Creep Properties of Mg–Gd–Y–Zr Alloys

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High temperature creep properties of Mg–Gd–Y–Zr alloys have been evaluated quantitatively. Creep test was carried out under a stress range of 50–100 MPa and a temperature range of 250–300°C. Within the limits of the creep test conditions used in this study, the activation energy for creep of investigated alloys is in the range of 160–240 kJ/mol, and the stress exponent is in the range of 3.7–5.2. Accordingly, the creep mechanism of the investigated alloys is considered to be power law creep. Creep resistance of the investigated alloys depends on chemical composition, it improves with increase in gadolinium content, which implies an increase in the quantity of precipitates. The creep resistance of the high gadolinium-containing alloys exceeds that of the existing heat resistant magnesium alloy, WE54A alloy.

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1. Introduction

As a part of the effort to improve fuel efficiency of automobiles through weight reduction, magnesium alloys are being developed as alternative lightweight materials to heavier ferrous and aluminum alloys. Presently, in the automobile industry magnesium alloys are being used for thermally mild applications such as instrument panel, steering wheel, cylinder head cover, etc. However, more significant weight savings could be achieved if magnesium is applied to components subjected to more thermally severe environment like engine parts. Regrettably, current magnesium alloys are not capable of such high temperature applications. Addition of gadolinium and other heavy rare earth elements has been found to improve the high temperature tensile properties and corrosion resistance of magnesium alloys. Particularly, it has been reported that in binary Mg–Gd alloys, the tensile strength increases linearly with gadolinium content. However, high gadolinium content results in an increase in the densities and also the cost of the alloys. Problems associated with the ductility and cost of the large amount of gadolinium required to obtain high strength led to the development of the Mg–Gd–Y–Zr and Mg–Gd–Nd–Zr alloys in which a part of gadolinium is replaced with yttrium or neodymium while zirconium is added for grain refinement. The yttrium-containing alloys are found to exhibit higher strength than the neodymium-containing alloys, because the lower solubility of neodymium in magnesium results in lesser solid solution strengthening and smaller age hardening. One of the alloys, GY103K (Mg–10 mass%Gd–3 mass%Y–0.45 mass%Zr) in T5 condition after hot rolling, exhibits a very high tensile strength of 420 MPa at 200°C. This value is about 70% higher than that of the existing heat resistant magnesium alloy, T6-treated WE54A, which is 244 MPa at 200°C. Optimum homogenization temperature for hot forging, and the possibility of forging the new alloys into a piston shape have been investigated. As a result, sound pistons with excellent high temperature tensile properties were obtained. However, since creep is an important parameter for evaluating the performance of heat resistant materials, the creep properties of the new alloys must be investigated in order to ensure that the alloys can withstand the high thermal load associated with automobile engines.

Therefore, this study is designed to give an insight into the creep behavior of Mg–Gd–Y–Zr alloys. The gadolinium and yttrium contents are systematically varied in order to understand the influence of the alloying elements on creep deformation of the alloys. Consequently, creep properties of five kinds of Mg–Gd–Y–Zr alloys are evaluated in the high temperature (250–300°C)-high stress (50–100 MPa) range. Peak aged-hot rolled samples of the alloys were used for the evaluation because it has been shown that the strength of Mg–Gd–Y–Zr alloys remarkably increase by age hardening after hot rolling.

2. Experimental Procedure

Binary Mg–Gd and Mg–Y master alloys and the ternary Mg–Gd–Y master alloy were first prepared using magnesium, gadolinium and yttrium of 99.9% purity. Melting was conducted in high frequency induction furnace under argon atmosphere, and the melt was poured into a mold held at room temperature. Then the master alloys were remelted in an atmosphere of a mixed gas of CO2 and SF6 using ordinary electric resistance furnace. Zirconium was added to the remelted master alloys at 830–850°C in form of Mg–30%Zr master alloy. After stirring and holding for about 10 minutes the melt was poured into a mold held at 250–300°C. Table 1 shows the chemical compositions, densities (ρ), hardness (HV) and specific hardness (HV/ρ) of investigated alloys. The marks given in Table 1, which indicate the symbols and contents of added alloying elements (mass%) are subsequently used as alloy names. Before hot rolling homogenization of the specimens was carried out at 500°C for 6 h, which results into small grain size and little residual Mg-RE eutectic compounds in the alloys. Hot-rolling was carried out at 500°C for a total reduction ratio of 80% at 10% per pass. After hot rolling, the specimens were solution-treated at 500°C for 2 h (rolled-T4) and then aged at 250°C. The hardness of the specimens was measured using a Vickers hardness tester in order to obtain aging characteristics of the investigated alloys and to determine the peak-aging condition (rolled-T6). The microstructure of the specimens was observed using Nikkon

