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Compression-creep response of magnesium alloy DieMag422 containing barium compared with the commercial creep-resistant alloys AE42 and MRI230D

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ABSTRACT

The development of creep-resistant magnesium alloys that avoid the use of rare-earth alloying elements is an important area of research. The creep response of Mg–Al–Ca alloy containing barium (DieMag422) was compared to that of commercially available creep resistant magnesium alloys AE42 and MRI230D. The creep tests were performed between 175 °C and 240 °C at stresses between 60 MPa and 120 MPa. From the temperature and stress dependence of the minimum creep rate, the apparent activation energy Q_c and the stress exponent n for creep were calculated. The concept of a threshold stress was applied. True stress exponents n_t close to 5 were calculated. Microstructural investigations and phase analysis were performed on the as-cast materials as well as after creep. Fine precipitates could be identified that justified application of the concept of threshold stress. The DieMag422 alloy shows an improvement in creep resistance at low stresses compared with the other two alloys AE42 and MRI230D.

1. Introduction

There is a need to develop new creep-resistant and cheap magnesium alloys for automotive applications such as powertrains. Solid-solution strengthening, precipitation strengthening and the prevention of grain boundary sliding are the most common mechanisms used to improve creep resistance. The atomic radius of barium is 1.36 times larger than the magnesium atom, which suggests a high solid-solution strengthening ability. In Mg–Ba alloys, the Mg–Ba phases such as $Mg_{17}Ba_2$, $Mg_{23}Ba_6$ and Mg_2Ba have been reported [1–5]. Recent principle studies show that $Mg_{17}Ba_2$ and $Mg_{23}Ba_6$ are brittle phases whereas Mg_2Ba is ductile [4]. The binary system Mg–Ba is modelled in [5] and the intermetallic compounds mentioned are consistent with experimental findings. There are various binary Al–Ba phases (AlBa, Al_2Ba, Al_4Ba, $Al_{13}Ba_7$, Al_5Ba_4) [6–12] existing in Al–Ba systems. The advantage of precipitates containing aluminium that may form during solidification is that they contribute to precipitation strengthening. In addition, the amount of β-phase $Mg_{17}Al_{12}$ may be limited, because the free aluminium prefers to form other precipitates. The β-phase is reported to be detrimental to the creep resistance of magnesium alloys containing aluminium [13,14].

The addition of alkaline earth metals like Ca and Sr to magnesium alloys that are located in the same column as Ba in the periodic table of elements result in improvement of their creep resistance. The present work investigated the creep behaviour of Mg–Al–Ca alloy containing barium. Its creep properties are compared with that of AE42 and MRI230D alloys.

When a good creep resistance is desired in magnesium alloys containing aluminium, rare earths (RE) are often chosen as alloying elements, due to the formation of $Al_{11}RE_3$, Al_4RE and Al_2RE precipitates [15–17]. AE42 and AE44 are alloys that are used for high-temperature applications. A strong reason for wanting to replace rare-earth elements is because their primary production is concentrated in China, whose price development is uncertain. Creep resistant MRI230D alloy has already been investigated in terms of microstructure, phase identification and creep behaviour

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Table 1
Nominal composition of the three alloys in wt-%.

Alloy	Al	RE[a]	Mn	Ba	Ca	Sr	Sn	Mg
AE42	4	2	0.3	–	–	–		Bal.
MRI230D	6–7	–	< 0.3	–	2.1	0.3	0.84	Bal.
DieMag422	4	–	–	2	2	–		Bal.

[a] RE: rare-earth elements, usually a mixture (Mischmetal).

[18–20]. It was shown that it has a similar composition to AXJ520 and therefore (Mg, Al)$_2$Ca precipitates form that strengthen the alloy. Minimum creep rates found in tests under 110 MPa at 100 °C and 180 °C are very similar. The nominal composition of all three alloys investigated is given in Table 1.

2. Experimental

A magnesium alloy containing barium, DieMag422, was produced from a melt with highly purified magnesium (Mg-HP, > 99.9%), pure aluminium (99.9%), barium (> 99.0%) and calcium (99.5%). The nominal composition is Mg–4Al–2Ba–2Ca. The temperature was kept constant at 720 °C for 10 min and the melt was stirred. AE42 and MRI230D are commercially available alloys. They were all melted and prepared in the same way. The alloys were cast in preheated rectangular moulds with a size of $150 \times 200 \times 20$ mm^3. The mould temperature was 300 °C. A rising cast process was used in order to avoid turbulence during filling and dross in the solidifying part. After filling, the mould was cooled with water.

The microstructure of each was examined using an optical microscope, a scanning electron microscope (SEM) and an electron transmission microscope (TEM). SEM investigations were performed using a Zeiss Ultra 55 equipped with an energy-dispersive X-ray analysis (EDX) system at an accelerating voltage of 15 kV. Additionally, a field emission gun (FEG) SEM FEI QUANTA 200 was used to characterize certain phases. Initially the specimens for TEM were ground mechanically to about 400 μm, and then discs of 3 mm diameter were cut using an abrasive-slurry disc cutter (Model 360, South Bay Technology, Inc.). These discs were again mechanically ground to 120 μm, and further thinned by two-jet electro-polishing using a solution of 1.5% $HClO_4$ and 98.5% ethanol at about −45 °C and 40 V. The TEM examinations were carried out on a Philips CM 200 instrument operating at 200 kV. X-ray diffraction (XRD) investigations were carried out using Siemens diffractometer operating at 40 kV and 40 mA with Cu Kα radiation. Measurements were obtained by step scanning from 20° to 120° with a step size of 0.02°. A count time of 3 s per step was used. The lattice parameters and space group were analyzed with the Riteveld refinement method using Software Topas 2.0.

