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Procedure of revealing and quantitative description of grain size in as-cast MSR-B alloy

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Summary

The goal of this paper is to work out a procedure of revealing, as well as qualitative and quantitative evaluation of magnesium as-cast MSR-B alloy microstructure. MSR-B alloy contains additions of silver and rare earth elements. It is characterized by high tensile strength and good creep resistance. These alloys are intended for elements working at higher temperatures (up to 250 ^{o}C). Significant stress was put on preparation of metallographic specimens and good conditions of image acquisition for light and scanning microscope. On the basis of testing six etching reagents it was found that the best results from the quality as well as quantity metalography point of view were given by reagent composed of 85% glycol and 15% nitric acid. Analysis of chemical and phase composition of the examinated alloy was performed on samples etched using this reagent. Procedures allowing for significant acceleration and simplification of estimation of quantitative description of α phase's grains and eutectic precipitation were elaborated.

Key words: Microstructure; Casting magnesium alloy; MSR-B magnesium alloy

1. Introduction

Magnesium alloys belong to the lightest construction materials. Because of the small density and high tensile strength, magnesium alloys are used in aircraft, automotive and elektrotechnical industry. Due to good mechanical properties (ultimate tensile strength 180 MPa, yield strength 90 MPa (20^{O}C), 70 MPa (200^{O}C), hardness 47HV) MSR-B alloy is used primarily in construction of aero engines and gear enclosures, as well as rotor heads in helicopters.

The main decisive factor of magnesium alloy's properties is solubility of alloy elements in magnesium. It depends on relative size of atoms, electronegativity and similarity of magnesium crystal structure and element being dissolved [2-4].

Magnesium MSR-B alloy is cast into sand forms. It contains silver, rare earth elements, and zirconium additives. Addition of silver and rare earth element enhances mechanical properties of alloy (high temperature creep resistance) and corrosion resistance. These additions worsen casting ability of the alloy. Rare earth elements solidify in thin temperature scope, which lowers material porosity. Addition of zirconium ensures formation of fine grained microstructure.

2. Material and microstructure revealing procedure

Research was conducted on 15 mm thick discs collected from 80 mm in diameter ingot of MSR-B alloy produced by continuous casting method. Chemical composition of the examined alloy is showed in table 1.

Table 1.
Chemical composition of examined magnesium alloy MSR-B [wt.%]

Ag	RE	Zr	Zn	Si	Cu	Mn	Fe	Mg
2.4	2.5	0.42	<0.05	<0.01	<0.01	<0.03	0.002	balance

The samples were cut out from disc according to the scheme shown in figure 1. Samples were grinded and polished using the authors' own procedure in which some recommendations of Buehler's expert system for magnesium alloys were taken into account [5].

Fig. 1. Ingot sampling scheme

The metallographic specimens prepared this way were etched using 6 etching reagents whose chemical composition and etching conditions are shown in table 2.

Table 2.
Chemical composition of reagents and etching conditions

Nr	Chemical composition of reagent	Comments
1	5 – 20 ml acetic acid 80 – 95 ml H_2O	Submerged for 3 s. in solution in room temperature
2	10 ml HF 90 ml H_2O	Etched till satisfactory contrast was achieved
3	2 ml HF 2 ml HNO_3 96 ml H_2O	Etched till satisfactory contrast was achieved
4	10 do 20 ml HNO_3 90 – 80 ml H_2O	Submerged for 3 s. in solution in room temperature
5	15 ml HNO_3 85 ml glycol	Etched till satisfactory contrast was achieved
6	4,2 g picric acid 10 ml glacial acetic acid 10 ml H_2O 70 ml etyl alcohol	Etched till satisfactory contrast was achieved

The chosen structures obtained using light microscope Olympus GX71 are shown in fig. 2. Quality of the obtained images were rated from computer-aided quantitative metallography requirements point of view. It was found that the best results were obtained by using reagent number 5.

