alloys suffered from serious corrosion problems [1]. In 1925 it was found that alloying with minor amounts of Mn reduces this problem substantially. Few alloys were added to the AZ91 group during the years since then; all of them are characterized by almost identical Mg–Al–Zn–Mn chemical composition. For pressure die casting, AZ91A and AZ91B were used, while for sand casting AZ91C was used [2]. Recently, a new group of AZ91 alloys with at least one order of magnitude lower allowable content of impurities was put into service. These new alloys show improved corrosion resistance [3,4]. The AZ91D alloy appears in the ASTM B 93 standard since 1988 as the one for pressure die casting and since 1990, AZ91E appears as the one for sand casting [2]. These two alloys have replaced alloys AZ91A, AZ91B and AZ91C.

The AZ91 alloy, which is the material of this investigation, contains in addition to magnesium three major alloying elements. These elements have the following beneficial effects [5]:

- **Aluminum**—increases room temperature strength and improves the fluidity of the alloy. Increasing the percentage of aluminum increases ductility, however, deteriorates elevated temperature strength.

- **Zinc**—improves room temperature strength through solid solution strengthening. Zinc addition also improves fluidity of the alloy. However, increasing zinc percentage beyond 2% can cause hot cracking.

- **Manganese**—manganese does not affect the mechanical properties, but produces beneficial results in the control of corrosion by forming an Fe–Mn–Al intermetallic.

Very little is known about the high temperature properties of this alloy, especially concerning its creep behavior. Only few creep studies were performed with the AZ91D magnesium alloy [1,3–17], all in the die cast condition. These studies present creep data for engineering usage, without any advanced metallurgical study or any correlation with models. More intensive research work was done on pure magnesium [18–27] and on magnesium which contained 0.8% aluminum in solid solution [28].

The current paper deals with creep properties of ingot casting, a subject which, as far as known to the authors, was not investigated at all until now.
2. Materials and experimental procedure

The material used for the investigation was a commercial ingot of the AZ91D alloy, the composition of which is shown in Table 1.

Optical microscopy of the ingot’s cross-section revealed three regions of very different microstructures, shown schematically in Fig. 1 [29]. The rim of the ingot is the chilled zone, ≈ 150 μm wide, consists of very fine, equiaxed grains. The grains in the center of the ingot are also equiaxed grains but are much larger ( ≈ 300 μm average grain diameter). The rest of the ingot consists of columnar, dendritic grains. All the creep specimens (Fig. 2) were prepared from the center section, their longitudinal direction being parallel to the length of the ingot. In order to ensure that all the specimens come from the equiaxed grains zone, only the middle longitudinal third of the ingot was used.

X-ray diffraction measurements were performed in the scanning range 20–150°. Step size was 0.02° and sampling time was 6 s.

The creep tests were performed in an oil bath in order to maintain temperature stability and to prevent oxidation of the specimens. The oil temperature was kept constant by using a heating element and an electronic controller with 0.1°C accuracy. A circulating device ensured temperature uniformity all over the bath up to 1/3°C. Specimen elongation was obtained by measuring the distance between the grips with a calibrated LVDT, the data was collected using a Yokogawa chart recorder. Creep tests were performed at three temperatures: 120, 150 and 180°C. Two types of creep tests were performed: (i) until rupture under constant load and (ii) under varying loading conditions, by changing the load when minimum creep rate seemed to be close. Seven creep tests were performed at 150 and at 180°C, three of them until rupture and the other four under varying loads. Three creep tests until rupture were conducted at 120°C. One creep test was performed on a polished and etched specimen at 180°C under the initial stress of 50 MPa. This specimen was deliberately scratched with a 1 μm abrasive polishing compound in order to notice relative grain displacement. This creep test was stopped immediately after loading, then after 4, 8 and 10%. The specimen was studied each time by optical microscopy. The creep tests parameters are listed in Table 2.

The maximum temperature of the creep tests were chosen for industrial reasons, since the AZ91D alloy is known to lose its creep strength at temperatures above 200°C [5] and therefore, it does not fit for any commercial use above this temperature.

