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Effect of long term annealing on the microstructure of hpdc AZ91 Mg alloy: A quantitative analysis by image processing

D.G. Leo Prakash [a,*], Doris Regener [b], W.J.J. Vorster [a]

[a] *Department of Engineering Science, University of Oxford, Parks Road, OX1 3PJ Oxford, UK*
[b] *Institut für Werkstoff- und Fügetechnik, Otto-von-Guericke-Universität Magdeburg, PF 4120, 39016 Magdeburg, Germany*

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Abstract

A computational image analysing algorithm was developed for the separation and quantitative characterisation of different $Mg_{17}Al_{12}$ phases in the long term annealed (LTA), high pressure die cast (hpdc) AZ91 magnesium alloy such as β_m (massive) and β_{c+d} (continuous + discontinuous). The size distribution, nearest neighbour distance (NND) distribution, number density, average size, average NND and area fraction of β_m and β_{c+d} phases with respect to different annealing temperatures were obtained by means of novel image processing techniques and compared with the as-cast (AC) material. The different trends associated with the changes of these quantities and morphologies, nucleation and agglomeration of the phases with respect to the annealing temperature is explained. These micro quantities are also correlated with the tensile properties of different annealed castings.

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Keywords: AZ91 Magnesium alloy; Pressure die casting; Long-term annealing; Microstructural quantification; Computational image processing; Tensile properties

1. Introduction

The past decade has witnessed significant usage of Mg-based alloys, especially in the automotive industry [1]. Die cast alloy component manufacturers are expected to become the largest end user of magnesium in the next decade [2] with AZ91 accounting for more than 50% of all high pressure die castings [3] because of its good strength, ductility and castability [4]. The typical microstructure of die cast magnesium alloy consists of cored dendrites of primary, non equilibrium eutectic constituents and aluminium rich intermetallic compounds. These grain boundary intermetallic compounds are known as β phase ($Mg_{17}Al_{12}$) in relevant literature.

Automotive components of Mg alloys may be exposed to moderate temperatures in the range of 60–200 °C. The relative low melting point of Mg alloys causes an increase in diffusion of atomic species during extended exposure to temperatures in this range. Long term exposure to moderate temperatures of engine parts influences the properties of the material which affects the vehicle's life time [6–10] hence, as-cast AZ91 is most commonly used in vehicle interiors and under the bonnet (mainly engine covers and gearbox housings).

Various authors reported the presence of discontinuous (β_d) and continuous (β_c) [5–7,9,10] precipitates in directly aged specimens of AZ91. However, very limited information is documented on the quantification of β phase. NMR spectroscopy was used recently for the bulk quantification of β phase [4,11]. However, there is no information about the quantification of individual massive, continuous and discontinuous β phases. As all the precipitates are the

* Corresponding author. Tel.: +44 1865 273108; fax: +44 1865 273813.
E-mail address: leo.prakash@eng.ox.ac.uk (D.G. Leo Prakash).

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same phase ($Mg_{17}Al_{12}$) chemical techniques or techniques that recognize crystal structure cannot be used to determine the proportion of individual morphology. Understanding the influence of these different particles on the macro properties is also important for structural applications as there is no significant research in this direction.

The aim of this work is to separate and quantify the different β_m and β_{c+d} phases from the microstructure and discuss the microstructural changes with respect to different LTA hpdc AZ91 materials treated at 100, 150 and 200 °C for 1000 h using computational image processing. The correlation of micro quantities with tensile properties is the additional scope of this contribution.

2. Experimental procedure

The investigated AZ91 Mg alloy was produced by cold chamber hpdc machine GDK 200 in the form of plates with the dimension of 200 × 53 × 10 mm. The chemical composition of the produced alloy is presented in Table 1. The high-pressure die cast plates were aged by long term annealing (a condition designated T5) in air at temperatures of 100, 150, and 200 °C for the time of 1000 h. Tensile specimens of a cross section of 10 × 10 mm with 50 mm gauge length were machined from the plates for the tensile analysis. Uniaxial tensile test was performed on these specimens at a constant strain rate of $10^{-4}\,s^{-1}$ in a computer controlled servohydraulic test machine at room temperature. Specimens of size 10 × 10 × 10 mm were machined from each tensile test (AC and LTA) and polished by standard methods for optical and scanning electron microscopy for microstructural characterisation. The cross-sections were prepared by standard methods and a combination of picric acid, water, acetic acid and ethanol solution was used as an etchant to reveal all the micro inhomogeneities of the AZ91 alloy. This cross-section was further introduced to optical and scanning electron microscopy for microstructural characterisation. The details of image processing procedure are described in the following sections.

