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Microstructure and quantitative analysis of cast ZRE1 magnesium alloy

T. Rzychoń [a,*], A. Kiełbus [a], J. Szala [a]
[a] Department of Material Science, Silesian University of Technology, 40-019 Katowice, Poland
*Corresponding author: e-mail: tomasz.rzychon@polsl.pl

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Abstract

A number of commercial magnesium casting alloys based on Mg-Zn binary alloy system contains some additions of rare earths metals and zirconium. The rare earth elements are added to improvement creep resistance, zirconium to improvement mechanical properties at room temperature due to strongly effect on grain size of magnesium alloys. Quantitative and qualitative characteristic of microstructure of Mg-3Zn-3RE (ZRE1) were analyzed. The optical and scanning electron microscopy were used to study the morphology of microstructural compounds in this alloy. The X-ray diffraction and X-ray energy dispersive spectroscopy analysis were used to determined phase composition of ZRE1 alloy. In the purpose quantitative description of microstructure automatic procedures using Met-Ilo image analysis program were developed. Prepared automatic procedures will be useful to future investigations the effect of long-standing heat treatment and influence parameters of sand casting on microstructure of this alloy. The results show that microstructure of ZRE1 alloy contains α-Mg matrix with discontinuous network intermetallic $(Mg,Zn)_{12}RE$ compound at the grain boundaries. The intermetallic phase content in as-cast state was 10.8 % and grain size was 766 μm^2.

Keywords: Metallography, Microstructure, Cast magnesium alloys

1. Introduction

Magnesium alloys are called green-engineering material with great development potential because of its low density, high specific strength and stiffness, superior damping capacity, good electromagnetic shielding characteristics and good machinability. However, the number of commercially available magnesium alloys is still limited especially for application at elevated temperature. The applications of most common magnesium alloys, such as AZ91 and AM50 with outstanding mechanical properties and die castability are limited to temperatures below 120 °C. This limitation is attributed to the low hardness of the intermetallic phase $Mg_{17}Al_{12}$ under high temperature [1,2]. The search for creep-resistant alloys has led to the development of rare earth containing magnesium alloys, for example Mg-Al-RE (AE42, AE44) and magnesium alloys with strontium or calcium. In these alloys, structure is characterized by the second phases from system Al-RE, Al-Sr or Al-Ca at the grain boundaries which are stable line compound with a relatively high melting point. However, at elevated temperatures, disadvantageous $Mg_{17}Al_{12}$ phase will precipitate from solid solution or will form during decomposition of intermetallic phases, e.g. $Al_{11}RE_3$ in AE42 alloy [3-10]. Therefore, magnesium alloys, which does not contain aluminum, has been developed. Among these alloys, important role plays alloy, designated Elektron ZRE1, exhibits superior high temperature creep and resistance to stress relaxation compared to that of benchmark alloy AE42. Elektron ZRE1 is a magnesium based alloy containing zinc, rare earths and zirconium. Zinc is usually used in combination with aluminum, zirconium, rare earths, or thorium to produce precipitation-hardenable magnesium alloys having good strength. Zinc also helps overcome the harmful corrosive effect of iron and nickel impurities that might be present in the magnesium alloy. Rare earth metals are added either as mischmetal to reduce of costs of production. Additions of the rare earths increase the strength at elevated temperatures. They also reduce weld cracking and porosity because they narrow the freezing range of the alloys. Zirconium has a powerful grain-refining effect on magnesium alloys. It is thought that because the lattice parameters of zirconium are very close to those of magnesium, zirconium-rich solid particles produced early in the freezing of the melt may provide sites for the heterogeneous nucleation of magnesium grains during solidification. The Elektron ZRE1 alloy exhibits excellent casting characteristics with components being both pressure tight and weldable. Castings are free from microporosity and the tendency to hot cracking in difficult castings is slow [11-14].

2. Experimental

The material for the research was a ZRE1 alloy after semi-continuous casting. The alloy was purchased from Magnesium Elektron, Manchester, UK. The chemical composition of this alloy is provided in Table 1. The rare earth additions were made as mischmetal with the approximate compositions: 50Ce-25La-20Nd-3Pr.

Table 1.
Chemical composition of the ZRE1 alloy in wt.-%

Zn	RE	Zr	Ni	Si	Cu	Mn	Fe	Mg
2.7	3.18	0.53	<0.001	<0.01	<0.01	0.02	0.002	balance

Metallographic specimens were prepared according to the procedure recommended for magnesium-base alloys by Buehler expert system [15]. Specimens for microstructure were taken from central part of cast billet (75 mm diameter) and etched with 3% nitric acid in ethanol. The microstructure was characterized by optical microscopy (Olympus GX-70) and a scanning electron microscopy (Hitachi S3400) equipped with an X-radiation detector EDS (VOYAGER of NORAN INSTRUMENTS). EDS analysis were performed with an accelerating voltage of 15 keV. The phase structure of investigated alloy was identified by X-ray diffraction (JDX-75) using Cu Kα radiation. For a quantitative description of the structure, stereological parameters describing the size and shape of the solid solution grains and phase precipitations were selected. To measure the stereological parameters, a program for image analysis "Met-Ilo" was used [16].

