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Creep Behaviour Under Compressive Stresses of Calcium and Barium Containing Mg-Al-based Die Casting Alloys

Hajo Dieringa[1,a], Daniela Zander[2,b] and Mark A. Gibson[3,4,c]

[1]Helmholtz Zentrum Geesthacht, MagIC – Magnesium Innovation Centre, Max-Planck-Str. 1, 21502 Geesthacht, Germany

[2]RWTH Aachen, Foundry Institute, Corrosion and Corrosion Protection, Inzestr. 5, 52072 Aachen, Germany

[3]CAST Co-operative Research Centre

[4]CSIRO Process Science & Engineering, Bayview Avenue, Clayton VIC 3168, Australia

[a]hajo.dieringa@hzg.de, [b]daniela.zander@gi.rwth-aachen.de, [c]mark.gibson@csiro.au

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Abstract. The development of creep resistant high pressure die casting (HPDC) alloys is one of the main focuses in magnesium research. Alloying elements like rare earths, calcium, strontium or scandium added to the necessary aluminium for die casting have already been introduced. Newly developed barium containing magnesium alloys with three levels of alloying additions were processed via HPDC and their compression creep response at 200 °C was evaluated. DieMag633 (Mg-6Al-3Ba-3Ca) displays the best creep resistance followed by DieMag422 (Mg-4Al-2Ba-2Ca) and then DieMag211 (Mg-2Al-1Ba-1Ca). Stress exponents from tests at different applied stresses were calculated. The creep tests were also accompanied by microstructural investigations and mechanical property evaluation.

Introduction

In recent years it has been an important driving force in magnesium alloy development to maintain or even improve creep resistance and similarly avoid rare earths elements for alloying. Die casting alloys like AE42 or AE44 are shown to have improved creep resistance compared to conventional AZ91 or AM50 and are still die castable [1]. AE44 has already been used for an engine cradle in a GM Corvette Z06. Rare earth containing QE or WE-alloys show even improved creep strength but are not castable by high pressure die casting machine (HPDC) due to a lack of fluidity and issues with hot tearing, but only in sand casting or gravity casting. It can be seen that usually improvement in creep strength is accompanied by a reduction in castability. Calcium is known to form Al_2Ca precipitates in aluminium containing magnesium alloys. These are quite stable even at elevated temperatures [2,3]. Barium in combination with magnesium and aluminium has also been described to form several stable precipitates. $Mg_{17}Ba_2$, $Mg_{23}Ba_6$ and Mg_2Ba were reported in combination with Mg [4-6] and AlBa, Al_2Ba, Al_4Ba, $Al_{13}Ba_7$, Al_5Ba_4 were reported in combination with Al [7-11]. Creep resistance of barium and calcium containing die-casting alloys shall be investigated in this paper and die castability shall be proven.

Experimental

Alloys were produced from pure elements. After melting pure magnesium, the alloying elements were added to produce DieMag alloys having three different concentrations with a nominal constant aluminium/barium/calcium weight ratio of 2:1:1. These are designated DieMag211, DieMag422 and DieMag633 and their composition is contained in Table 1. Test specimens were produced using a 250 tonne Toshiba cold chamber HPDC. During melting and holding the melt was protected using HFC-134a in CO_2 carrier gas. No burning was observed in the furnace for any of the alloys. The alloy melts were cast into a 3-cavity test bar die, consisting of two ‘dumb-bell’ and one flat test specimens.

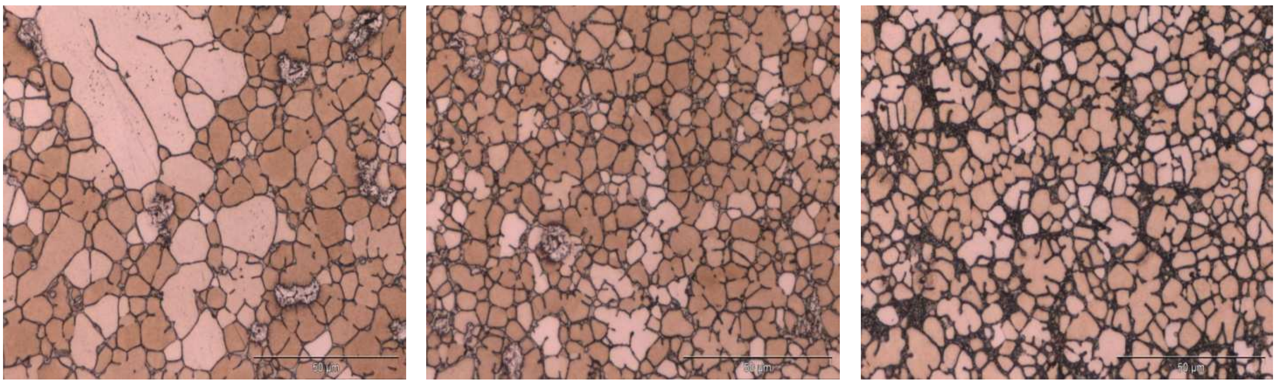
For microstructural investigations specimens were ground and polished, then etched with picric acid. Optical microscopy was done with a LEICA DMI5000 M microscope.