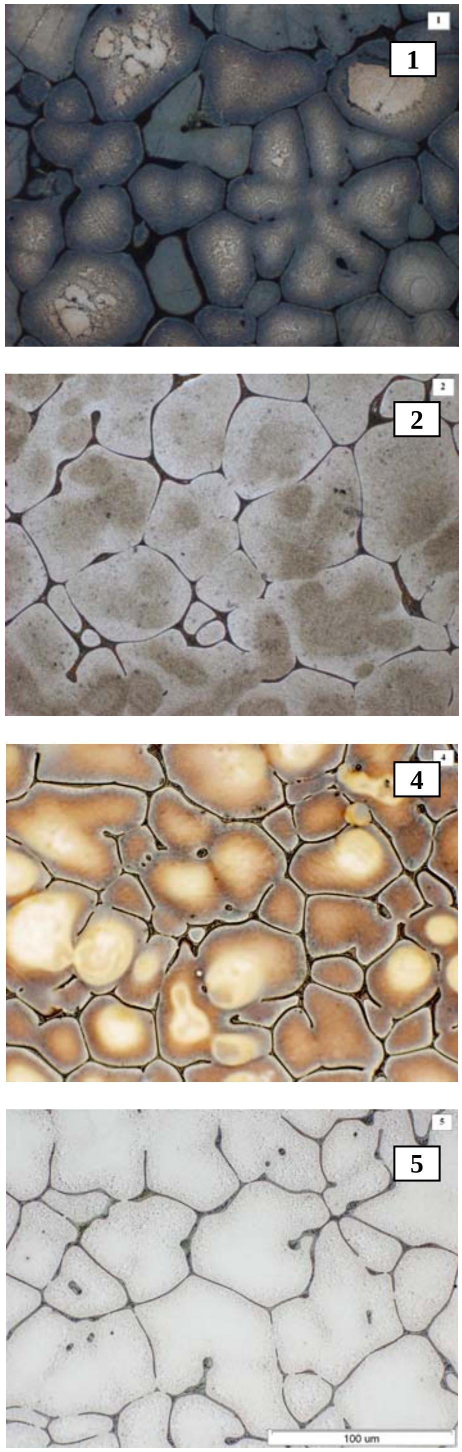
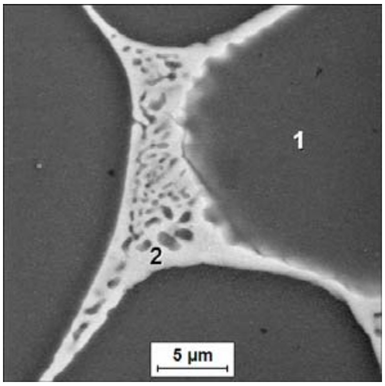


Fig. 2. Structure of MSR-B alloy revealed using various etching reagents. The numbers correspond to these in table 2. Light microscope, magnification 500x

3. Chemical composition of phases in MSR-B alloy

Analysis of chemical composition of phases occurring in the MSR-B alloy in cast state was conducted using a scanning microscope Hitachi S-4200 coupled with EDS Voyager 3500 system. On SE images of this sample, the presence of two morphologically different structural components can be observed: grains with homogeneous grey level and eutectic areas (fig. 3). Results of X-ray microanalysis show, that these components differ considerably in the chemical composition.



Point	Mg	Ag	Nd
1	99.6	0.4	0.0
2	83.7	6.3	10.0

Fig 3. SE images of MSR-B alloy structure in cast state and chemical composition in marked points of structure

These observations and results of XRD analysis conducted on Jeol JDX-75 diffractometer allow to conclude that the structural components in the examined alloy solid solution α are rich in magnesium and eutectic areas of this solution and $(Mg,Ag)_{12}Nd$ phase precipitation (fig. 4). Unequivocal identification of lines visible on this figure (originating probably from Mg_4Ag phase) requires conduction of further research.