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Epiphott TME optical microscope, and TEM observation was carried out using JEOL 2000FX to identify the precipitates contributing to age hardening and to observe dislocation substructure. High temperature creep properties were evaluated by conducting tensile creep tests at temperatures ranging from 250–300°C under a stress range of 50–100 MPa using plate specimens with 3 mm thickness and 15 mm gauge length. The temperature was maintained at ±3°C.

3. Results and Discussion

3.1 Microstructure

Figure 1 shows the microstructures of as-rolled and rolled-T4 specimens of investigated alloys. In as-rolled specimens, streaks parallel to the rolling direction are observed and this indicates that the specimens experience strong working strain during hot rolling. Recrystallized grains with a diameter of about 30 to 70 µm are also observed. This implies that the specimens may have experienced partial recovery and recrystallization either during hot rolling or during subsequent cooling after hot rolling. Rolled-T4 specimens indicate that the streaks decrease and the grains coarsen to about 50–100 µm after solution treatment of the rolled specimens. This result shows that the working strain introduced by hot rolling is relieved during solution treatment after hot rolling.

3.2 Aging Characteristics

Figure 2 shows the aging curves of the investigated alloys aged at 250°C. The high aging temperature was chosen in order to reduce the effect of over aging during creep test. All of the specimens show appreciable age hardening at this temperature, but the degree of age hardening is higher in high gadolinium-containing alloys. The peak hardness and the specific hardness of the alloys are listed in Table 1. The high specific hardness of high gadolinium-containing alloys shows that the strengthening effect of high gadolinium addition is enough to offset the slight increase in the densities of the alloys.

Figure 3 shows TEM images and diffraction patterns of peak aged specimen of GY38K alloy. Similar TEM images and diffraction patterns are obtained for the other alloys. As observed in (a) and (b), there are needle-like precipitates, and the diffraction patterns show that these precipitates are oriented towards the [100] direction. The diffraction pattern of (a) reveals three diffraction spots between the direct spot and [010] plane. The diffraction spots are thought to correspond to those of the β' phase with bco (body centered orthorhom-
from the precipitates can also be observed by rotating the diffraction pattern of (a) through 120 degrees. The TEM image of (c) shows the same type of internal structure, but there is no alignment of large precipitates as observed in (a) and (b). Instead, subboundaries are observed. In the other investigated alloys, the same types of precipitates are also observed, but the amount of precipitate increases with an increase in gadolinium content.

3.3 Creep properties

Figure 4 shows the typical creep curves obtained for the investigated alloys. In high gadolinium-containing alloys, primary, secondary and tertiary creep regions are clearly observed. However, as gadolinium content decreases, distinct secondary creep region is no longer observed, and in Y9K alloy, tertiary creep region is reached almost immediately after the start of creep test. Figure 5 shows the relationship between the minimum creep rate of the alloys and the reciprocal of test temperature at an applied stress of 100 MPa. The data for WE54A alloy is also indicated for comparison with the investigated alloys. There is a linear relationship between the minimum creep rate and the reciprocal of test temperature. From Fig. 5, it can be seen that the minimum creep rate tends to decrease in alloys having higher gadolinium con-

![Fig. 2 Aging curves of rolled-T4 specimens aged at 250°C.](image)