3. Image processing details

β_m, β_c, and β_d phases are clearly visible in SEM at 4000X and only β_m phase is visible in optical microscope at 1000X for the present etchant. The light-dark variations in the microstructure are related to the different aluminium concentrations in the solid solutions. Fig. 1 shows the optical microstructure of hpdc AZ91 alloy contains primary Mg solid solution (α-primary), compact eutectic β phase, supersaturated solid solution (α-secondary) and the pores. The non equilibrium solidification of hpdc AZ91 alloy due to high cooling rate results in the development of these different phases [12].

Table 1
Chemical composition of alloy investigated

Alloy	% Al	% Mn	% Zn	% Si	% Cu	% Ni	% Fe
AZ91	9.3	0.12	0.79	0.02	0.0007	0.0006	0.0046

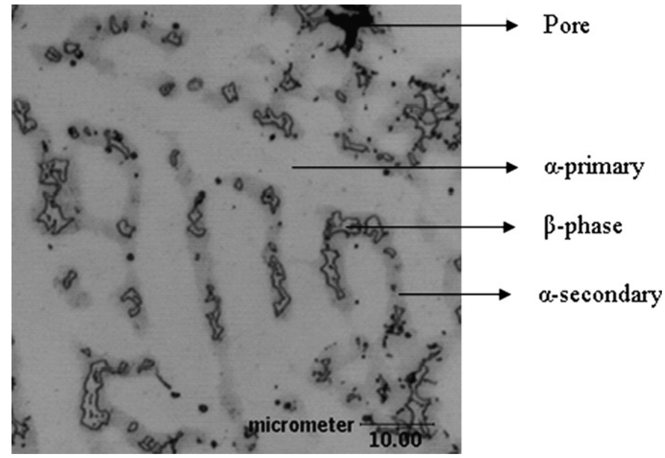

Fig. 1. Typical microstructure of hpdc AZ91 magnesium alloy which shows the different micro features.

An algorithm of the image processing procedure adopted in the present work is shown in Fig. 2. Continuous microstructural fields using optical microscopy were grabbed at 1000X from the centre of the cross-section of LTA and AC specimens. These microstructures were used to create a montage of an area 0.18 mm^2 with a high magnification in one field of view. The procedure of montage creation is documented elsewhere [13,14]. The β_m phase was quantified from these montages and covering such a high area of microstructure minimises the error in the results.

The microstructures of a selected region, which was grabbed using optical microscopy, was viewed in SEM and the microstructures were grabbed from the same location. Attention was given to grab the microstructures, which cover the exact area from the exact location in both optical and SEM microscopy. The location of the β_m phase was computed as Cartesian coordinates by using the microstructures obtained from the optical microscopy as datum. These (X, Y) coordinates of β_m phase were used to identify the same phases in the SEM microstructures and to separate the β_m phases from the β_{c+d} phases. The separated β_m and β_{c+d} phases underwent microstructural quantification studies. A montage of 0.74×10^{-2} mm^2 from the SEM is used to quantify the β_{c+d} phase.

The area of each separated β_m and β_{c+d} phase from the corresponding montage was obtained by a computer coding and the corresponding size distribution of the features was plotted. The NND of every individual feature was calculated using the coordinates from these montages by a computer code. The distance between two points (X_1, Y_1) and (X_2, Y_2) can be calculated from the following formula:

$$D = ((X_2 - X_1)^2 + (Y_2 - Y)_1)^2)^{1/2}$$

The clustering nature of these features was explained by comparing the spatial arrangement of them from the pres-