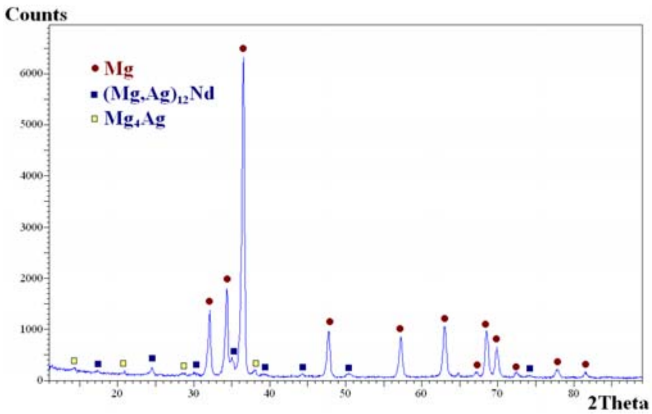


Fig. 4. Image of XRD diffraction of MSR-B magnesium alloy

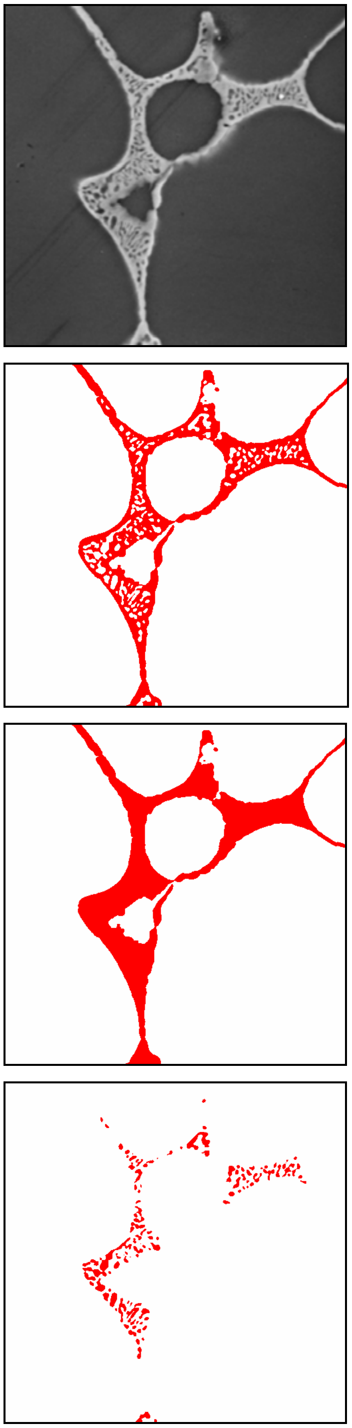


Image A

Initial image

Image B

b=k_means(A,1)

Image C

x=not(b)
y=erosion(x,10)
c=reconstruction(x,y)
c=not(c)

Image D

d=diff(c,b)

Fig. 5. Procedure of determining area fraction of $(Mg,Ag)_{12}Nd$ precipitations in eutectic areas

4. Detection of eutectic areas and α phase grain

A significant variety of grey level and/or colour of α phase grains and eutectic areas visible on images of the structure recorded using a light microscope allows for complete automation of detection process and quantitative estimation of these areas. Structure images taken using scanning microscope at appropriately high magnification can be used to determine area fraction of phases occurring in eutectic areas.

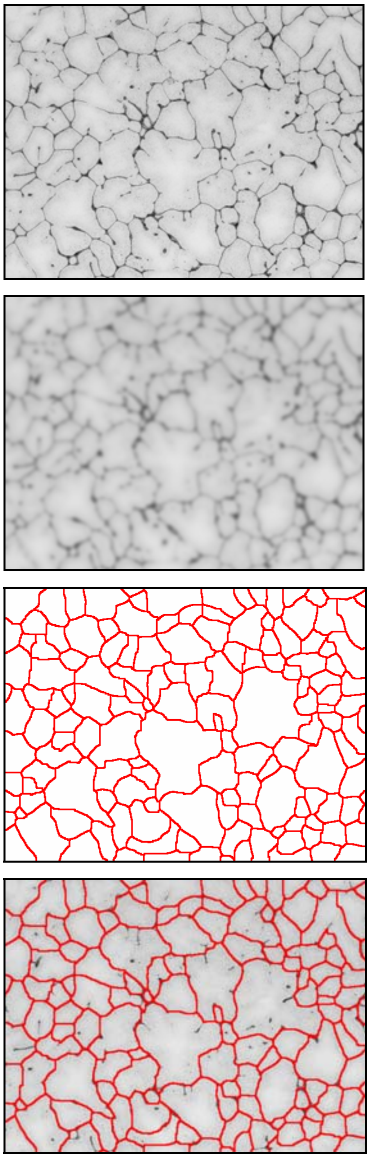


Image A

Initial image

Image B

B=mean(A,5,5,5)

Image C

B=invert(B)
c=watershed(0,2)

Image D

d=manual_corr(c)

Fig. 6. Detection procedure of grain boundaries in MSR-B alloy recorded using scanning microscope

For this goal a procedure whose primary steps are shown in fig. 5, can be used. Area fraction of $(Mg,Ag)_{12}Nd$ phase precipitation in eutectic areas in this case is equal to quotient of area fractions of the detected elements in pictures d and c. In the examined alloy it equals about 20%.

Far more complex is a detection procedure of the α phase grain. It refers to images of the structure recorded using light as well as scanning microscope and is the result of partial revealing of grain boundaries. Various methods of grain boundaries reconstruction have been analysed. The most effective proved to be a procedure using the modified water shade method. But even this procedure didn't allow for full automation of detection process, which is the reason why manual correction of detected grain boundaries is needed before the measurement. The most important stages of the procedure developed is shown on fig. 6.

The discussed procedure was used to determine size of α phase grain on cross-section of the ingot. The measurements were conducted using the MeTilo program [6] in 3 measuring points of the ingot – centre, surface, half of the radius. No significant differences in grain sizes in ingot were confirmed. Average area of the plane section of grains on the whole ingot is 1870 μm^2, and coefficient of variation of area between the evaluated measuring points is only 12%.

Acknowledgments

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