Tensile tests of the round 'dumb-bell' HPDC samples were conducted at room temperature using a screw driven Instron 4505 with a 100 kN load cell. A cross-head speed of 5 mm/min and an extensometer of gauge length 25 mm were used. Four tests were performed for each alloy. HV 5-Vickers hardness was measured with a EMCOTEST M1C 010 equipment, with a 5 kg load and a 30 s dwell time.

For compression creep tests cylinders with a diameter of 6 mm and a height of 15 mm were prepared by electric discharge machining the round 'dumb-bell' HPDC samples. Compressive creep tests at 200 °C and constant stresses between 60 and 100 MPa were performed in compression using ATS lever arm creep testing systems.

Results and Discussion

Microstructure. Micrographs of the three alloys in the as-cast condition are shown in Fig. 1. They consist mainly of primary dendrites of α-Mg with eutectic in the interdendritic interstices. For the DieMag211 alloy a number of externally solidified grains were observed in the microstructure, as represented by the large dendrite in the bottom left-hand corner of Fig. 1a. As can be expected the amount of precipitates increases with increasing content of alloying elements, the actual alloy compositions are contained in Table 1. In addition, the grain size decreased as well. Two different kinds of precipitates could be distinguished. These are, firstly Al_2Ca, a phase known from Ca modified AZ91 alloys, which is temperature stable and therefore improves the creep resistance [2]. The second phase was found to be $Mg_{21}Al_3Ba_2$ [12]. No evidence of hot tearing was observed in the as-cast specimens for any of the alloys which is a good indication that they all have reasonable die castability [13].



(a) ----50µm--- (b) ----50µm--- (c) ----50µm---

Fig. 1. Optical micrographs of (a) DieMag211, (b) DieMag422 and (c) DieMag633 in the as-cast state.

Table 1. Measured composition of the three DieMag-alloys (average of two analyses).

Alloy	Al [wt.%]	Ca [wt.%])	Ba [wt.%]	Fe [ppm]	Cu [ppm]	Ni [ppm]
DieMag211	2.01	0.97	1.01	50	4	1
DieMag422	4.05	1.79	1.82	31	4	1
DieMag633	6.54	2.71	2.76	49	5	3

Mechanical Properties. Vickers hardness HV5 of the three alloys was measured and the results are given in Table 2. As expected, an increase in the volume fraction of precipitates results in increased hardness. Similarly, the 0.2 % proof strength and the ultimate tensile strength (UTS) increase significantly with an increase in the volume fraction of the second phases, at both room temperature and 150 °C. However, the elongation at room temperature is limited and decreases with an increase in the volume fraction of the second phases. The variability in the elongation of DieMag211 at room temperature is probably related to the variability of the amount of externally solidified grains within any particular specimen. At the elevated temperature the ductility improves for all the alloys, for DieMag211 and DieMag422 in particular.

Table 2. Vickers hardness and tensile properties at both room temperature and 150 °C for the three DieMag alloys in the as-cast condition.

