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Effective alloy development using Thixomolding® focussing on improved creep strength of AZ91

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1 Introduction

In search of new Magnesium cast alloys for high pressure die castings, engineers have to accept a lot of time consuming melt preparations in adequate volumes and scores of crucible replacements for adjusting element additions. Moreover alloying in die casting is limited by critical factors like melt dross, oxidation, evaporation, segregation or dissolution of the alloying elements.

In contrast to this conventional alloy development technique the Thixomolding® (TM) process enables an easy way to screen a wide range of alloy compositions with much less effort in a much shorter time period. As the Thixomolding process uses Magnesium granules as feedstock material, additional alloy components can be dosed parallel to the main feedstock directly into the barrel by a second feeding system as separate granules. In addition the Thixomolding process enables a unique possibility to modify the microstructure of Magnesium cast alloys by generating a user defined solid phase fraction f_s between 0% and approximately 50%. The solid fraction is controlled by the barrel temperature of the TM machine.

Nowadays the majority of Magnesium alloy developments intend to improve the creep resistance – one of the most critical properties of Magnesium for power train applications. In order to strengthen Magnesium cast alloys for high temperature applications, alloying seems to be one of the most promising methods. Hence an adequate manipulation of the microstructure is indispensable for a reduction of creep dislocation movement in Magnesium solid solution.

In this study an alloy development survey based on AZ91 with the main intention to improve the creep resistance is presented. Starting with a series of pure AZ91 with varying solid phase content (2 - 45%) the influence of the changing microstructure on compression creep testing was investigated. Furthermore the feasibility of separate Ca addition to AZ91 by an additional dosing system for the Thixomolding process was analysed and the potential of creep improvement with varying Ca content was quantified.

2 Experimental

The casting experiments were performed on a Japan Steel Works Thixomolding (TM) machine with a clamping force of 220 t. Simple plates with a thickness of 6 mm and a dimension of 120 x 120 mm were cast. Compression creep samples with a diameter of 5 mm and a length of 7 mm as well as flat tensile specimens were machined out of these casting plates. Process parameters were: die temperature 150°C, injection speed 2 m/s, holding pressure 300 bar, shot weight 280 g, cycle time 50 s. As feedstock AZ91D granules supplied by Ecka

Granules were used. The solid phase content f_s was adjusted between 0% - 45% by reducing the barrel temperature starting from 605 °C down to 585 °C.

For adding Ca to the main feedstock a separate feeding system was installed (Fig. 1). By controlling the mass flow of either dosing screws the desired Ca content can be adjusted. Since pure Ca has a high affinity to oxygen, MgCa30 granules (30% Ca + 70% Mg) were used for the alloying element additions.

Compression creep measurements were carried out until a steady state creep rate minimum or a maximum testing time of 200 h was reached. The setup and parameters for mechanical testing and creep experiments as well as the microstructural investigation methods are explained in detail in [1].

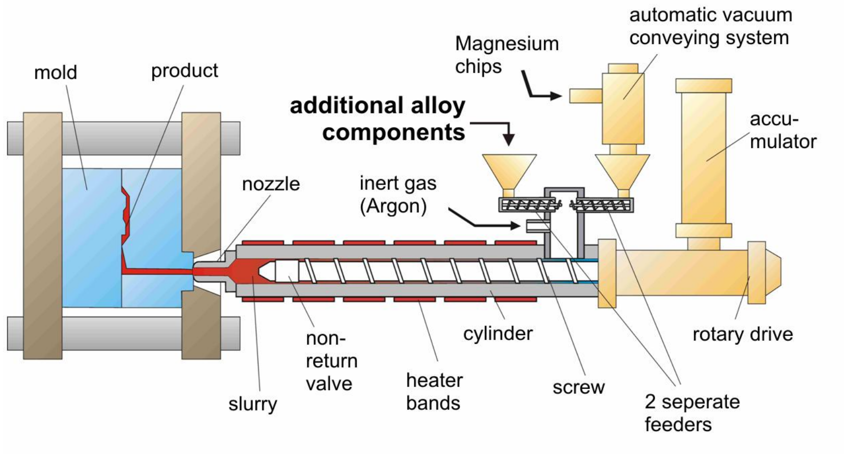

Fig. 1: Schematic setup of a Thixomolding machine with an additional feeding system for alloying components (patented [2]).

