High-temperature behaviour of as die-cast and heat treated Mg–Al–Si AS21X magnesium alloy

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Abstract

The mechanical behaviour of the magnesium alloy AS21X (Mg–2% Al–1% Si alloy AS21 modified by addition of rare-earth elements) was experimentally investigated in the as die-cast and in the solution treated and artificially aged conditions in the temperature range from 70 to 210 °C. The as die-cast microstructure was characterized by coring effects and by the presence of Mg2Si and Mg17Al12 particles. Holding at temperatures below 180 °C caused massive formation of fine-scale precipitates at the grain boundaries. The homogeneous distribution of Al in the Mg-rich H9251-phase of heat treated material caused at these temperatures the formation of further amounts of Mg17Al12 particles, also within the grains. At 210 °C, Mg17Al12 particles dissolved in both material conditions. At low temperatures, longer times to creep rupture and better creep resistance were displayed by the as cast (AC) material. The properties in the heat treated (HT) condition approached those of the as cast material at the highest test temperature. The suggested deformation mechanism of the alloy up to 180 °C is climb-controlled dislocation creep, for which core diffusion could play a significant role at 70 °C in the as cast material.

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1. Introduction

The high temperature structural stability and mechanical behaviour of Mg die-cast alloys are of great importance for structural parts where thermal exposure up to 150–180 °C can be experienced during service. Nevertheless, even moderate temperatures, below 100 °C, were reported to cause marked changes of the non-equilibrium structure and mechanical properties of die-cast alloys [1,2], and led to creep phenomena of importance for industrial components [3–5]. Among Mg die-cast alloys, the Mg–2% Al–1% Si alloy (AS21) was specifically developed as a creep-resistant alloy for use in car engines [6]. The presence of Mg2Si particles at grain boundary was reported to enhance the long-term creep properties of this alloy so that in the low strain rate and high temperature regime, the material exhibits better creep resistance with respect to the widely diffused Mg–9% Al–1% Zn (AZ91) and to the other alloys containing only Mg17Al12 particles [7]. The AS21 alloy has recently been modified by minor additions of rare earth in order to improve its corrosion resistance [8]. The high-temperature behaviour of such modified alloy, AS21X, has been studied in the present paper in as die-cast and solution treated and aged conditions.

2. Material and experimental procedures

The Mg–Al–Si alloy AS21X, of chemical composition (mass%): Al = 2.1, Mn = 0.09, Si = 0.94, Zn = 0.16, RE = 0.1, Mg = balance, was investigated in the present study. Test bars having the shape of cylindrical specimens of 6-mm gauge diameter were die-cast at Hydro Light Metals Research Centre in Norway by a cold-chamber machine. Experiments were carried out both on specimens in the as cast (AC) condition and in the heat treated (HT) condition. The heat treatment consisted in a solution quenching at 415 °C for 2h and water quenching followed by aging at 150 °C for 4h. Such heat treatment corresponds to that reported as optimal for the mechanical properties of AZ91D, usually
taken as reference material for magnesium die-cast alloys [9,10]. This material condition guarantees more homogeneous structure and an improved thermal stability.

Uniaxial creep tests in tension were carried out on AC and HT specimens on constant load machines at temperatures ranging from 70 to 210 °C. The microstructural evolution of the material was investigated by optical and scanning electron microscopy (SEM) on crept specimens.

3. Results

3.1. Creep tests

The times to rupture under creep conditions (\(t_r\)) are plotted in Fig. 1 for tests carried out at 70, 150 and 210 °C. Under the same applied stress, longer creep rupture times were reached by AC material at 70 °C. Higher test temperatures reduced the difference in terms of rupture times between the two material conditions. Minimum strain rate versus stress diagrams here in Fig. 2a and b show the creep resistance of the AS21X alloy in the AC and HT conditions, respectively. In these plots, the true stress level corresponding to the onset of secondary creep was used. Where available, the maximum true stress of the tensile curves, were also plotted as full symbols (these latter data were not described in detail in the present paper). The improved creep resistance (i.e. a lower strain rate at a fixed stress) of the AC material with respect to HT material is evident at the lower investigated temperatures.

Between 70 and 210 °C, the times to creep rupture (\(t_r\)) of the alloy well correlated to minimum creep strain rate \(\dot{\varepsilon}_m\) by the Monkman–Grant relationship:

\[
\dot{\varepsilon}_m = \frac{C}{t_r} \tag{1}
\]

The constant \(C\), corresponding to creep strain accumulated by secondary creep, was computed as the average value of single test data at 70–180 °C. Slightly different values were obtained for the AC and HT condition (0.097 and 0.096, respectively). At 210 °C, the strain accumulated by secondary creep reduced to about 0.05 for both materials due to the reduced material ductility and to the increased weight of tertiary creep displayed at this temperature.

3.2. Microstructural observations

The rapid solidification and cooling conditions experienced during die-casting process resulted in a fine grained AC microstructure (the grain size was about 15 μm) featuring primary grains of magnesium-rich solid solution surrounded by divorced eutectic. Secondary phases at grain boundaries mainly consisted of Mg2Si and Mg17Al12...
(β-phase) precipitates. The first particles had Chinese script or blocky aspect, the latter ones had a globular shape and were present in low amount. Microsegregation of Al was observed towards the external portion of the grains. The solution treatment experienced by HT samples brought about the homogenization of the Al content in the Mg-rich solution and the disappearance of Mg17Al12 precipitates at grain boundaries, while it left substantially unaltered the Mg2Si particles, whose pinning effect prevented the coarsening of grains during solution treatment. Microporosity in the form of grain boundary shrinkage voids was occasionally found, being substantially similar in size and distribution between the AC and HT materials.