Alloy	HV5	Room Temperature			150 °C		
		0.2 % Proof [MPa]	UTS [MPa]	Elong. [%]	0.2 % Proof [MPa]	UTS [MPa]	Elong. [%]
DieMag211	54.1 ± 2.4	140.6 ± 2.3	165.9 ± 14.6	2.5 ± 1.8	111.5 ± 5.2	144.0 ± 7.1	6.8 ± 2.1
DieMag422	73.3 ± 4.3	172.6 ± 4.5	196.9 ± 6.5	1.4 ± 0.4	142.6 ± 1.2	182.1 ± 3.5	7.6 ± 1.6
DieMag633	85.3 ± 9.7	202.6 ± 0.7	229.6 ± 5.8	1.6 ± 0.5	160.0 ± 1.5	196.1 ± 2.9	3.2 ± 0.7

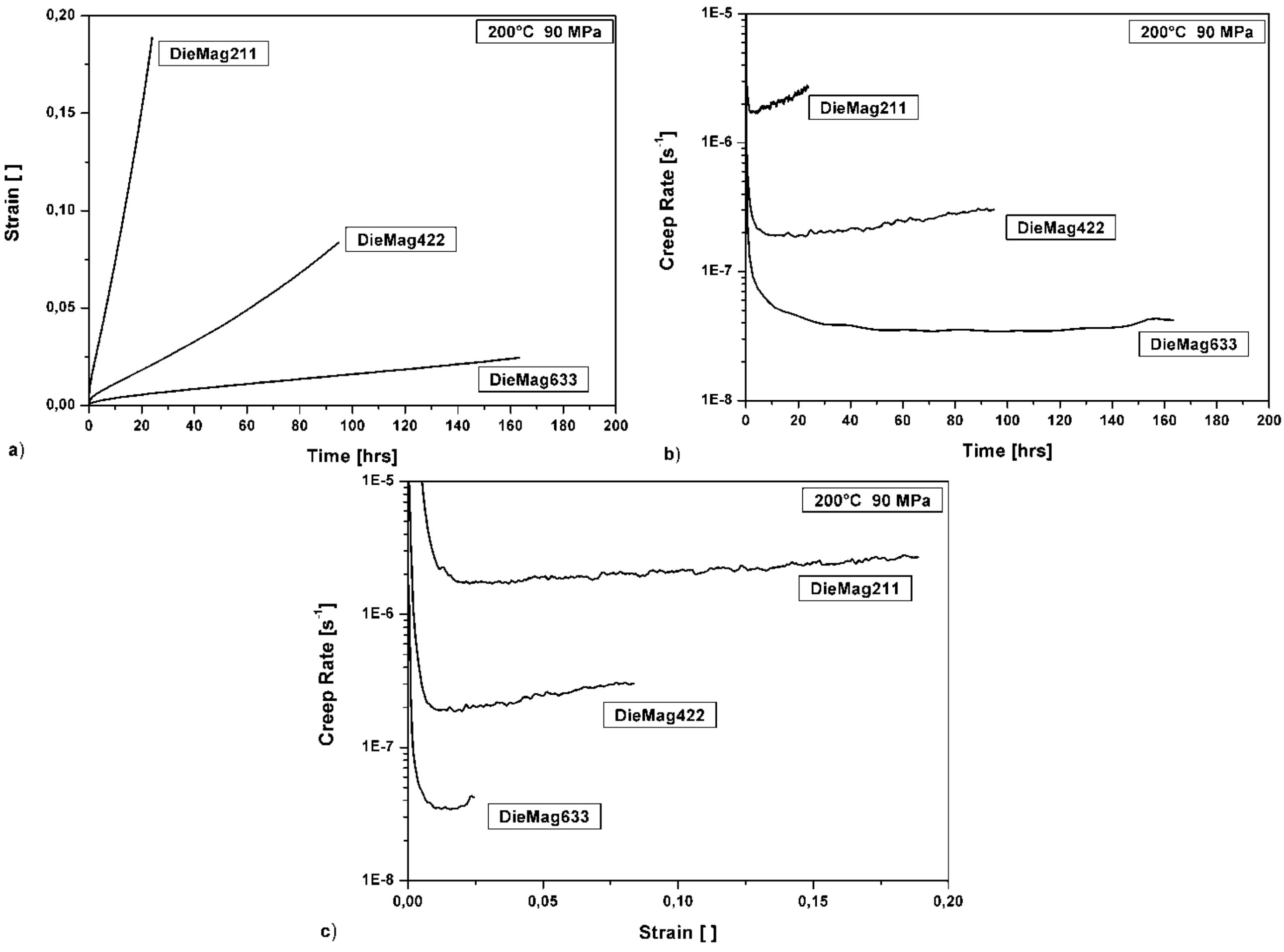


Fig. 2. A typical compression creep curve for each of the three alloys tested under the same conditions of 200 °C and 90 MPa (a); the first derivative vs. time, showing creep rate as function of time (b) and creep rate as function of strain (c).

