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Influence of Al addition on microstructure of die casting magnesium alloys

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Methodology of research

ABSTRACT

Purpose: In the following paper there have been the structure and properties of the MCMgAl9Zn1 magnesium cast alloy as-cast state and after a heat treatment presented.

Design/methodology/approach: A casting cycle of alloys has been carried out in an induction crucible furnace using a protective salt bath Flux 12 equipped with two ceramic filters at the melting temperature of 750±10ºC, suitable for the manufactured material. The following results concern transmission and scanning microscopy, X-ray qualitative and quantitative microanalysis.

Findings: The analysis of the thin foils after the ageing process has confirmed that the structure of the magnesium cast alloy consists of the solid solution α – Mg (matrix) of the secondary phase β – $Mg_{17}Al_{12}$ evenly located in the structure. The structure creates agglomerates in the form of needle precipitations, partially coherent with the matrix placed mostly at the grain boundaries.

Research limitations/implications: According to the alloys characteristic, the applied cooling rate and alloy additions seems to be a good compromise for mechanical properties and microstructures, nevertheless further tests should be carried out in order to examine different cooling rates and parameters of solution treatment process and aging process.

Practical implications: A desire to create as light vehicle constructions as possible and connected with it low fuel consumption have made it possible to make use of magnesium alloys as a constructional material in automotive industry.

Originality/value: The undertaken examinations aim at defining the influence of a chemical composition and precipitation processes on the structure and casting magnesium alloy properties in its as-cast state and after heat treatment with a different content of alloy components.

Keywords: Electron microscopy; Metallography; Magnesium alloys; Structure

1. Introduction

A contemporary technological development makes it necessary to look for new constructional solutions that aim at the improvement of the effectiveness and quality of a product, at the minimization of dimension and mass as well as the increasing of reliability and dimension stability in the operation conditions. For a dozen or so years one can observe a rising interest in the non-ferrous metals alloys including magnesium alloys which are an examination subject in many research and university centres in the country and abroad as well as in major manufacturers of mechanical engineering industry, chemical, power, textile, electronic, paper and aeronautic industries and in particular automotive, shipbuilding, aircraft, sports and even nuclear industries (Fig. 1) [1-12].

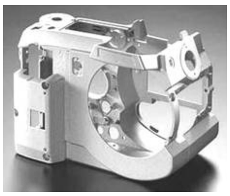
Magnesium alloys which are successfully used for a long time in different industry branches are a combination of low density and high strength. The above features together with low inertia have significantly contributed to the wide use of magnesium alloys in fast moving elements, in locations where rapid velocity changes occur and in products in which lowering a final mass of a product is required. The greatest interest in magnesium alloys was shown and is still shown by an automotive industry [7, 8, 15-25]. A desire to create as light vehicle constructions as possible and connected with it low fuel consumption have made it possible to make use of magnesium alloys as a constructional material in car wheels, engine pistons, gear box and clutch housings, skeletons of sunroofs, framing of doors, pedals, suction channels, manifolds, housings of propeller shafts, differential gears, brackets, radiators and others. A number of companies as well as the use of magnesium and its alloys are still growing. Products made of magnesium and its alloys are still relatively expensive, however customers get high quality products, advanced both in technology and functionality. Thanks to a technological development light section castings, complex in shape, are made that meet high quality requirements [1-6]. Thanks to the introduction of new technologies the costs of processing could be lowered and, above all, the casting methods have been improved. The ways of making elements out of partly crystallized magnesium alloys, moulding in a liquid and solid-liquid state, vacuum casting, pressure die casting on cold- and hot-chamber machines have been worked out. Moreover, the magnesium alloys demonstrate good corrosion resistance, no aggressiveness towards the mould material and low heat of fusion what enables the use of pressure die casting ensuring good shape reproducibility. A general tendency of a present stage of cast materials development is the increase of their plasticity together with the increase of their resistance properties [11-19].

2. Experimental procedure

The investigations have been carried out on test pieces of MCMgAl9Zn1 magnesium alloys in as-cast and after heat treatment states (Table 2) made in cooperation with the Faculty of Metallurgy and Materials Engineering of the Technical University of Ostrava and the CKD Motory plant, Hradec Kralove in the Czech Republic. The chemical composition of the investigated materials is given in Table 1. A casting cycle of alloys has been carried out in an induction crucible furnace using a protective salt bath *Flux 12* equipped with two ceramic filters at the melting temperature of 750±10ºC, suitable for the manufactured material. In order to maintain a metallurgical purity of the melting metal, a refining with a neutral gas with the industrial name of *Emgesalem Flux 12* has been carried out. To improve the quality of a metal surface a protective layer *Alkon M62* has been applied. The material has been cast in dies with betonite binder because of its excellent sorption properties and shaped into plates of 250x150x25. The cast alloys have been heated in an electrical vacuum furnace *Classic 0816 Vak* in a protective argon atmosphere.