3. Results and discussion

3.1 Influence of semi-solid phases on creep for AZ91

In general the dependence of the steady state creep rate $\dot{\varepsilon}$ on stress and temperature can be expressed for most metals as

$$\dot{\varepsilon} = A\sigma^{n} \exp\left(-Q_c/RT\right) \tag{1}$$

where A is a constant, σ the applied true stress, n the stress exponent, Q_c the activation energy for creep, R the gas constant and T the absolute temperature. The stress exponent is determined from a powerlaw plot (Fig. 2a) and the activation energy can be calculated from the Arrheniusplot (Fig. 2b). In both diagrams $\dot{\varepsilon}$ of AZ91 samples with varying solid phase content is significantly reduced with increasing f_s. The stress exponent as well as the activation energy does not change significantly with varying solid fraction.

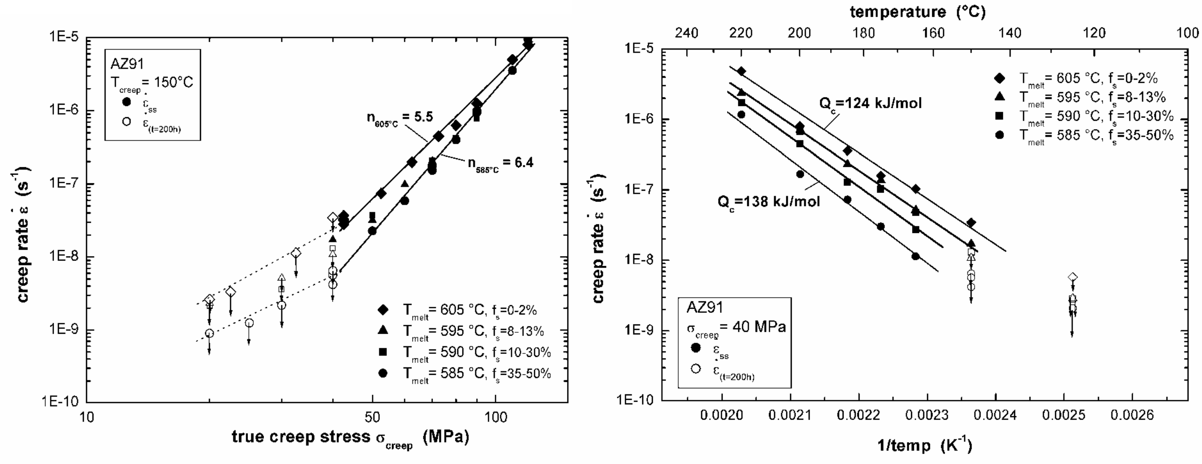

Fig. 2: Steady state creep rates of AZ91 with varying solid fraction versus (a) applied true stress and (b) testing temperature.

Since all process and creep testing parameters remained unchanged except the process temperature of the TM castings, it is clear that the microstructure is the crucial parameter, which has to explain the observed behavior.

Fig. 3 shows the microstructure of AZ91 with 0% (a) and 45% (b) solid fraction. The solid fraction, consisting of primary solidified α-solid solution in the barrel, contains only 3% Al and slightly increases with raising f_S. This is consistent with the equilibrium phase diagram, since the Al solubility in α-Mg raises with decreasing process temperature. As a result the Al content in the residual liquid melt increases with rising f_S. After injection of the slurry into the mold the residual liquid melt itself solidifies as secondary α-grains and finally as a fully divorced eutectic consisting of eutectic α and precipitated β-phases (mainly $Mg_{17}Al_{12}$).

Moreover the β-phase content in the residual liquid melt increases with raising f_S. Thermodynamic calculations using Computherm with the Scheil solidification model verify that the β-phase fraction in the residual liquid melt equals 10.0% for f_S= *0%* and 17.1% for f_S= *40%*.