Creep. Compression creep tests were performed at 200 °C with a constant stress of either 60, 70, 80, 90 or 100 MPa. Fig. 2 shows a set of typical creep curves for the three alloys tested at 90 MPa. In Fig. 2a the original creep curves with deformation as function of time can be seen.

It is obvious from a visual inspection of these response curves that the creep resistance is best in DieMag633 and worst in DieMag211. Fig. 2b shows the same tests but with the local creep rate as a function of time. From these curves the minimum creep rates are determined readily. In Fig. 2c the creep rate as function of deformation is shown. Collecting all minimum creep rates and plotting them according to the Norton-equation:

$$\dot{\varepsilon}_s = \frac{ADGb}{kT}\left(\frac{\sigma}{G}\right)^n \tag{1}$$

which describes the temperature (T) and stress (σ) dependence of the minimum creep rate, $\dot{\varepsilon}_s$ ($=d\varepsilon_s/dt$), is shown in Fig. 3. In the Norton equation, A is a dimensionless constant that depends on the material, D the diffusion coefficient ($=D_0 \exp(-Q_c/RT)$ where D_0 is the frequency factor, Q_c the activation energy for creep, R the gas constant, and T the absolute temperature), G is the shear modulus, b the Burgers vector and k the Boltzmann constant.

Stress exponents n can be derived from these plots. They were found to be 10.0 for DieMag211, 8.1 for DieMag422 and 7.8 for DieMag633. All of these values are higher compared to value of n = 5.4 at 180 °C, which were found in common die cast alloys like AZ91 [14]. Luo [15] reported, in a comparable alloy AC53 (5 % Al, 3 % Ca), stress exponents of 8.5 at high stress levels (70-97 MPa) and concluded, that creep deformation was dislocation controlled.

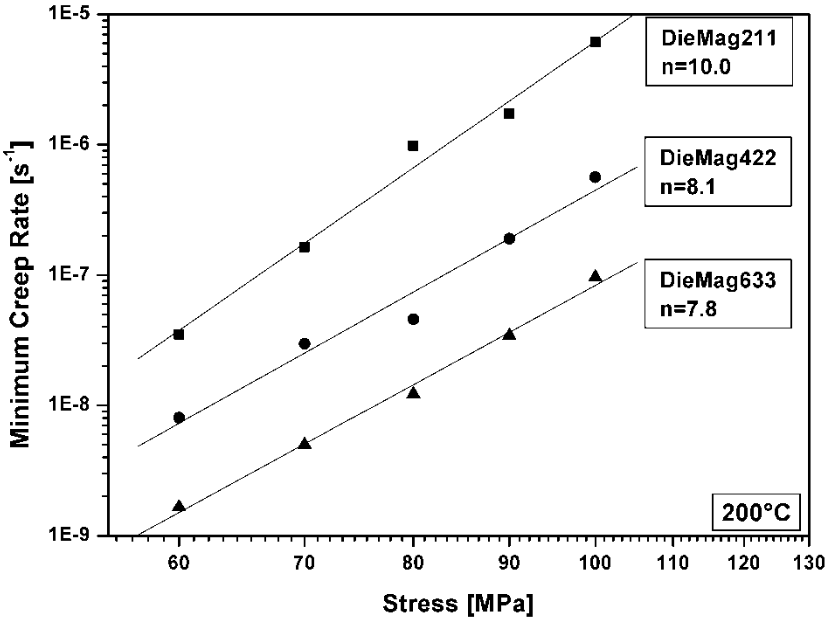


Fig. 3. Plots of creep rate as a function of applied stress with resulting stress exponents n.

Summary

Three different magnesium alloys containing aluminium, barium and calcium with a nominal a ratio of 2:1:1 were prepared and processed via HPDC. Microstructure analysis revealed that with an increasing amount of alloying elements more secondary phases precipitate and the grain size decreases. The mechanical properties increase significantly. In compression creep tests it could be shown that the creep resistance is best in DieMag633, followed by DieMag422 and DieMag211. Calculation of stress exponents from creep tests at 200 °C leads to n=10.0, n=8.1 and n=7.8 for DieMag211, DieMag422 and DieMag633, respectively, which is evidence for dislocation motion being the rate controlling deformation mechanism during creep.

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