Solution heat treatment for two creep specimens was performed at 410°C for 16 h.

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### Table 1

The chemical composition of the AZ91D alloy (wt.%) [2]

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Mn_{max}</th>
<th>Zn</th>
<th>Si_{max}</th>
<th>Cu_{max}</th>
<th>Ni_{max}</th>
<th>Fe_{max}</th>
<th>Others</th>
<th>Mg</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>8.5–9.5</td>
<td>0.17</td>
<td>0.45–0.9</td>
<td>0.05</td>
<td>0.015</td>
<td>0.001</td>
<td>0.004</td>
<td>0.01</td>
<td>Remainder</td>
</tr>
</tbody>
</table>

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Table 2
Creep tests parameters

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
<th>Temperature (°C)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>180</td>
<td>Ingot</td>
</tr>
<tr>
<td>80</td>
<td>180</td>
<td>Ingot</td>
</tr>
<tr>
<td>75</td>
<td>180</td>
<td>Ingot</td>
</tr>
<tr>
<td>40,50,60,70</td>
<td>varying 180</td>
<td>Ingot</td>
</tr>
<tr>
<td>50-interrupted</td>
<td>180</td>
<td>Ingot</td>
</tr>
<tr>
<td>90</td>
<td>170</td>
<td>Ingot</td>
</tr>
<tr>
<td>100</td>
<td>150</td>
<td>Ingot</td>
</tr>
<tr>
<td>100</td>
<td>150</td>
<td>Thermally treated ingot</td>
</tr>
<tr>
<td>95</td>
<td>150</td>
<td>Ingot</td>
</tr>
<tr>
<td>95</td>
<td>150</td>
<td>Thermally treated ingot</td>
</tr>
<tr>
<td>90</td>
<td>150</td>
<td>Ingot</td>
</tr>
<tr>
<td>60,70,80-varying</td>
<td>150</td>
<td>Ingot</td>
</tr>
<tr>
<td>50-stopped</td>
<td>150</td>
<td>Ingot</td>
</tr>
<tr>
<td>100</td>
<td>140</td>
<td>Ingot</td>
</tr>
<tr>
<td>100</td>
<td>130</td>
<td>Ingot</td>
</tr>
<tr>
<td>115</td>
<td>120</td>
<td>Ingot</td>
</tr>
<tr>
<td>110</td>
<td>120</td>
<td>Ingot</td>
</tr>
<tr>
<td>100</td>
<td>120</td>
<td>Ingot</td>
</tr>
</tbody>
</table>

Matrix is 61.9 ± 6.8 HV and the hardness of the precipitates is 188.2 ± 22.8 HV.

Most of the β particles were dissolved in the matrix during solution treatment, but no grain growth was observed. However, some β particles, mainly in grain boundaries could still be detected (Fig. 5).

3.2. Creep Tests

Creep curves of the constant load tests are presented in Figs. 6–8. Elongations until fracture vary from 9.25% (180°C, 75 MPa) to 15.38% (120°C, 115 MPa). These elongations were calculated after subtracting the immediate strain from the total elongation in order to eliminate the possibility of some slip inside the grip. Examining the slope of the curves shows that the creep rate decreases to a minimum, which is reached after approximately two thirds of the time to rupture. This fact was verified by differentiating the creep curves with respect to time. These curves that exhibit the creep rate as a function of time for the constant load tests are

3. Test Results

3.1. Microstructure

The microstructure of the material was studied by metallography, EDS and XRD. The main component is the magnesium matrix which contains aluminum in solid solution. EDS analysis showed that this solid solution contains ≈5 wt.% Al. The second phase is Mg17Al12 (β) intermetallic located at the grain boundaries but also inside the grains. Particles of Mn–Al–Fe were also found in minor amounts. Optical micrographs of the equiaxed grains zone are given in Fig. 3, which show that the grains have no preferred directionality. A scanning electron micrograph (Fig. 4) shows that the size of the precipitates varies between a few microns and tens of microns. Ten microhardness tests yielded that the room temperature hardness of the matrix is 61 ± 6.8 HV and the hardness of the precipitates is 188.2 ± 22.8 HV.