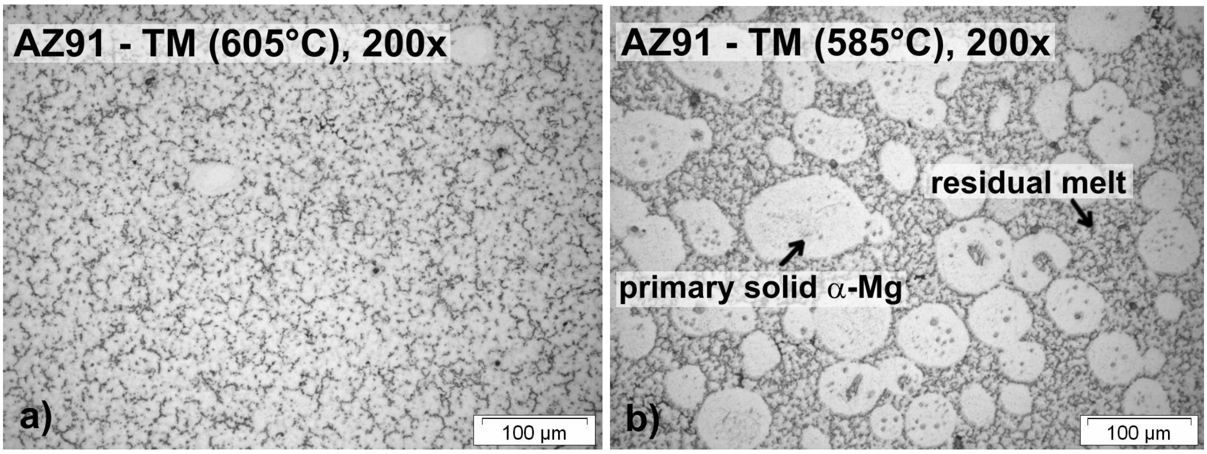

Fig. 3: Microstructure of AZ91 with (a) 0% and (b) 45% solid fraction.

The increasing Al content in the primary solid phases, the secondary α-grains and in the overall residual liquid melt was quantitatively proven by microprobe element mapping (Fig. 4). Although the Al content rises in both α-phases, the overall Al content keeps constant at about 9%, because the overall β-phase content decreases with raising f_s.

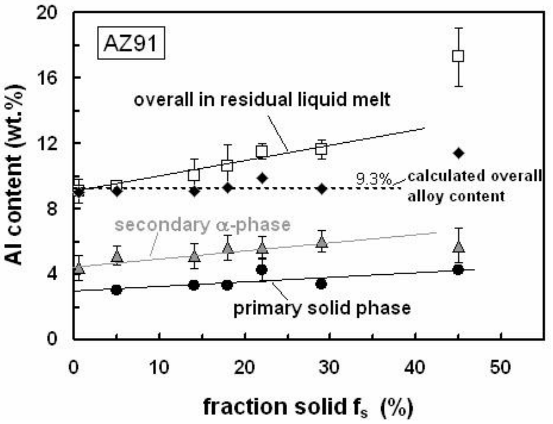

Fig. 4: Al content in different phases of AZ91 versus f_s, measured by microprobe element mapping.

Creep mechanisms

It is obvious, that the main creep mechanism of AZ91 in the tested temperature and stress range is dominated by dislocation creep, since the stress exponent varies between 5.5 and 6.4, which is in accordance with literature data of numerous Mg alloys [3]. Nevertheless a significant reduction of creep deformation with increasing solid fraction is noticed. While the absolute alloy composition of AZ91 with and without primary solid phases keeps unchanged, the microstructural investigations show three main distinguishing characteristics with increasing solid fraction:

- an increase of the average α-Mg grain size due to the bigger primary solid phases and
- an Al enrichment of the residual melt associated with
- an increase of β-phase in the residual liquid melt

The *grain size* is an important influencing factor for creep of most metals [4]. By reaching the micro scale a contribution of boundary sliding gains more and more importance. Thus the very fine grained microstructure of fully liquid cast AZ91 leads to an amplified creep deformation. To consider this effect the general equation (1) is modified to

$$\dot{\varepsilon}_{ss} = A\frac{1}{\bar{d}}\sigma^n \exp\left(-Q_c \big/ RT\right) \qquad (2)$$

with $\bar{d}$ as average α-grain size calculated by a simple rule of mixture from the bimodal α-grain size distribution of the TM samples, namely the medium primary solid α-grain size $\bar{d}_{a,prim}$ and the medium secondary α-grain size $\bar{d}_{a,sec}$ of the residual liquid melt, as follows:

$$\bar{d} = f_s \cdot \bar{d}_{a,prim} + (1 - f_s) \cdot \bar{d}_{a,sec} \qquad (3).$$

With the measured mean values of $\bar{d}_{a,prim} = 10\ \mu m$ and $\bar{d}_{a,sec} = 45\ \mu m$ the steady state creep rates of the AZ91 with varying solid fraction can be plotted versus the calculated inverse average α-grain size $1/\bar{d}$ (Fig. 5). The dotted line in Fig. 5 has a slope of 1 and demonstrates clearly an indirect proportional dependency of the creep rate from the average α-grain size.