![Fig. 3 Transmission electron micrographs and diffraction patterns of peak-aged GY38K alloy (rolled-T6). Aging was carried out at 250°C for 64 h.](image)
tent. This is most likely due to large amount of precipitates in high gadolinium-containing alloys. GY38K seems to be an exception as it exhibits lower minimum creep rate than GY56K. However, it should be noted that GY38K exhibits larger age hardening than GY56K as shown in Fig. 2. The activation energy for creep of the alloys was calculated from the slope of the graphs in Fig. 5, and the result is shown in Table 2. As shown in the table, the activation energy for creep of the alloys within the evaluated stress and temperature ranges is higher than that of self diffusion of magnesium, which is 130–140 kJ/mol.13–15 Similar results are obtained for WE54A,10,16 WE43A,17 Mg–6.6 mass%Y–3 mass%Mn–0.45 mass%Zr18,19 and Mg–7.9 mass%Y.20 Henning et al.19 proposed two different creep deformation mechanisms in the regime of high activation energy: the cross slip of dislocations from basal to prismatic planes and the drag of jogs by screw dislocations to leave a trail of interstitials. From the TEM observation of the creep specimens of Mg–7.9 mass%Y alloy, Suzuki et al.20 have also reported that the dynamic precipitation of $\beta''$ phase leads to the activation of the non-basal slip systems. Similar creep deformation mechanism may also occur in the investigated alloys as described later and, therefore, the apparent activation energy is expected to be larger than that of self-diffusion of magnesium. Also, the apparent activation energy decreases with an increase in gadolinium content. The activation energy is influenced by the slip planes

![Fig. 4 Creep curves of investigated alloys. Creep test was carried out at a temperature of 300°C and a stress of 100 MPa.](image)

![Fig. 5 Relationship between minimum creep rate of the investigated alloys and the reciprocal of test temperature.](image)

![Fig. 6 Relationship between minimum creep rate of the investigated alloys and applied stress.](image)

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Apparent activation energies for creep stress exponents of the investigated alloys crept at temperature range of 200–250°C under a stress range of 50–100 MPa.</th>
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<tr>
<td><strong>Alloys</strong></td>
<td><strong>Temperature, T/°C</strong></td>
</tr>
<tr>
<td>GY152K</td>
<td>250–300</td>
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<tr>
<td>GY84K</td>
<td>&quot;</td>
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<td>GY56</td>
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<td>GY38K</td>
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<td>Y9K</td>
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<td>WE54A</td>
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<tr>
<td>Mg–6%Y–3%Nd–0.7%Zr&lt;sup&gt;19&lt;/sup&gt;</td>
<td>&gt; 277</td>
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<tr>
<td>WE54A&lt;sup&gt;20&lt;/sup&gt;</td>
<td>250–300</td>
</tr>
<tr>
<td>WE43A&lt;sup&gt;17&lt;/sup&gt;</td>
<td>230–270</td>
</tr>
<tr>
<td>Mg–7.9 mass%Y&lt;sup&gt;18,20&lt;/sup&gt;</td>
<td>257–277</td>
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of dislocations, i.e. basal, prismatic and pyramidal planes, which are controlled by the amount of precipitates and solute atoms. Further investigations are needed to fully understand the creep mechanism of the investigated alloys. The variation of minimum creep rate with applied stress is shown in Fig. 6. Within the investigated stress range, there is a linear relationship between the minimum creep rate and the applied stress. The stress exponents calculated from the slope of the graphs in Fig. 6 are shown in Table 2. The values are similar to those reported earlier for WE54A, WE43A, and Mg–7.9 mass% Y. As shown in Table 2, stress exponent tends to increase with a decrease in gadolinium content. The small differences in the values of stress exponents can be attributed to the different microstructural instabilities relating to

Fig. 7 Transmission electron micrographs and diffraction patterns of peak-aged GY38K alloy (rolled-T6) after creep test. Creep test was carried out under a stress of 50 MPa at a temperature of 300°C.
the natures and quantities of precipitates and solute atoms in each alloy. Considering the stress exponents, which vary from 3.7–5.2, it can be assumed that the prevalent creep mechanism in the investigated alloys within the evaluated stress and temperature ranges, is dislocation creep (power law creep).

