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Structural and quantitative analysis of die cast AE44 magnesium alloy

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Materials

ABSTRACT

Purpose: The main objective of this study was development of determination of phase fraction methodology in cast magnesium alloy containing aluminum and rare earth elements.
Design/methodology/approach: The study was conducted on magnesium alloy containing 4 %wt. aluminum and 4 %wt. mixture of rare earth elements (mischmetal) in the as-cast condition. The mischmetal includes cerium, lanthanum, neodymium and praseodymium. In this study, several methods were used such as: optical light microscopy, quantitative metallography, scanning electron microscopy and X-ray diffraction. The Rietveld method with Hill and Howard procedure was applied for determination of lattice parameters and phase abundance.
Findings: The microstructure of investigated alloy consists of α-Mg solid solution, globular, lamellar and acicular precipitations of $Al_{11}RE_3$ and Al_2RE phases. The results show that the accurate determination of phase contents in AE44 alloy can not perform using quantitative metallography. In this purpose X-ray investigations should be applied.
Research limitations/implications: Developed methodology will be used to quantitative phase analysis of investigated alloy after creep tests and die cast with different parameters.
Practical implications: AE44 magnesium alloy is used in automotive industry. Moreover, this alloy has a new potential application and results of investigations may be useful for preparing optimal technology of die casting.
Originality/value: Procedure described in this paper may be useful as the best experimental techniques for quantitative phase analysis of the intermetallic phases occurring in the AE series magnesium alloys.
Keywords: Metallic alloys; Magnesium alloys; Microstructure; X-ray diffraction

1. Introduction

Magnesium alloys are widely employed in aerospace, automotive and electronic industries, everywhere where weight reduction is essential. In fact, they exhibit low density, high specific strength and excellent machinability [1÷6]. Cast magnesium alloys containing aluminum are used in several automotive applications such as covers, door structures, seat frames, valve covers, wheels, housings and frames [7]. More effective weight reduction could be achieved by applying magnesium alloys to powertrain parts. Powertrain components like automatic transmission cases operate at elevated temperatures and typical Mg-Al alloys (AM, AZ sereies) are not sufficient to these applications [8]. Mg-Al-RE alloys in which RE is added in form of Ce-rich mischmetal (50%Ce-25%La-20%Nd-5%Pr) have been systematically developed to simultaneously satisfy the two requirements of high heat resistance and excellent die-casting properties [9-11]. Among these alloys, new Mg-4Al-4RE (AE44) alloy combines excellent die-casting properties with satisfactory creep resistance. Consequently, AE44 alloy is being considered for structural components such as automotive front engine cradle. The microstructure of the alloy includes precipitates of intermetallic phases such as $Al_{11}RE_3$, Al_2RE, Al_3RE and particles from Al-RE-Mn system [11-12]. These precipitates are difficult to distinguish for optical and scanning electron microscopy and it cause difficulties in quantitative description of microstructure of AE44 alloy. Therefore, the best solution can be applying of X-ray diffraction method for quantitative phase analysis.

The X-ray diffraction methods are of great importance in the microstructure characterization of multiphase materials. The Rietveld method is the most popular and widely used technique for determination of structure and quantitative phase analysis directly from whole X-ray, synchrotron or neutron powder diffraction. Moreover, this method is very useful tool in the verification of the qualitative phase compositions [13-17].

The aim of the work was to study the suitability of the Rietveld method to determining phase concentrations in as cast AE44 magnesium alloy and also in the lattice parameters refinement.

2. Experimental procedures

The samples were obtained using hot chamber die casting machine. The melt and die temperatures for investigated alloy were 680 °C and 150 °C, respectively. The plunger velocity in the second phase was 350 cm/s. Chemical composition of AE44 alloy is presented in Table 1. The rare earth additions (RE) were made as mischmetal with the approximate compositions: 50 wt% cerium, 26 wt% lanthanum, 15 wt% neodymium and 3 wt% praseodymium. Specimens for microstructure studies were mechanically polished using standard methods, etched with reagent contains 10 ml nitric acid and 90 ml water. The microstructure was characterized by optical microscopy (Olympus GX-70) and a scanning electron microscopy (Hitachi S4200) equipped with an X-radiation detector EDS. EDS analysis were performed with an accelerating voltage of 15 keV.

