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EDS technique appliance for investigation of laser remelted and alloyed magnesium cast alloys

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ABSTRACT

Purpose: The paper presents the results of the qualitative and quantitative microanalysis of the chemical composition of the MgAl6Zn1 alloy.

Design/methodology/approach: The magnesium alloy has been heat treatment at 505°C for 600 min and ageing at 170°C for 720 min. The study was performed on a transmission electron microscope FEI TITAN company operating at 300kV operating voltage. The qualitative and quantitative microanalysis of the chemical composition of the Mg alloy microareas was examined using EDS.

Findings: Analysis of the results of the concentration of the main alloying elements in the separation test using various magnifications revealed that with an increase in the share of the magnification of the alloying elements. This increase is referred to a linear, the regression coefficient R^2, depending on the test element is in the range 0.84-0.97.

Practical implications: Tested MgAl6Zn1 alloy can be applied among the others in automotive industry but it requires additional researches.

Originality/value: It was demonstrated that the lower magnesium concentration in the EDS results is connected with the increase of magnification induces an effect of X-rays scattering only from the analysed particles and the effect of Mg matrix is limited.

Keywords: Magnesium cast alloy; Laser remelting; X-ray spectroscopy; Stanisz scale; Guillford scale

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PROPERTIES

1. Introduction

Electron beam has a dual wave-particle nature. To make it possible to obtain an image, it is necessary to form a beam, which initially diverges. Electron beam falling on the test material, produces different effects [1-7]. One of the effects used in the microanalysis of the chemical composition is characteristic X-ray [1, 3, 4]. It arises as

a result of energy emission due to electron jumps from orbit with a higher level of energy to free space in orbit with a lower energy level the electron knocked out by the electron from the primary beam. The wavelength and energy of the radiation depends on the atomic number of the element and do not depend on the physical and chemical state of the material. Based on the analysis wavelength or energy of the radiation intensity can be carried out a qualitative and quantitative analysis of chemical composition of the test material micro regions. This analysis is not possible for the lightest elements - hydrogen or beryllium [1, 8].

Analysis of chemical composition by chemical analysis is micro regions in a small volume irrespective of the measuring technique. EDS microanalysis is based on one of the effects of interaction of electrons with atoms of the analyzed material characteristic X-ray emission. The basis of all measurements micro analytical is that the energy (wavelength) and intensity of the radiation characteristic depends on the chemical composition of the test sample micro volume [1, 6].

Depending on the method of detection of X-rays, there are two types of spectrometers: a spectrometer to measure the wavelength of X-rays (called wavelength dispersive spectrometry- in short WDS) and a spectrometer measures the energy of X-rays (called. Energy dispersive spectrometry EDS at a glance or energy dispersive X-ray spectroscopy- in short EDXS) [1, 9].

Characterizing the X-ray microanalysis in general, should be underline, the minimum detection is typically less than 0.1% by mass of the element. With a precision in the range of 1-5% analyzed concentration. The relative measurement error after applying the correction is approximately 2% of the analyzed concentrations [1, 3, 10-17].

2. Material and methods

The investigations were performed of the MgAl6Zn1 alloy with the chemical composition presented in Table 1.

The magnesium alloy MgAl6Zn1 was used for investigations, supplied in form of moulds, with a standard heat treatment carried out, including solution heat treatment at 505°C for 600 min and ageing at 170°C for 720 min.

The examinations of thin foils microstructure and phase identification were carried out on the S/TEM Titan 80/300 transmission electron microscope (TEM), at the accelerating voltage of 300kV. TEM specimens were prepared by cutting thin plates from the material. The specimens were ground down to foils with a maximum thickness of 80μm before 3mm diameter discs were punched from the specimens. The disks were further thinned by ion milling method with the Precision Ion Polishing System (PIPS™), used the ion milling device model 691 supplied by Gatan until one or more holes appeared. The ion milling was done with argon ions, accelerated by a voltage of 15kV, energy and angle are presented in Table 2.

Table 1.
Chemical composition of investigation alloy

The mass concentration of main elements, %							
Material type	Al	Zn	Mn	Si	Fe	Mg	Rest
MgAl6Zn1	5.92	0.49	0.15	0.037	0.007	93.33	0.0613

Table 2.
Ion milling parameters using for polishing

Angle [°]	Energy [KeV]	Time [min]
5.5	3.6	190
3.5	3.0	10

3. Investigation results

X-ray microanalysis based on an analysis of X-ray characteristic emission of the chemical composition makes it possible to examine the micro regions of laser remelted and alloyed magnesium cast alloys. On Fig. 1a-c there are presented microareas of analyzed EDS sample with subsequent magnifications. On the basis of observations there was determined the size of the precipitation of approx. 140 nm.

Based on the analysis results obtained from X-ray scattering analysis (Fig. 2) it was found that the investigated particle is the matrix Mg phase.