Micro-hardness and macro-hardness tests were carried out using a Shimadzu Dynamic Ultra Micro-Hardness Tester DUH-211/S and a Wolpert Dia Testor 2RC, respectively. The load used for the hardness tests was 5 g (HV 0.005) and 10 kg (HV 10). Quantitative phase analyses were performed on SEM images by means of Phase-Fraction software from PixelFerber.

The compression tests of cylindrical specimens with an aspect ratio of 1.5 (dia: 10 mm; L_0: 15 mm) were carried out using a Gleeble®3800 device. The samples were deformed at 0.001 s^{-1} strain rate in a temperature range of 20–240 °C where the temperature was controlled by a K-thermocouple.

The cylindrical specimens with a diameter of 6 mm and a length of 15 mm for compression-creep tests were prepared by electrical discharge machining. The creep tests were performed with ATS Lever Testing Systems at a range of constant temperatures of 150 °C, 175 °C, 200 °C and 240 °C, and under constant stresses of 60, 70, 80, 100 and 120 MPa. 150–200 °C is the temperature range typical for automotive powertrain components such as transmission cases [21]. The higher creep-test temperature of 240 °C provides further information about possible changes in deformation mechanisms. The lower stresses chosen for compression creep of 60–100 MPa are service stresses that apply, for example, where parts of transmission cases are bolted. The stress of 120 MPa is again to provide information about changes in the deformation behaviour above service stress levels. The tests were stopped after reaching the minimum creep rate.

3. Results and discussion

3.1. Microstructural observations before creep and hardness

Fig. 1 shows the microstructure of the as-cast DieMg422 alloy that was obtained by light optical microscopy. The size of dendrites is about 70 μm. Two types of particles were found, with different morphologies distributed at the dendrites and at grain boundaries, one is lamellar and the other blocky. The SEM micrograph shows that the white blocky phase has a size of about 5–10 μm (Fig. 2). The lamellar phase has a thickness of about 1–3 μm. The EDX line-scan analysis indicates that the lamellar phase is enriched with Al and Ca, whereas Ba is not present here more than in the surrounding area (Fig. 3a). The ratio of Al to Ca is close to 2, which is obtained by the statistical analysis of 20 EDX quantitative point measurements as shown in Fig. 3b. The plot of the atomic percentages of Al against Ca renders a fitting line with a slope of ratio 1.98 ± 0.22. This value is almost identical to the intermetallic Al_2Ca ratio and thus it can be concluded that this lamellar phase is Al_2Ca. The XRD results also demonstrate that the phase Al_2Ca exists in the as-cast DieMag422 alloy (Fig. 4). For the white blocky phase, any error in its EDX quantitative analysis caused by the disturbance from the matrix magnesium should be very small, because the size of these particles is really big with a value of 5–10 μm. Monte Carlo simulations indicate that under an accelerating voltage of 15 kV the disturbance from the matrix magnesium is very small when the particle size is more than 2 μm. EDX analysis shows that the white blocky phase is possibly the ternary phase, which contains Mg, Al and Ba with very little Ca. Statistical investigation using an EDX 20-point analysis indicates that the composition of this phase is 80.2 at% Mg, 11.5 at% Al and 7.5 at% Ba with only 0.7 at% Ca, see Table 2. The standard errors obtained are 1.1 at%, 0.8 at% and 0.5 at% for Mg, Al and Ba, respectively. This means that the quantitative results of the EDX

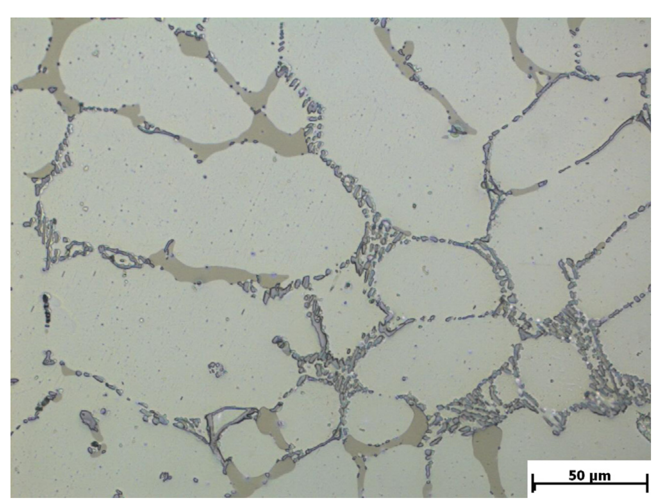

Fig. 1. Light-optical microstructure of the as-cast DieMag422 alloy.