Table 1.
Chemical composition (wt. %) of experimental alloy

Alloy	Composition, wt.%				
	Mg	Al	Mn	Si	RE
AE44	Bulk	4.15	0.39	0.03	4.01

X-ray diffraction patterns were collected using X-Pert Philips diffractometer equipped with curved graphite monochromator on diffracted beam and with the following slits (in the sequence form Cu tube counter); Soller (2°), divergence (1/2°), antiscatter (1/2°) and receiving (0.15 mm). The X-ray data collection was performed for 10-120° 2θ range with 0.04° step. The Rietveld analysis was performed applying DBWS-9807 program that is an update version of the DBWS programs for Rietveld refinement with PC and mainframe computers [19]. The pseudo-Voigt function was used in the describing of diffraction line profiles at Rietveld refinement. The R_{wp} (weighted-pattern factor) and S (goodness-of-fit) parameters were used as numerical criteria of the quality of the fit of calculated to experimental diffraction data.

The quantitative phase analysis was performed using the relation proposed by Hill and Howard [15,18-19]. X-ray diffraction investigations were performed on the solid samples received from of die-cast material.

3. Results and discussion

3.1. Microstructure of AE44 alloy

Fig. 1 is a LM micrograph taken from the die-cast specimen of the investigated alloy, from which matrix and precipitates with diversified morphology can be clearly seen.

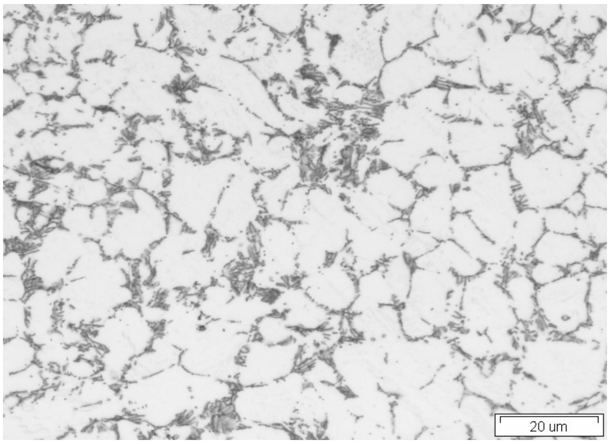

Fig. 1. Microstructure of core region of die-cast AE44 alloy

Table 2.
Chemical composition (at. %) of precipitates in AE44 alloy

Phase	Mg	Al	La	Ce	Nd	Al/RE
A	80.3	15.7	1.9	2.1	-	3.9
B	82.7	12.4	1.8	2.8	0.3	2.5
C	97.9	2.1	-	-	-	-

SEM observations (Fig. 2) revealed that the microstructure of die-cast AE44 alloy consisted of fine grains of α-Mg matrix and two sorts of tiny precipitates. The first one was with lamellar morphology (phase type A, Tab. 2, Fig. 2). The second was coarse with globular shape (phase type B, Tab. 2, Fig. 2). Micro-zone compositions analysis was performed on these white phases by EDS, and the results show that they contain aluminum and rare earth elements, cerium, lanthanum and neodymium. The ratio of Al/RE atoms indicates for the two different phases in alloy. In acicular and regular precipitates this ratio is approximately 3.8, whereas in globular particles it is close to 2.5 (Tab. 2). According to XRD analyses, these precipitates should be $Al_{11}RE_3$ and Al_2RE, respectively (Fig. 3). Moreover, a ratio of La/Ce in $Al_{11}RE_3$ phase is higher than in particles of Al_2RE phase. The high amount of magnesium in fine precipitates was caused by interaction between the electron beam and the α-Mg matrix. The aluminum dissolved in α-Mg is higher than its maximal solid solubility at room temperature (point D, Tab. 2, Fig. 2).