Metallographic examinations have been made on magnesium cast alloy specimens mounted in thermohardening resins. In order to disclose grain boundaries and the structure and to distinguish precisely the particular precipitaions in magnesium alloys as an etching reagent a 5% molybdenic acid has been used. The time of the etching for each specimen was between 5-10 s. The observations of the investigated cast materials have been made on the light microscope LEICA MEF4A at magnification 500x as well as on the electron scanning microscope Opton DSM-940 using a secondary electron detection.

The X-ray qualitative and quantitative microanalysis and the analysis of a surface distribution of cast elements in the examined magnesium cast alloy specimens in as-cast and after heat treatment have been made on transverse microsections on the Opton DSM-940 scanning microscope with the Oxford EDS LINK ISIS dispersive radiation spectrometer at the accelerating voltage of 15 kV and on the JEOL JCXA 733 x-ray microanalizer.
Observations of thin foil structure were carried out in the JEM 3010UHR, JEOL transmission electron microscope using an accelerating voltage of 300 kV.


Housing of the photographic camera


Mobile phone


Housing of the computer


Housing of the camera


Wheel bands


Exhaust manifold


Housing of the gearbox


Cylinder head cover

Fig. 1. Elements from magnesium alloys [1-19]

Table 1.
Chemical composition of investigation alloy

The mass concentration of main elements, %								
Al	Zn	Mn	Si	Fe	Pb	Ce	Mg	Rest
9,4	0,84	0,24	0,035	0,007	0,059	0,01	89,4	0,0021

Table 2.
Parameters of heat treatment of investigation alloy

Sing the state of heat treatment	Solution treatment			Aging treatment		
	Temperature	Time	Cooling	Temperature	Time	Cooling
0	As-cast					
1	430	10	water	-	-	-
2	430	10	air	-	-	-
3	430	10	furnace	-	-	-
4	430	10	water	190	15	air

3. Discussion of experimental results

During the process of sand casting there have been mulitcomponent Mg-Al-Zn magnesium cast alloys made with a diversified concentration of alloying components, especially aluminium, as well as Zn and Mn, at a definite plane of micro supplements Pb, Ce, Zr, Sn and Be which ensure obtaining a desirable as-cast and after the heat treatment structure As a result of thin foils examinations on the transmission electron microscope it has been stated that the structure of a newly worked out, experimental magnesium cast alloy MCMgAl9Zn1 after solutioning makes a supersaturated solid solution α – Mg with visible dislocation ranges (Fig.2).

The analysis of thin foils after the process of ageing has validated the fact that the structure of the magnesium cast alloy consists of the solid solution α – Mg (matrix) and an intermetallic secondary phase β – $Mg_{17}Al_{12}$ in the form of needle precipitations with different crystallographic orientations inside the matrix grains (Fig.3).

The differences of contrasts and the crossing atom bands obtained in high resolution pictures of the solid solution range α constituting the alloy matrix and the intermetallic phase β – $Mg_{17}Al_{12}$, explicitly indicate a big defect and micro deformations of lattice caused by the heat treatment. Moreover, the examinations of the thin magnesium cast alloy foils after the ageing process confirm the existence of a high density of crystal structure defects identified as a series of straight and parallel dislocations resembling a network. As a result of the examinations of micro ranges of the thin foils with the method of the x-ray quantitative micro analysis using the EDS dispersive radiation spectrometer one has ascertained that Mg and aluminium are included in the phase which causes the hardening of the magnesium alloys after ageing.

As a result of metallographic investigations made on the light and scanning microscopes it has been confirmed that the magnesium cast alloys MCMgAl9Zn1 in the cast state are characterized by a microstructure of the solid solution α constituting the alloy matrix as well as the β – $Mg_{17}Al_{12}$ discontinuous intermetallic phase in the forms of plates located mostly at grain boundaries (Fig. 4a). Moreover, in the vicinity of the β intermetallic phase precipitations the presence of the needle eutectics (α + β) has been revealed. In the structure of the examined magnesium cast alloys one can observe, a part from $Mg_{17}Al_{12}$ precipitations, turning grey phases, characterized by angular contour with smooth edges in the shape of hexahedrons. Out of the chemical composition examinations with the use of the EDS dispersive radiation spectrometer as well as literature data, one can conclude that it is the Mg_2Si compound which, when precipitating, increases the hardness of castings.