Fig. 3. Optical micrograph of the equiaxed grains zone.

Fig. 4. A scanning electron micrograph of the equiaxed grains zone.

Fig. 5. Optical micrograph of a grain boundary of a solution treated specimen.
given in Figs. 9–11. For easy comparison, the time axis is normalized by dividing creep time with the time to rapture of the individual specimen.

A log–log plot of the minimum creep rate versus stress is shown in Fig. 12. The data points of this plot, namely the minimum creep rates obtained in the various creep tests, were taken from the creep rate versus time curves (such as Figs. 9–11). The stress was corrected to the real stress at minimum creep rate by calculating the reduction of area from the longitudinal strain at this point assuming homogenous strain without necking at least until the minimum creep rate is obtained. There are two types of data points in Fig. 12: (i) derived from creep tests until fracture and (ii) estimated from load changing creep tests. The solid lines are the best-fit to data points which were derived from the tests until fracture. The dotted lines, are extrapolation of the solid lines for each temperature. One should note that all three lines of Fig. 12 are more or less parallel, with a slope of \( \approx 1.1 \).

The data points that were estimated from the load changing creep tests are placed above the dotted lines. The reason for this is that the apparent minimum creep rate, which was measured just before the load was changed, is equal to or higher than the true minimum creep rate that could have been obtained if the creep test had been performed until fracture under the same conditions. The arrows show that the true minimum creep rate might be lower, while the stress at the true minimum might be higher (due to further reduction of area in case of the true minimum creep rate).

The creep curves of the solution treated specimens are presented in Fig. 13, while Fig. 14 displays the variation of the creep rate with time in a normalized
plot. It can be noted that the minimum creep rate is lower than that of the untreated specimens that were tested under the same conditions (0.05% h\(^{-1}\) for 100 MPa with respect to 0.135% h\(^{-1}\) for the untreated specimen and 0.04% h\(^{-1}\) for 95 MPa with respect to 0.07% hr\(^{-1}\) for the untreated specimen). Minimum creep rate of the solution treated specimens was also reached after about two thirds of the time to rupture. Optical micrographs of the surface of a creep specimen tested under 180°C and 50 MPa and was interrupted after loading, after 4, after 8 and after 10% are given in Fig. 15. These micrographs present clear evidence for grain boundary separation at the interface between the matrix and the β phase. This separation occurs immediately after loading and becomes more and more intensive as the creep test proceeds. Certain details of the micrographs of Fig. 15 are not entirely in focus because height differences of adjacent grains due to the 'orange peel' effect (see below).

3.3. Fractography

All creep specimens failed in a brittle manner, i.e. no substantial necking could be detected. The average reduction of area was found to be 3.6% with a S.D. of 2.1%. The crept specimens surface had the ‘orange peel’ appearance and in addition, many intergranular cracks could be seen all over their length. An optical micrograph of a longitudinal cross section near the fracture surface is given in Fig. 16. Scanning electron micrograph of the fracture surface of Fig. 17 exhibits a typical intergranular fracture.
4. Discussion

Probably the most interesting result of this investigation is the large elongation to failure, compared with pure magnesium. The elongation to failure of pure magnesium reported by Milicka et al. [18] in the temperature range 400–800 K and stress range of 10–100 MPa, did not exceed 5%, while in this investigation up to 16% elongation was obtained. Dunlop et al. [30] recently reported the results of constant load creep testing of AZ91D pressure die cast within the temperature range of our experiments. The creep tests were terminated after 100 h, and therefore total elongations to fracture were not obtained. However, from the shape of the creep curves, one can conclude that relatively high elongation could have been reached if the tests had been carried out to fracture. The minimum creep rates reported by Dunlop et al. [30] at 150°C and under 50 MPa are 0.018% h⁻¹ and 0.031% h⁻¹ for specimens which have section thicknesses of 1 and 6 mm, respectively. These creep rates are about one order of magnitude higher than the creep rates obtained at this work under the same parameters (0.0021% h⁻¹). Mordike and Lukac [31] point at the influence of the grain size on the minimum creep rate. They claim that the minimum creep rate is proportional to $d^{-2}$, where $d$ is the average grain size. Keeping in mind that the average grain size of the pressure die cast is around 15 μm, the differences in the minimum creep rates obtained by the authors comparing with the minimum creep rates reported by Dunlop et al [30] can be explained.