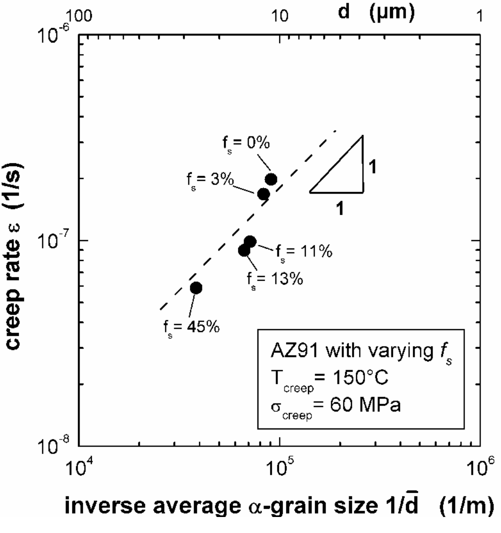

Fig. 5: Creep rates of AZ91 with varying solid phase content versus the calculated inverse average α-grain size.

As a result one explanation for the improved creep resistance of AZ91 with raising f_S can be ascribed to a reduced boundary sliding effect due to the increased grain size.

A second contribution to the improved creep deformation behavior might be attributed to the higher Al and therefore higher β-phase content in the residual liquid melt. In consequence of the increasing solid fraction, the structure of the accumulated β-phase precipitations become more contiguous and the dislocation movement might therefore be interfered with the more and more linked β-phase network structure.

A similar correlation between the morphology of the precipitates and the creep behavior can be observed by Ca addition to AZ91, as discussed in the following chapter.

3.2 Improved creep resistance of Ca additions to TM AZ91

Unlike before, the experiments with Ca addition to AZ91 were performed at a constant process temperature of 605 °C, i.e. without any primary solid phase formation. The content of Ca was increased by 1 wt.% steps up to 5 wt.% to the main feeding control of AZ91. Glow discharge optical emission spectrometry (GDOES) as well as scanning electron microanalysis measurements (EPMA) of the actual Ca content of the castings are in quite good accordance with the intended Ca addition (Fig. 6).

The feasibility of this new alloying technique by adding separate alloying granules directly into the TM machine could therefore be evaluated successfully.

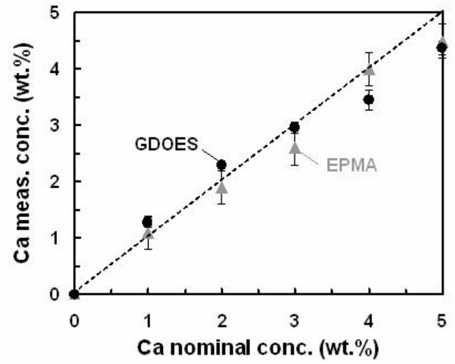

Fig. 6: Correlation between the intended and measured Ca content in AZ91.

Concerning the creep properties of AZ91 alloyed by adding Ca, a remarkable creep rate reduction of up to 3 magnitudes for 5% Ca can be observed. Fig. 7 shows the creep rate versus the applied stress of AZ91 samples with varying Ca content. According to the ASTM convention the Ca containing alloys are labeled with "X" in the following (e.g. AXZ951 for AZ91 with 4.5 - 5.5% Ca). The stress exponent increases from *n = 5.5* for AZ91 to *n = 13* for AXZ951. This is an obvious indication of a fundamental change of the creep mechanism, thus the dislocation movement is not anymore the dominant mechanism.

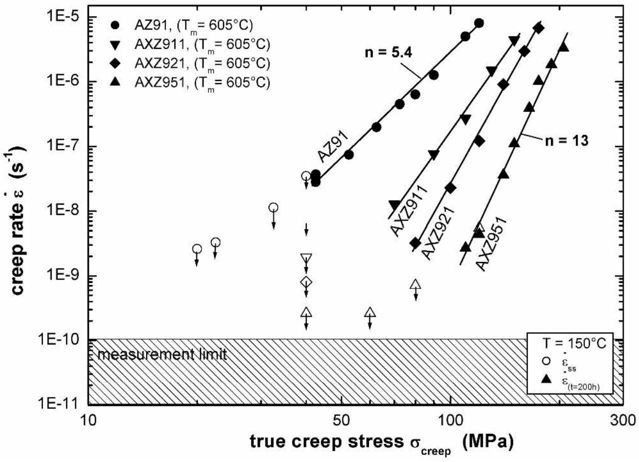

Fig. 7: Steady state creep rates of AZ91 with varying Ca addition versus true creep stress.

