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Quantitative procedure for evaluation of microstructure of cast Mg-Al-Ca-Sr magnesium alloy

T. Rzychoń
Department of Material Science, Silesian University of Technology, 40-019 Katowice, Poland
Corresponding author. E-mail address: tomasz.rzychon@polsl.pl

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Abstract

In this paper the microstructural characterization of ingot MRI-230D magnesium alloy and quantitative procedure for evaluation of microstructure are presented. The optical and scanning electron microscopy were used to study the morphology of microstructural compounds in this alloy. The X-ray diffraction was used to determination of phase composition. The as-cast microstructure of MRI-230D magnesium alloy containing aluminum, calcium and strontium consists of the dendritic α-Mg and such intermetallic compounds as: Al_2Ca, Al_4Sr and Al_xMn_y. In the purpose quantitative description of microstructure semi-automatic procedures using Met-Ilo image analysis were developed. Prepared semi-automatic procedures allow a fast determination of phase content in MRI-230D alloy using light microscopy and will be useful in the quality control of MRI-230D ingots.

Keywords: Metallography, Microstructure, Cast magnesium alloys, Quantitative description of microstructure.

1. Introduction

Calcium and strontium are important elements in advanced magnesium alloys, which are relevant for weight savings in the automotive industry or other applications [1]. The applications of most common magnesium alloys, such as AZ91 (Mg-9Al-0.8Zn) and AM50 (Mg-5Al-0.5Mn) 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 [2,3]. The search for creep-resistant alloys has led to the development of rare earth containing magnesium alloys, for example AE42 (Mg-4Al-2RE, AE44 (Mg-4Al-4RE) and magnesium alloys with silicon - AS21 (Mg-2Al-1Si), strontium - AJ62 (Mg-6Al-2Sr) or calcium - AX51 (Mg-5Al-1Ca). In these alloys, structure is characterized by the second phases from system Al-Si, Al-RE, Al-Sr or Al-Ca at the grain boundaries which are stable line compound with a relatively high melting point [1-6]. The new series of Mg-Al-Ca-Sr alloys offer excellent creep resistance up to 180°C and low cost compared to magnesium alloys containing yttrium or rare earth metals, which are conventionally used to improve the heat resistance [7]. Typical alloys representative of Mg-Al-Ca-Sr system are commercial MRI-153 and MRI-230D alloys, which are designated for die casting technology. The both alloys was developed by Dead Sea Magnesium and Volkswagen AG for high temperature applications, such as engine blocks and automatic transmission cases, where the operating temperatures can be as high as 250°C and 175°C, respectively. These alloys contain aluminum to ensure their yield strength and castability, while calcium and strontium are added to form intermetallic phases at grain boundaries and in the grain interior to improve creep resistance [8-13]. In the present study, quantitative procedure for determination of phase content in cast MRI-230D magnesium alloy was developed.

2. Experimental procedures

The ingots of MRI230D alloy was the material for the research. The alloys were purchased from Dead Sea Magnesium Ltd. The chemical composition of this alloy is provided in Table 1. The content of iron, nickel and cooper is below 0.001 wt.%.

Table 1.
Chemical composition of the MRI-230D alloy in wt.%

Al	Zn	Mn	Ca	Sr	Sn	Mg
6.8	0.01	0.23	1.91	0.25	0.5	balance

Metallographic specimens were prepared according to the procedure recommended for magnesium-base alloys by Buehler expert system [10]. 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 25 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 [11].

3. Results

3.1. Microstructure

The as-cast microstructure of MRI-230D alloy consists of dendritic α-Mg matrix and interdendritic second-phases distributed at grain boundaries (Fig. 1). SEM observations linked with EDS microanalysis revealed the presence of three sorts phases in the interdendritic regions (Fig. 2). The first one was coarse irregular-shaped eutectic. The results of EDS microanalysis showed that this eutectic phase is composed of mainly aluminium, calcium and magnesium (point 1, Tab. 2). The second one was thin lamellar eutectic phase containing mainly magnesium, aluminum and strontium (point 2, Tab. 2). The third one was globular particles composed of mainly aluminum and manganese (point 3, Table 2). It is worth a note that precise determination of magnesium content in the second phase is difficult, due to interaction between electron beam and magnesium matrix. Calcium and strontium were not detected in solid solution of α-Mg due to their low solubility in magnesium (point 4, Tab. 2). The content of aluminum dissolved in α-Mg is higher than its maximal solid solubility at room temperature. It is connected with high cooling rate during solidification of ingot.