Figure 7 shows the TEM images and diffraction patterns of ruptured specimen of GY38K alloy crept at 300°C under an applied stress of 50 MPa. The TEM image of Fig. 7(a) reveals that precipitates coarsen considerably during creep test. Since the diffraction pattern is similar to that obtained before creep test, it means that the β’ phase did not transform into equilibrium phase, but it only coarsened. As shown in the TEM image of Fig. 7(b), only particle-like precipitates, indicated by arrows, can be observed on the dislocation lines and there is heavy dislocation in that region. This result suggests that while the old precipitates that precipitated after aging coarsen during creep test, new ones may precipitate dynamically during creep test. The diffraction pattern shows that diffuse diffraction spots are observed half way between the direct spot and [010] plane. This may be due to β” phase with DO_{19} crystal structure.1,2,8,10–12 These precipitates may also be responsible for obstructing dislocation motion on the basal plane, thereby, reducing the creep rate and increasing the activation energy for creep. The TEM image of Fig. 7(c) is observed from the same grain as Fig. 7(b). Some of the dislocation lines that are observed in Fig. 7(c) are parallel to the Burger’s vector whose direction is (11–20), while others deviate by about 60° from direction of the Burger’s vector (arrow1). It is difficult to adequately classify the dislocation lines from Fig. 7(c) alone. Another TEM observation under a different beam condition is necessary to determine the type of dislocations and the exact plane on which they glide. However, Suzuki et al.20 have reported that during creep of Mg–7.9 mass% Y alloys dynamic precipitation occurs at the stage of minimum creep rate, resulting in the control of slip of dislocations on the basal plane while the cross slip of dislocations from the basal to the non-basal planes is activated. Furthermore, as shown in Table 2, the values of the apparent activation energy for creep of the investigated alloys are much larger than that of the activation energy for self-diffusion of magnesium, and they are almost the same as those reported by Suzuki et al.20 for Mg–Y alloys. The result of TEM observations using the ruptured specimen is also similar to that reported by Suzuki et al.20 for the Mg–Y alloys using specimens at the minimum creep stage. Thus it can be assumed that the TEM images of Fig. 7 reflect the microstructure of the specimen at the stage of minimum creep rate. Based on the above results, it may be concluded that the slip of dislocations occurs on the basal plane at the early stage of creep while the dynamic precipitation of β” phase on dislocation lines results in the control of slip of the dislocations on the basal plane, and then non-basal slip can occur. Therefore, if the precipitates effectively control dislocation motion on the basal planes, then the activation of the non-basal slip planes, which requires high critical shear force,21 may be the creep rate controlling mechanism.

Figure 8 shows the SEM images of creep ruptured surfaces and microstructures near the ruptured surfaces of investigated alloys tested at 300°C under a load of 75 MPa. In all the examined alloys, cavities are observed on grain boundaries perpendicular to the tensile direction. This shows that cracks that originate at triple points and grain boundary precipitates and cavities that develop from the pile-up of vacancies on the grain boundaries link up through grain boundary sliding, leading to creep rupture. In GY152K alloy, equilibrium precipitates are observed in grains and precipitation free zones (PFZ) are also observed along the grain boundaries. This is because precipitation occurs preferentially around dislocation pile-ups within the grains and along the grain boundaries, resulting in PFZ at the regions adjacent to the grain boundaries. Furthermore, the fracture mode observed in all investigated alloys is intergranular fracture. This is because numerous precipitations occur at the grain boundaries during creep and rupture originates from the cavities that develop around the grain boundary precipitates during creep.
4. Conclusions

In this study, high temperature creep properties of hot rolled Mg–Gd–Y–Zr alloys were investigated within a temperature range of 250–300°C and applied stress range of 50–100 MPa. The results obtained are summarized below:

1) All of the investigated alloys exhibit age hardening and the amount of age hardening increases with an increase in gadolinium content.

2) Within the range of temperature and applied stress evaluated in this study, the activation energy for creep of the investigated alloys varies from 160–240 kJ/mol, which is higher than the activation energy for self diffusion of magnesium, and the stress exponent varies from 3.7–5.2. Therefore, dislocation creep (power law creep) is considered to be the dominant creep mechanism.

3) Creep properties depend on the chemical composition of the alloys, such that, creep resistance of the alloys improves as the Gd content increases, exceeding that of WE54A alloy in high Gd-containing alloys.

4) β" phase with DO19 crystal structure is precipitated on dislocation lines during creep test, and this may help to impede dislocation motion on the basal plane.

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REFERENCES