Associated with the remarkable increase of creep strength, a continuous change in microstructure with increasing Ca content can be observed (Fig. 8). While for pure AZ91 $Mg_{17}Al_{12}$ is the predominantly precipitation, additional Al_2Ca is precipitated with raising Ca content. For AXZ921 no β-phase can be detected any more. Further Ca addition leads consequently to an increase of the Al_2Ca volume fraction and therefore to a more and more continuous precipitation network.

On the one hand the positive effect of Ca addition to Mg-Al alloys is most likely attributed to the hindrance of dislocation movements from one α-grain to another due to that continuous precipitation network, which act as a sustaining framework against plastic deformation. On the other hand a fine dispersed Ca precipitations within the α-grains may also be a possible contribution to reduce dislocation movements, which is discussed in detail in [1].

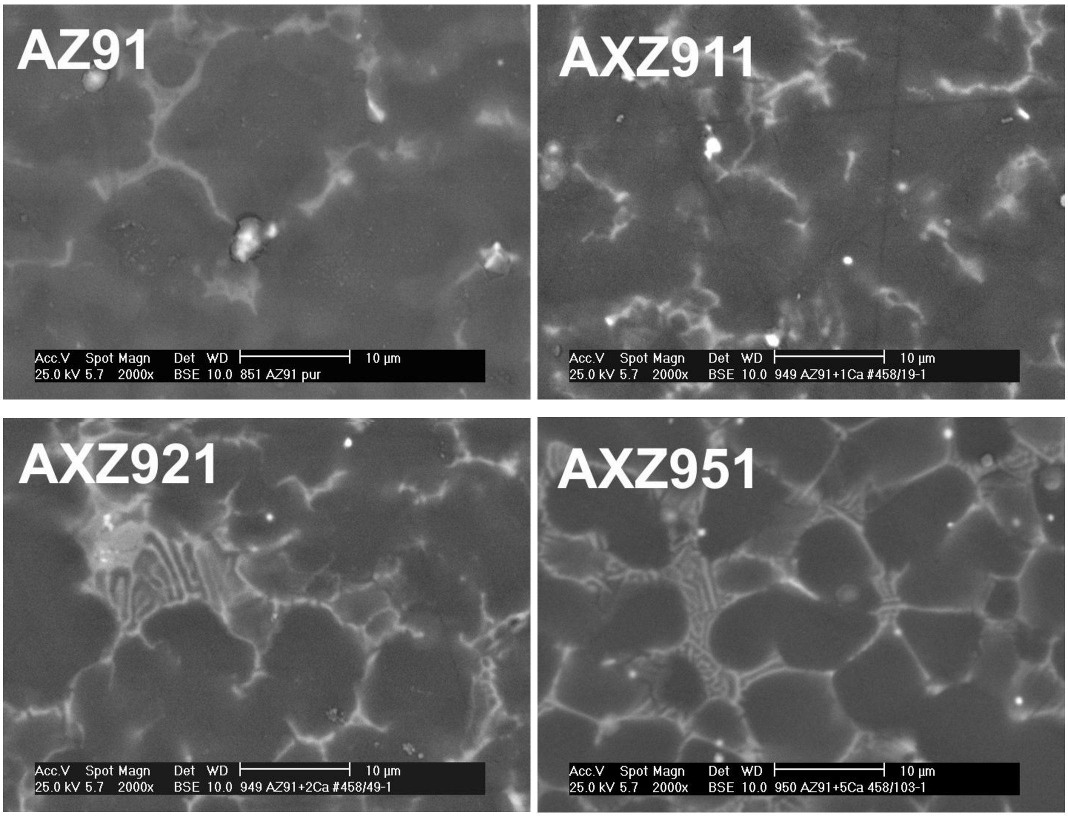

Fig. 8: Change of microstructure with increasing Ca addition to AZ91 (SEM in as cast condition).

5 References

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