On Fig 2 there are presented the investigation results of quantitative microanalysis of the chemical composition of the alloy. The analysis shows that the higher the magnification of the microscope and the measurement area, the main element quantitative analysis of Mg showed that the concentration decreases from 82.6% to 70.4%. In contrast, silicon, and aluminum concentration is increased from the minimum value of 4.01% and 5.11% to 11.2% and 9.5%. respectively. A reduced magnesium concentration in the results of the EDS analysis is associated with the fact, that the increase of magnification induces an effect of X-rays scattering only from the analyzed particles and the effect of Mg matrix is limited (Fig. 3).

The electron beam diameter is in the range of the scale marker length, so the scattered X-Rays are coming from the same configuration of the electron beam and therefore the electron counts.

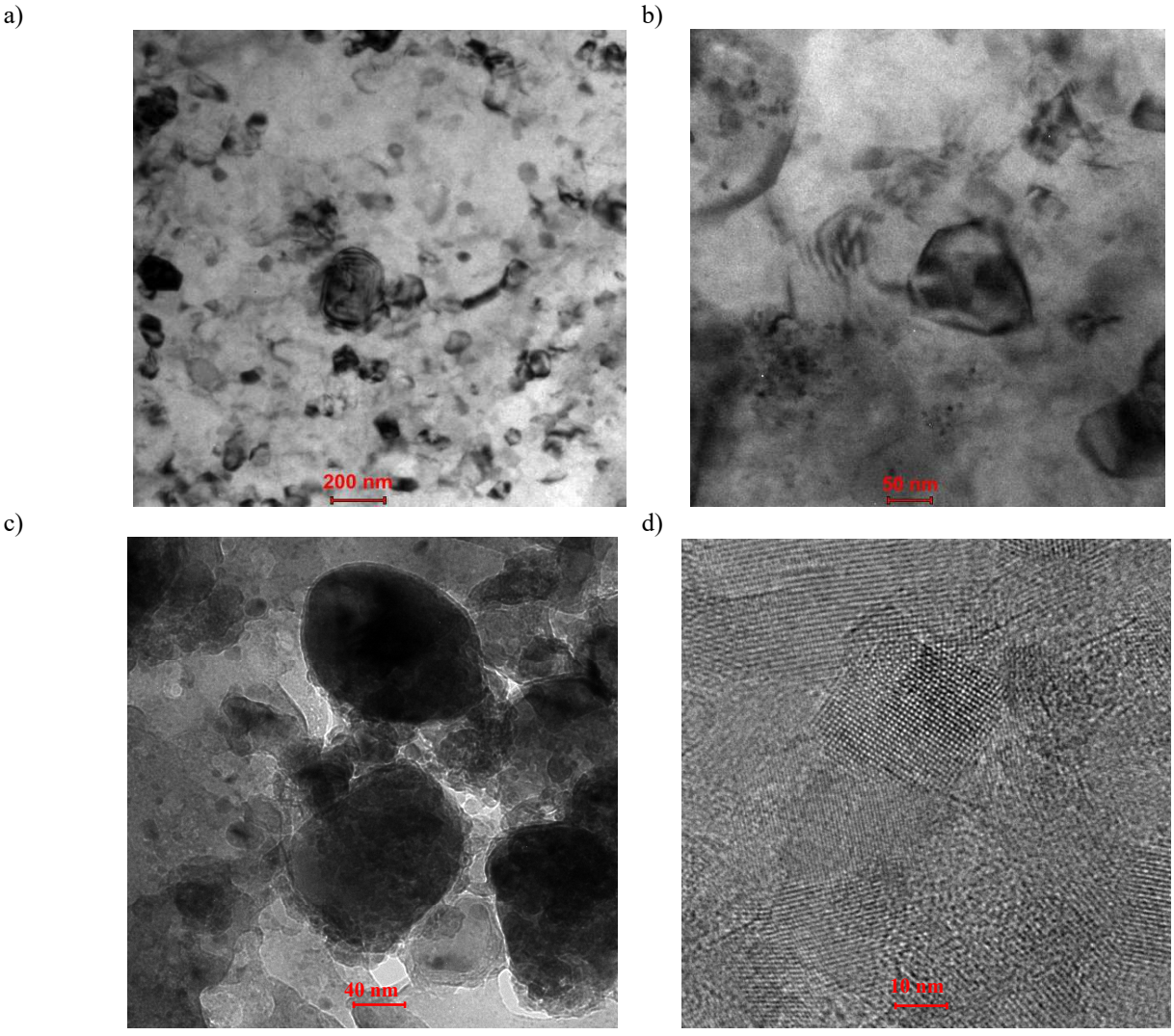

Fig. 1. Subsequent images with increasing magnifications

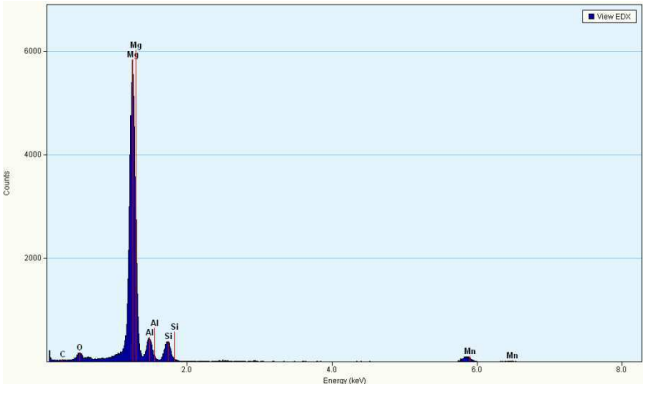

Fig. 2. Results of chemical analysis of micro-regions from the area as shown in Fig. 1b

Analyzing the results of microprobe chemical composition, it was found that the alloy components Si, Al and Mn for multiplying designate a trend line-increase of the concentration of the element used with increasing magnification. The trend line in this case is a linear one, the regression coefficient R^2, depending on the test element is in the range of 0.84-0.97 (Fig. 4 and 5). According to the