From previous studies carried out on pure magnesium in the temperature range of our experiments [18–27], it appears that dislocation glide on basal planes...
controls creep behavior. However, in magnesium, which is h.c.p., only three active slip systems exist and therefore the fracture is that of the brittle type. The relatively large elongations to failure obtained with our alloy seem to contradict the basal dislocation glide controlled creep concept (as known for pure magnesium) as being the major creep mechanism. Very recent TEM study, done by the authors, of crept specimens detected many mobile dislocations on non-basal planes [32](unpublished research), a fact which suggests cross slip and therefore extended creep elongation. Also according to Vagarali and Langdon [20], the value of $n$ for dislocation glide on basal planes in pure magnesium is 4.5, while we found values of $\approx 11$, close to that found by Milicka et al. [18]. However, their $n$ values were temperature-dependent, while no such dependence was detected for our alloy in the examined temperature range. Another major difference between creep of pure magnesium and the magnesium alloy of our investigation is that the minimum creep rate of the latter is a few orders of magnitude lower. This difference is probably due to solid solution and precipitation hardening.

It is suggested that at least two factors are responsible for the relatively large elongation to failure of our alloy in comparison with pure magnesium: creep cavitation and cross slip to non-basal planes.

The specimen of the interrupted creep test (180°C, 50 MPa), which was monitored at each interruption, showed clear evidence of separation between the $\beta$ phase and the matrix. This separation occurs probably due to poor adhesion. As creep proceeds, this phenomenon gives rise to 'internal necking' which leads to the reduction of the effective cross-section of the specimen without any detectable external necking. The inter-

![Fig. 12. Minimum creep rate vs. stress.](image1)

![Fig. 13. Creep curves of the solution treated specimens.](image2)
granular cavitation also contributes to the total creep strain, however preliminary estimates indicate that this contribution is relatively small. However, the intergranular cavitation may be considered to be a stress relief mechanism and hence contribute to the dislocation based deformation process mentioned above.

It is suggested therefore, that the relatively high elongation to fracture with respect to pure magnesium is the result of the mutual action of the above described two mechanisms, e.g. cross slip and intergranular cavitation. What is the extent of contribution of each and how these mechanisms depend on the temperature and the applied stress is still to be studied.

By means of creep tests at various temperatures, it is possible to calculate the value of the activation energy for creep. According to Bird et al. [33], the temperature dependence of the minimum creep rate is given by:

$$\dot{\varepsilon} = \frac{AGb}{kT} \left( \frac{a^n}{G} \right) D_0 \exp \left( \frac{-Q}{RT} \right)$$

where \(A\), a dimensionless constant; \(G\), the shear modulus; \(b\), Burgers vector; \(k\), Boltzmann’s constant; \(D_0\), frequency factor; \(Q\), activation energy; \(R\), gas constant; \(p\) and \(n\), constants.

By differentiating Eq. (1) with respect to \(1/RT\) [20] and using the final differences approximation, the activation energy can be calculated:

$$Q = \frac{\partial \ln(\dot{\varepsilon}G^n/\dot{\varepsilon}^{-1}T)}{\partial (-1/RT)} \approx \frac{R \ln(\dot{\varepsilon}_2G^n_2^{-1}T_2/\dot{\varepsilon}_1G^n_1^{-1}T_1)}{(T_2 - T_1)/T_1T_2}$$

The shear modulus is assumed to vary with temperature in accordance with Eq. (3) (for pure magnesium) [20]:

$$G = (1.92 \times 10^4 - 8.6T) \text{ MPa}$$

Having substituted Eq. (3) in Eq. (2), Eq. (2) was then solved for six regions of temperature and the results are as follows:

1. 189 kJ mol\(^{-1}\) between 120 and 130°C.
2. 220 kJ mol\(^{-1}\) between 130 and 140°C.
3. 200 kJ mol\(^{-1}\) between 140 and 150°C.
4. 220 kJ mol\(^{-1}\) between 150 and 160°C.
5. 105 kJ mol\(^{-1}\) between 160 and 170°C.
6. 94 kJ mol\(^{-1}\) between 170 and 180°C.