Holding the AC material in the range 70–180 °C had no effect on Mg2Si particles, but caused the formation of fine precipitates of Mg17Al12 at the grain boundary regions.

Fig. 3. Fine precipitates of Mg17Al12 formed in the Al-rich α-phase at grain boundary in the AC material held at 70 °C for 290 h.

Fig. 4. Precipitates formed within grain and at grain boundaries in the HT material held at 150 °C for 100 h.
Slightly higher 10.2 and 9.3 for the AC and HT conditions, respectively. The parameter. A best fit of experimental data gave n-values of 10.2 and 9.3 for the AC and HT conditions, respectively. Slightly higher n-values were found when fitting isothermal data at 70 °C, while at 210 °C the n-value decreased to 7 and 6 for the AC and HT materials, respectively.

The apparent activation energy of the AC material in the temperature range from 120 to 180 °C can be described by the conventional power law equation:

\[ \dot{\varepsilon}_m = A\exp\left(\frac{-Q}{RT}\right) \]  

where \( n \) is the stress exponent, \( Q \) is the apparent activation energy for creep, \( R \) is the gas constant and \( A \) is a material parameter. A best fit of experimental data gave n-values of 10.2 and 9.3 for the AC and HT conditions, respectively. Slightly higher n-values were found when fitting isothermal data at 70 °C, while at 210 °C the n-value decreased to 7 and 6 for the AC and HT materials, respectively.

The apparent activation energy of the AC material in the temperature range from 120 to 180 °C (136 kJ/mol) corresponded to the activation energy for lattice self diffusion in magnesium (135 kJ/mol) [11]. Since at these temperatures the use of a single stress-exponent well described the stress dependence of strain rate, it can be concluded that the activation energy for creep is equivalent to the activation energy for self diffusion. As far as the temperature ranges 70–120 °C and 180–210 °C are concerned, \( Q \) was roughly estimated on the basis of the slope of experimental data in 1/T versus minimum strain rate plots at fixed stress levels. In the lower temperature range, \( Q \) reduced at high stresses (114 kJ/mol was computed at 160 MPa). This fact could be associated to a transition towards a control of creep deformation by core diffusion, for which an activation energy of 92 kJ/mol was reported for pure magnesium [12]. In the HT material, the relative importance of core diffusion was greater, since the apparent activation energy was generally lower than that of the AC material (114 kJ/mol in the temperature range from 120 to 180 °C, 103 kJ/mol in the low-temperature range at 160 MPa).

Between 180 and 210 °C, the apparent activation energy \( Q \) increased with stress in both materials. A similar trend at significantly higher temperatures was referred in literature for magnesium [11], and it was associated to the transition from dislocation climb-controlled creep to creep controlled by cross slip of dislocations from basal to prismatic plane. In the present alloy, the increased content of Al in the Mg-rich \( \alpha \)-phase due to dissolution of Mg17Al12 precipitates observed between 180 and 210 °C could also induce a modification in the creep deformation mechanism. However, any definite conclusion about the rate-controlling mechanisms in the high-temperature regime cannot be drawn only on the basis of the present experimental results. Considering the activation energies of the processes and the stress-exponents, it was possible to state that the AC and HT materials experienced the same deformation mechanisms. The slight difference in transition temperatures could be correlated to the different Al content in the \( \alpha \)-phase in the interior of the grains, which also allowed the formation of fine precipitates only in the HT condition.

In spite of the same deformation mechanisms controlling creep, the creep resistance of the AC condition was higher up to 180 °C. This fact can be correlated to the strengthening effect of the fine Mg17Al12 precipitates formed during creep in highly Al-supersaturated grain boundary regions. Due to increased diffusion and solubility of Al in the \( \alpha \)-phase, the amount of these fine \( \beta \)-phase particles and the thickness of the grain boundary region where they formed increased with stress in the AC material, reduced as temperature increased. Correspondingly, the strengthening effect was reduced. At the higher investigated temperatures, when the only phases in equilibrium were the \( \alpha \)-phase and the Mg2Si phase, the AS21X alloy behaved similarly in the two examined material conditions.

5. Conclusions

The high-temperature mechanical behaviour of die-casting magnesium AS21X alloy was investigated in the as cast and solution treated and aged HT conditions. At test temperatures up to 180 °C, longer times to creep rupture and improved creep resistance were displayed by the AC material. The microstructure of the AC samples was characterized by the formation of fine-scale Mg17Al12 precipitates within the \( \alpha \)-phase located also on the grain boundaries where Al enrichment occurred during solidification. Similar creep properties were found at 210 °C due to the dissolution of Mg17Al12 particles for both materials. The evaluation of activation energies for creep suggested that up to 180 °C the deformation mechanism of the alloy was climb-controlled dislocation creep, for which core diffu-
sion played a significant role at the lower investigated temperature.

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