3.2. Determining phase contents

Etching metallographic sections in different reagents cannot to distinguish the intermetallic phases, e.g. $Al_{11}RE_3$ and Al_2RE, using classical metallographic technique. Therefore, to estimation of phase abundance in AE44 alloy applied the Rietveld method.

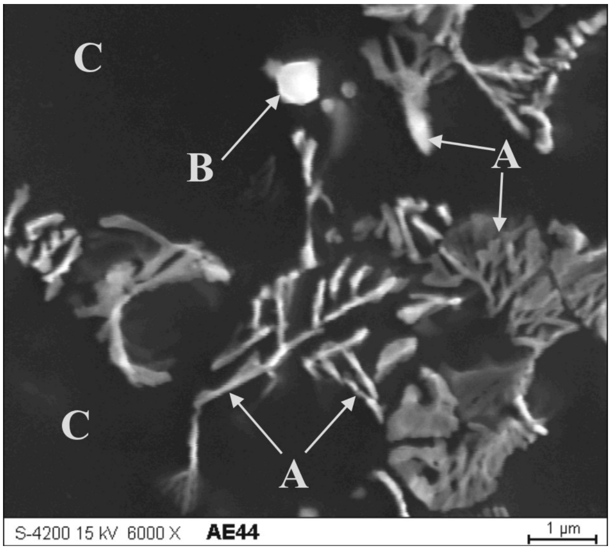

Fig. 2. SEM image of die-cast AE44 alloy

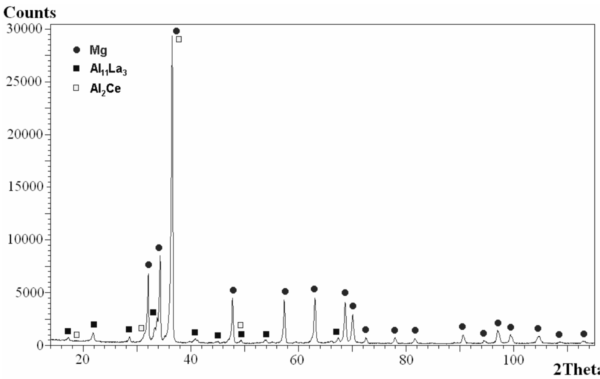

Fig. 3. X-ray diffraction pattern of die-cast AE44 alloy

Analysis of the X-ray diffraction pattern of studied alloy reveals the presence of magnesium, $Al_{11}RE_3$ and Al_2RE phases (Fig. 3), however diffraction lines of Al_2RE phase are very weak and its the strongest peak overlapped with line of magnesium. The peak positions for magnesium, the major phase, are shifted to higher angles, a shift that is consistent with dissolved aluminum in the matrix (D, Fig. 2). The intensities of α-Mg peaks are proportional to data from the ICDD standard (ICDD PDF 35-0821), indicating the random distribution of grain orientations. If the Rietveld method is performed on such a sample showing the proportional intensities to standard data, it is easy to obtain a good structure refinement results, and consequently correct determining phase content in material. Moreover, this X-ray technique was used in order to confirmation results of qualitative phase analysis.

After identification all the phases in alloy, Rietveld refinement was performed, following the turn-on sequence of parameters suggested by Young [16]. Because of the low intensity of the diffraction lines for secondary phases, the positional coordinates and isotropic thermal parameters were fixed to the proper values. Only scale factor, lattice parameters, FWHM (full width at half maximum) parameters, background parameters, profile asymmetry and specimen displacement were refined for this alloy. The phase abundance by weight was calculated by using the relation [19]:

$$W_p = \frac{S_p (Z \cdot M \cdot V)}{\sum_{i=1}^{n} S_i (Z \cdot M \cdot V)} \cdot 100\% \qquad (1)$$

Where:
W_p - the relative weight fraction of phase p in the mixture of n phases (wt.%), S - the Rietveld scale factor, Z - the number of formula unit per units cell, M - the mass of the formula unit (in atomic mass units) and V - the unit cell volume.