Fig. 18 shows the variation of \(Q\) with temperature. There is a significant decrease of \(Q\) at the high temperature region, while the values of the activation energy at the lower temperatures are close to each other. It is possible that in the low temperature range the value of \(Q\) is constant, however there is a trend of lowering the activation energy with increasing temperature and this trend should not be neglected. This trend may be related to changes in the microstructure during the creep test. The fact that the microstructure of binary magnesium alloys are not stable at elevated temperatures has been reported (Mordike and Riehemann [34] and Wei et al. [35]) for various alloy compositions. Very recent aging studies carried out by the authors on AZ91D also prove the instability of the microstructure at the relevant temperature range. The value of the activation energy for basal planes dislocation motion in pure magnesium (which is the self diffusion energy in a Mg lattice) does not seem to be agreed by the various investigators and vary between 92 and 135 kJ mol\(^{-1}\) [20].

To support the above proposed mechanism, further experiments are required. One of these experiments, which has already been started, involves creep study of the solution treated alloy, which has no \(\beta\) phase in the \(\alpha\) grain boundaries. Two creep tests of solution treated
specimens were already carried out. As mentioned before, the minimum creep rates obtained were lower than these of the untreated specimens, a fact which supports the idea that the Mg17Al12 phase has an important influence on the creep process. This behavior may be related to solid solution hardening with higher level of super-saturation and to pinning effect due to precipitation of Mg17Al12 phase on the dislocations in addition to the absence of the proposed stress relief mechanism by intergranular cavitation.

Observation of the fracture surfaces of the crept specimens supports the proposed creep mechanism. The brittle appearance of the fracture proves that in the temperatures range of our experiments the alloy does not have a large number of active slip systems. On the other hand, the large elongations to fracture, the intergranular cracks all over the specimen and the intergranular fracture prove that besides the above proposed mechanisms deformation takes place also at the grain boundaries. The ‘orange peel’ appearance further proves the existence of relative granular displacement.

5. Conclusions

The investigation of the microstructure and creep properties of specimens prepared from an ingot casting of the AZ91D at the temperature range 120–180°C and in the load range 40–115 MPa yielded the following results.

1. The microstructure of the as-cast alloy is composed of a magnesium matrix with an average grain size of ≈300 μm and a second phase of β (Mg17Al12). Minor amounts of manganese-rich precipitates were also observed. The size of the β phase varied between a few microns and tens of microns and they were located mainly in the grain boundaries, however some of them could be detected inside the grains.

2. Creep rates were found to be about three orders of magnitude lower than those reported for pure magnesium. No steady-state stage was observed, however a minimum creep rate was reached after approximately two thirds of the creep life of the specimens.

3. The stress exponent was found to be ≈11, independent of temperature and stress. Activation energy was found to decrease with increasing the temperature from 220 to 94 kJ mol⁻¹.
(4) Solution treated specimens, which contained significantly less $\beta$ phase crept at a slower rate under the same conditions.

(5) Interrupted creep test performed on a polished and etched specimen showed intergranular separation, starting at the matrix–$\beta$ phase interfaces. Microcracks and microvoids were observed immediately after loading and their number and size proceeded with creep time.

(6) All the specimens failed in a brittle manner, i.e. without any detectable external necking. Fractography study showed that creep fracture was intergranular.

(7) It is proposed that creep is controlled by a few mechanisms: dislocation motion on basal planes (similar to that reported for pure magnesium), cross slip to non-basal planes and intergranular cavitation. All these mechanisms may explain why the AZ91D alloy elongates more than pure magnesium.

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References