In order to identify the crystalline structure of these intermetallic compounds existing in the as-cast microstructure of the alloys studied, X-ray diffraction analysis has been carried out and the results are shown in Fig. 3. It can be seen that the alloy consists of the α-Mg matrix, Al_2Ca and Al_4Sr compounds. Moreover, a few diffraction lines with low intensity were observed which were not positively identified. Presumably, these peaks are produced by particles containing aluminum and manganese (Al_xMn_y phase).

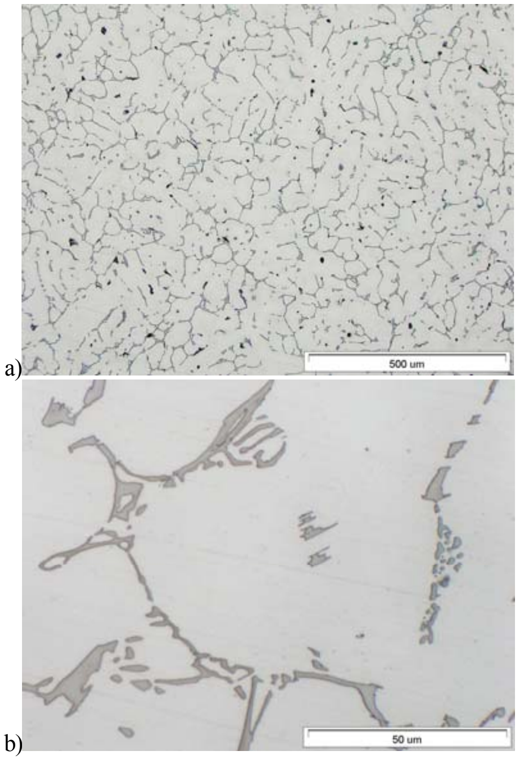

Fig. 1. The optical micrographs of MRI-230D alloy.

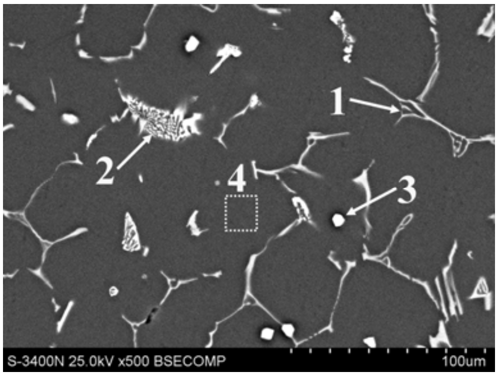

Fig. 2. BSE image of MRI-230D alloy.

Table 2.
Average compositions of the intermetallic compounds and solid solution in MRI-230D alloy analyzed by EDS (SEM) – from Fig. 2.

Point	Composition (at.%)				
	Mg	Al	Ca	Mn	Sr
1	28.3	52.1	18.8	-	0.8
2	41.6	47.0	1.7	-	9.8
3	9.0	49.6	-	41.4	-
4	96.3	3.7	-	-	-

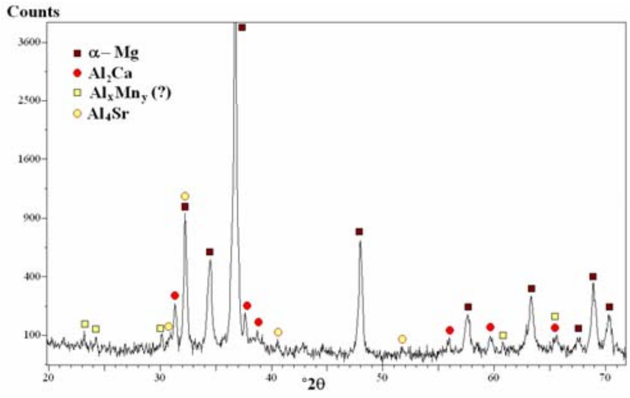

Fig. 3. XRD pattern of MRI-230D alloy.

3.2. Quantitative metallography

The majority of the structural compounds occurring in MRI-230D magnesium alloy were clearly visible on images of microstructure received using a bright field technique and without etching. Therefore, these images are suitable for quantitative evaluation.

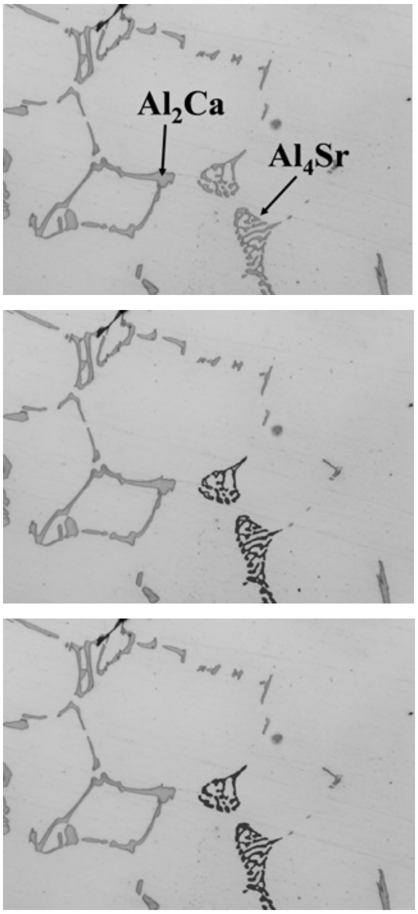

Image A
Initial image

Image b
B = normalization (B)
b = binarisation (B, 90,145)
b = manual correction (b)
b = filling holes (b)