Table 3.
Phase contents and their lattice parameters for die-cast alloy

Phase	Space group	Contents [wt.%]	Lattice parameters [Å]	
			Rietveld	ICDD
Mg	P6$_3$/mmc	92.8	a_0= 3.2043(6) c_0= 5.203(1)	a_0= 3.209 c_0= 5.211
$Al_{11}RE_3$	Immm	5.8	a_0= 4.4089(9) b_0=10.161(2) c_0=13.113(3)	a_0= 4.431 b_0= 10.13 c_0= 13.142
Al_2RE	Fd-3m	1.4	a_0= 8.045(2)	a_0= 8.052

The information on the phase concentration obtained from Rietveld refinement is presented in Table 3. It is obviously, that the magnesium is the main component of the sample (92.8 wt.%). The content of $Al_{11}RE_3$ phase (5.8%) is above four times higher than the content of Al_2RE phase (1.4%). These phases are responsible for good creep resistance of AE44 alloy, especially of Al_2RE [10,11] and determining their contents may be useful in future researches the influence phase compositions on the structural stability and creep resistance. The values of lattice parameters determined by Rietveld method (the accuracy in their determination found using alumina plate SRM 1976 standard is ±0.015%) and these found in ICDD files given in Table 3. The lattice parameters of matrix are lower than these ones from ICDD card. It is consistent with formation solid solution of alloy elements, mainly aluminum, in magnesium. Aluminum atoms have smaller atomic radius than the magnesium atoms and caused contraction of crystal lattice of matrix. Also the lattice cell parameters of the secondary phases are changed compare to data obtained from ICDD cards. It is probably consistent with their chemical composition and substituting of cerium, lanthanum and neodymium atoms in their crystal lattice.

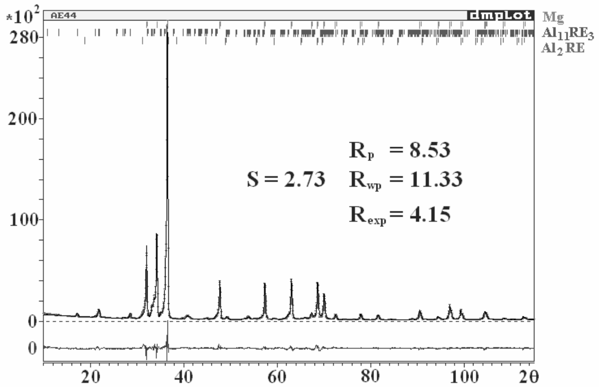

Fig. 4. X-ray diffraction pattern fitting by Rietveld method

The Rietveld refinement plot of the die-cast specimen is presented in Fig. 4. Owing to the presence in the investigated alloy of three phases with one phase of low symmetry and the presence strongly asymmetry of reflection caused probably by in homogeneity chemical composition of solid solution, the fitting of calculated pattern to the experimental one seems to be sufficient.

4. Conclusions

1) Microstructure of AE44 alloy after hot-chamber die-casting consists of α-Mg matrix and precipitates of $Al_{11}RE_3$ and Al_2RE intermetallic phases.
2) The decreasing lattice parameters of magnesium is consistent with the presence of aluminum atoms in magnesium, due to smaller atomic radius of aluminum compare to magnesium atoms.
3) Diffraction lines of magnesium characterize of strongly asymmetry.
4) The presence of intermetallic phases was confirmed by the Rietveld method, which is powerful tool to analyzing diffraction data and allows the detection phases even in a very small quantity and even if some overlap occurs with the peaks of the main phase.
5) The amount of $Al_{11}RE_3$ and Al_2RE phases in AE44 alloy were 5.8 wt% and 1.4 wt%, respectively.

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