Stanisz scale [10] this relationship is known as a very high correlation of 0.9 and therefore very well, whereas in the Guillford scale [11] is also very high and the dependency coefficient is defined as significant (more than 0.9).

The angular coefficient of the line in the approximated equations assumes values > 0.03. This means that the increase in the concentration of the analyzed element in the separation is low.

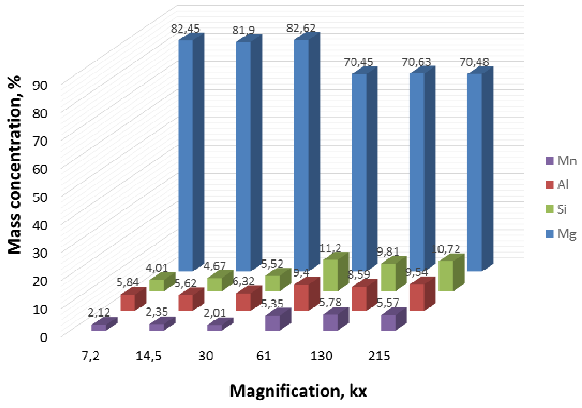

Fig. 3. Mass concentration of the measured elements

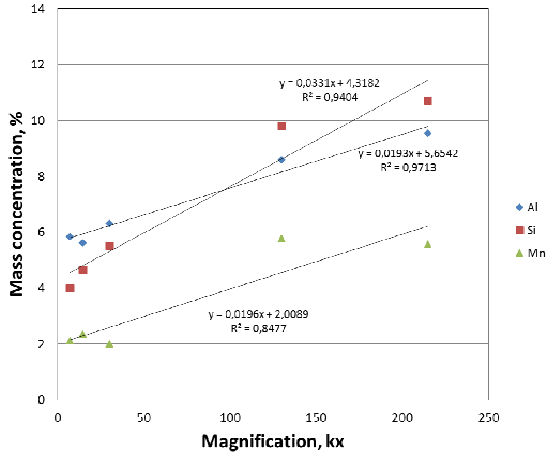

Fig. 4. Mass concentration of the elements found in the following measurements

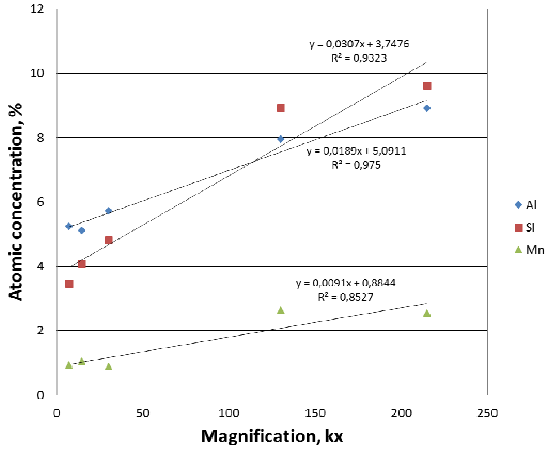

Fig. 5. Atomic concentration of the elements found in the following measurements

4. Summary

The metallographic investigations performed on the transmission electron microscope using the EDX analysis confirm the occurrence of alloying additives like Si, Fe, Mn as well as the carbon and other ceramic powders compounds distributed in the Al matrix.

Lower magnesium concentration in the EDS results is connected with the increase of magnification induces an effect of X-rays scattering only from the analysed particles and the effect of Mg matrix is limited.

In the mass concentration results there is a clear step of the concentration values between 30kx and 60kx, the exactly reason is not now, but it is probably due to the non-uniformity of the matrix chemical composition of the investigated area.

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