Image c
c = removal objects smaller than 5 pp (b)

Fig. 4. Semi-automatic procedure used to detection of Al_4Sr phase.

Simple semi-automatic procedures using the Met-Ilo image analysis program were used to detection of intermetallic phases in investigated alloy (Fig. 4-6). In these figures grey images are designated in capital letters and binary images with small letters. In brackets the letter indicates analyzed image, digit means value of step for image transformation that was used in Met-Ilo program [11].

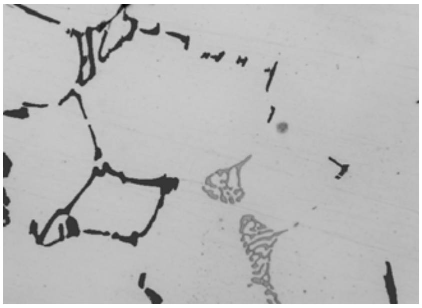

Image A
A = normalization
a = binarisation (A, 100,160)
a = manual correction (a)
a = filling holes (a)
a = removal objects smaller than 5 pp (a)

Fig. 5. Semi-automatic procedure used to detection of Al_2Ca phase.

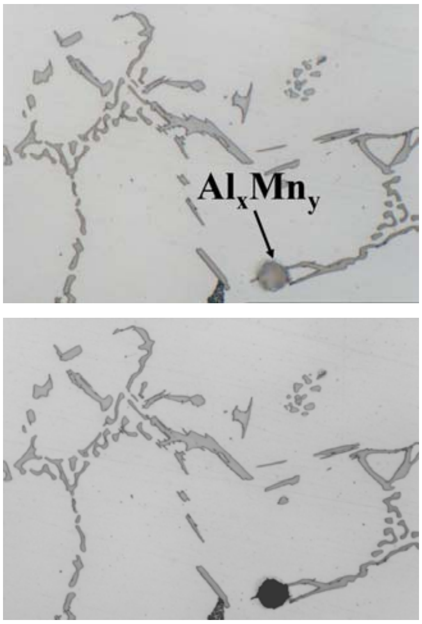

Image A
Initial image

Image b
B = normalization (B)
b = binarisation (B, 0,155)
b = manual correction (b)
b = erosion (b,1)

Fig. 6. Semi-automatic procedure used to detection of Al-Mn compound.

When the light microscopy is used to estimation of phase content in MRI-230D alloy, the most appropriate magnification for correct detection of second phases in MRI-230D alloy is 500x. The results of quantitative analysis are shown in Tab. 3. It can be seen that the dominant intermetallic compound is Al_2Ca Laves phase. The low values of the variation coefficient of area fraction $\nu(A_A)$ indicates that Al_2Ca and Al_4Sr phases are homogenous arrangement in microstructure. The low values of shape factor ξ of these compounds correspond to their irregular shape. In the case of Al_xMn_y phase the high value of shape factor indicates on near-globular morphology.

Table 3.
Results of quantitative analysis in MRI-230D magnesium alloy.

Parameters	Phase		
	Al_2Ca	Al_4Sr	Al_xMn_y
A_A [%]	6.1	1.5	0.6
$\nu(A_A)$	35.1	47.3	105
ξ	0.43	0.52	0.86

4. Summary

The as-cast microstructure of MRI-230D alloy consists of dendritic supersaturated α-Mg matrix, coarse irregular-shaped eutectic α-Mg+Al_2Ca, thin lamellar eutectic α-Mg+Al_4Sr distributed in the interdendritic regions and globular particles of Al_xMn_y compound inside the grains. The dominant intermetallic compound is Al_2Ca and its area fraction is 6.1%.

The performed investigations have shown that the detection of the particles occurring in the MRI-230D magnesium alloy can be performed on unetched samples. Difference in grey level between precipitates is sufficient for detection of Al_2Ca, Al_4Sr and Al_xMn_y phases. Therefore simple semi-automatic procedure using the Met-Ilo image analysis program can be applied to estimation of phase content in MRI-230D alloy. These procedures can be used in the control of quality of ingots, sand and squeeze casts.

Acknowledgements

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