3. Results

3.1. Microstructure

Optical micrograph taken from as-cast alloy is shown in Fig. 1, from which it can be seen that as-cast microstructure of this alloy consist of α-Mg matrix and second phase crystallize along the grain boundaries. SEM observations of analyzed alloy reveal that the grain boundary second-phase shows a kind of massive morphology with bright contrast (Fig. 2). The results of microanalysis by EDS showed that second phase was composed of magnesium, zinc, lanthanum and cerium (point 1, Tab. 2), however, precisely determined of magnesium content in the second phase is difficult, due to interaction between electron beam and magnesium matrix. The content of alloying elements in solid solution is below of quantity sensitivity of X-ray energy dispersive spectroscopy analysis (point 2, Tab. 2).

XRD analyses were performed on selected specimens to identify the phases existing in the alloys studied and the results are shown in Fig. 3. It can be seen that the alloy consisted of two phases, the α-Mg matrix and $Mg_{12}Ce$ intermetallic compound. Based results of chemical microanalysis this second phase can be identified as $Mg_{12}Ce$ with some lanthanum substituting cerium. Moreover, this intermetallic compound contains zinc, which substitutes part of magnesium due to small differences between atomic radiuses of these elements. Therefore, molecular formula of this phase can be written as $(Mg,Zn)_{12}RE$.

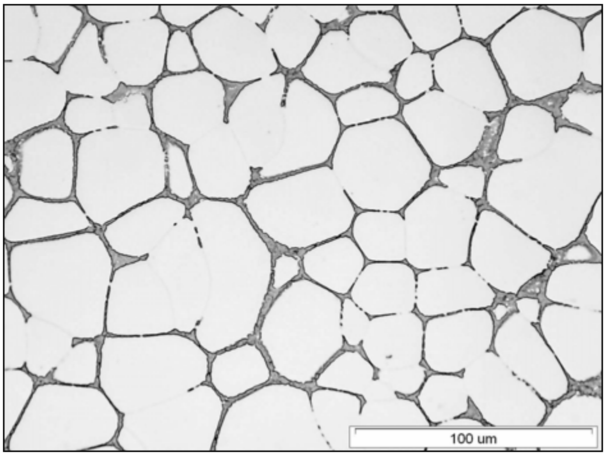

Fig. 1. Optical micrograph of as-cast ZRE1 alloy.

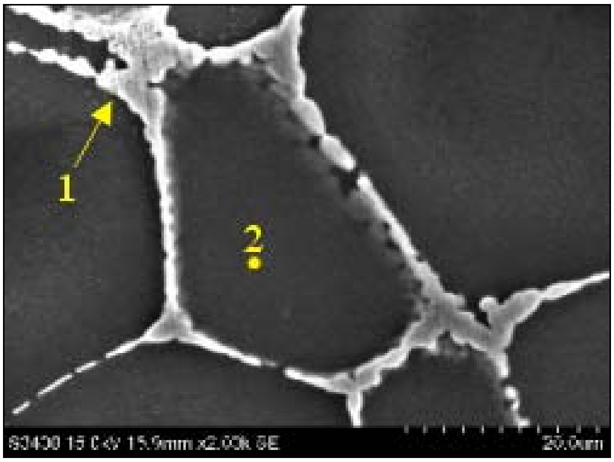

Fig. 2. SEM micrograph of as-cast ZRE1 alloy with marked points of chemical analysis

Table 2.
EDS results of ZRE1 alloy for points from Fig. 2.

Point	Chemical composition, %at.			
	Mg-K	Zn-K	La-K	Ce-K
1	84,41	10,41	1,31	3,87
2	100,00	-	-	-

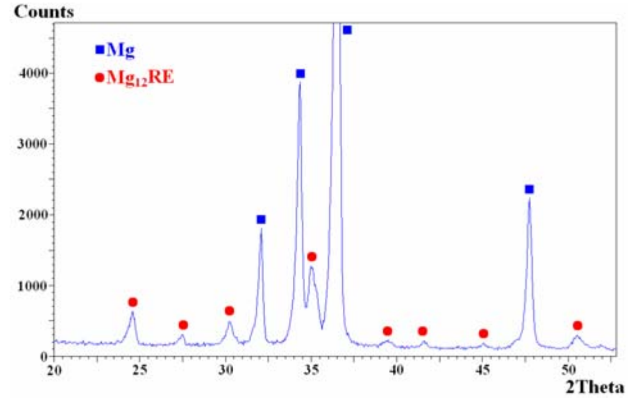

Fig. 3. XRD pattern of ZRE1 alloy.

3.2. Quantitative metallography

The majority of the structural compound occurring in ZRE1 magnesium alloy was clearly visible on images of microstructure etched with 3% nitric acid in ethanol. Therefore, these images are suitable for quantitative evaluation. Simply automatic procedure using the Met-Ilo image analysis program were used to detect of $(Mg,Zn)_{12}RE$. Results of the detection are given in Fig. 4. Area fraction of intermetallic compound in analyzed alloy was 10.8% and coefficient varation was 21%. Measurements were carried out on eleven measure field.

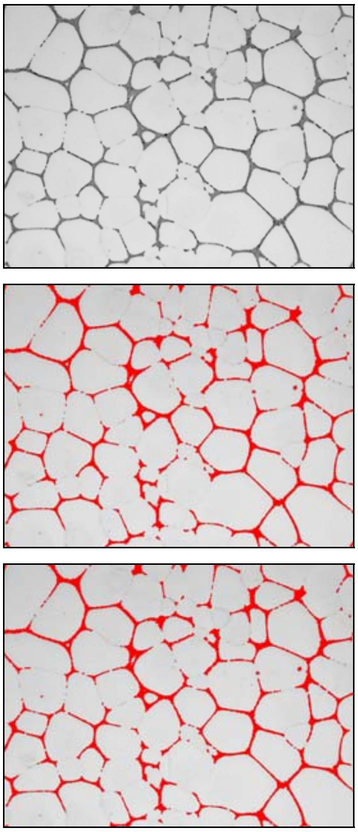

Image A
Initial image

Image b
b = k_means (A,1)

Image c
c = remove small objects (b,2)

Fig. 4. Automatic procedure used to detection of $(Mg,Zn)_{12}RE$.

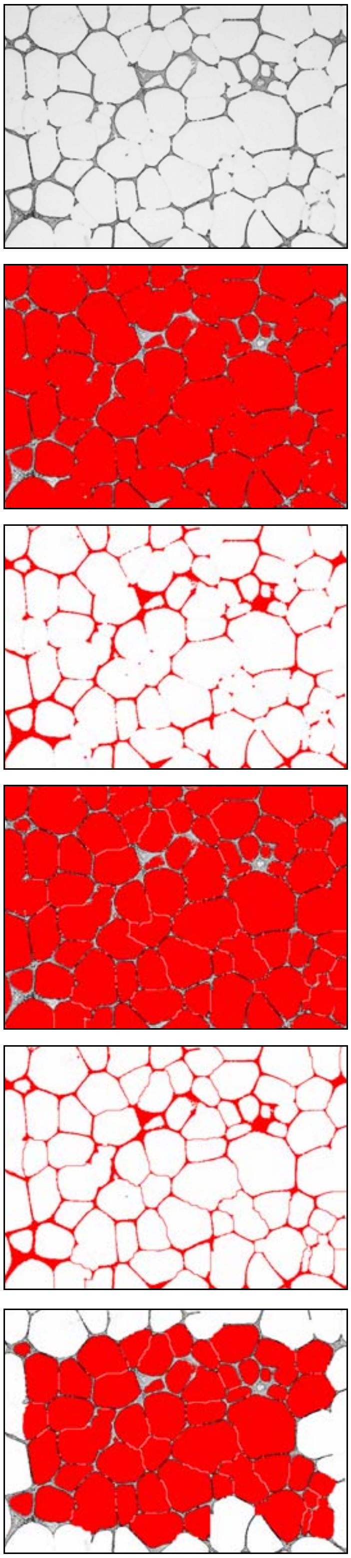

Image A
Initial image

Image b
B = shadow correction (A,8)
b = maximum entropy (B,3)
b = removal objects smaller than 50 pp (b)

Image c
c = not (b)
c = removal objects smaller than 5 pp (c)

Image d
d = modified segmentation (b,3)

Image e
e = not (d)
e = skeleton cut (e,0)
e = manual correction (e)
e = OR(e,b)

Image f
f = not (e)
f = removal margin (f)

Measured image

Fig. 5. Semi-automatic procedure used to determine stereological parameters of grains in ZRE1 alloy.

Etching in different reagents could not on distinctly detection of grain boundaries. The best results were obtained in the case of the sample etched in a solution containing 3% nitric acid and methanol (Fig. 1). Developed procedure (Fig. 5) with modified segmentation and manual correction allows on satisfactory determine stereological parameters of grains in as-cast ZRE1 alloy. The results of quantitative analysis are shown in Tab. 3.

Table 3.
Results of quantitative analysis of grains in ZRE1 alloy.

Parameter	Unit	Mean value	Coefficient variation
Mean area of plane section	μm^2	766	64
Shape factor	-	0.86	21
Maximum diameter	μm	38.9	52
Minimum diameter	μm	27	37

4. Summary

Microstructure of ZRE1 alloys consists of equiaxed grains of magnesium matrix and discontinuous intermetallic network of $(Mg,Zn)_{12}RE$. Etching samples with reagents contains 3 ml nitric acid and 97 ml methanol allows on application automatic procedures to detection of intermetallic compound and grain boundaries. Developed automatic procedures will be useful to future investigations the effect of long-standing heat treatment and influence parameters of sand casting on microstructure of this alloy.

Acknowledgments

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