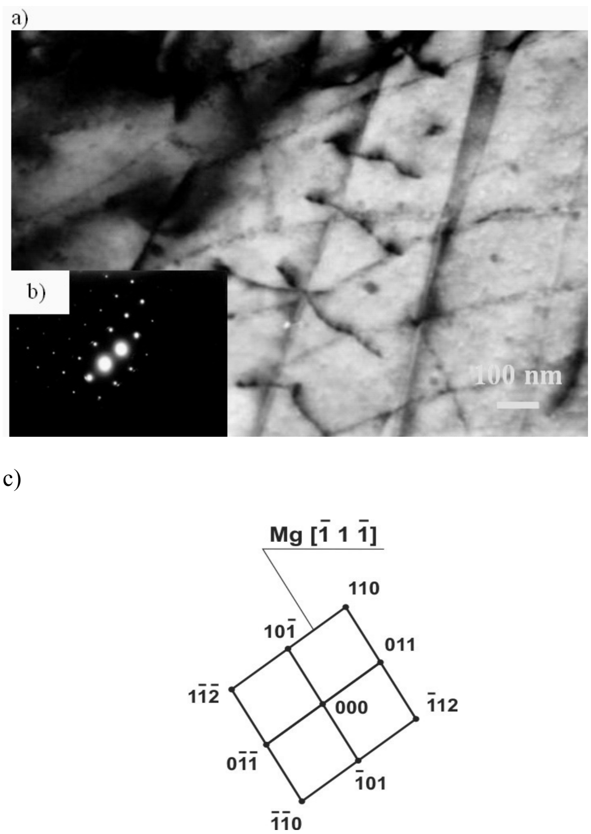
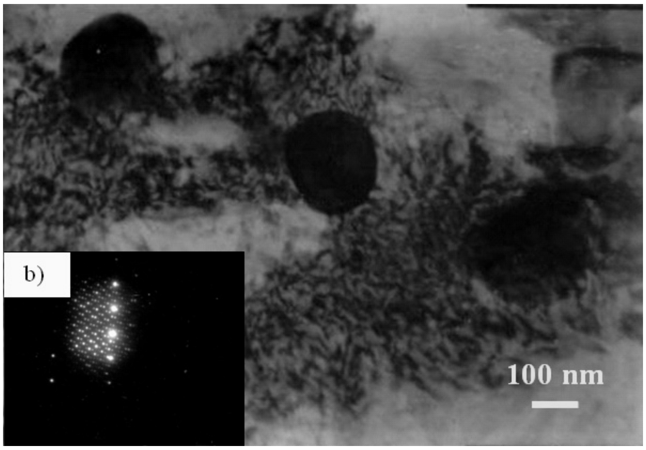


Fig. 2. a) TEM image of the MCMgAl9Zn1 alloy with solid solution α – Mg and visible dislocation ranges, b) diffraction pattern of area shown in a, c) part of solution for diffraction pattern shown in b

a)



c)

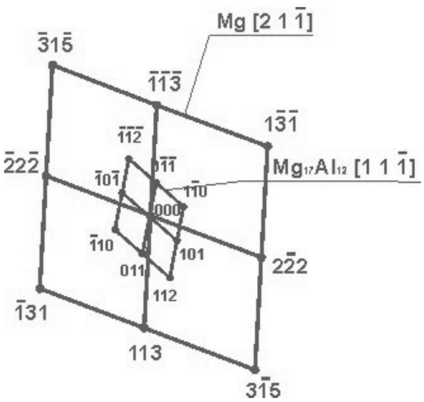
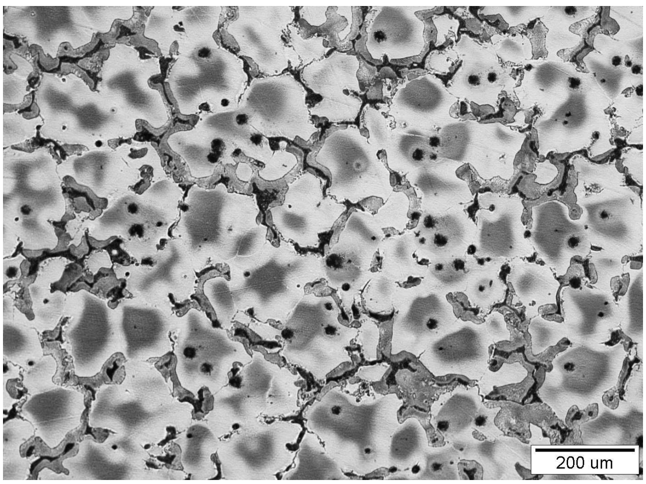
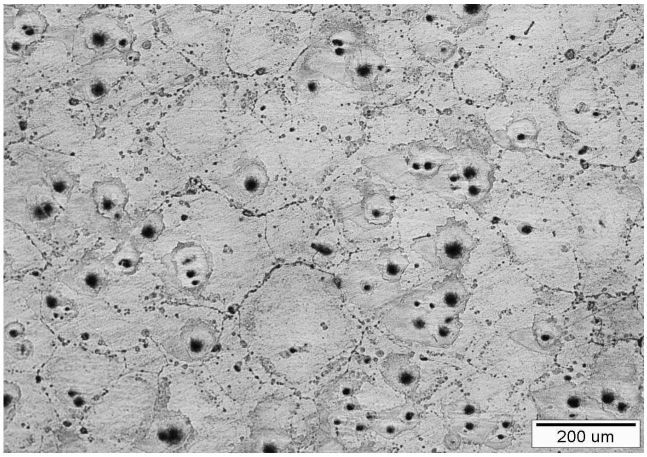


Fig. 3. a)TEM image of the MCMgAl9Zn1 alloy, b) diffraction pattern of area shown in a, c) part of solution for diffraction pattern shown in b

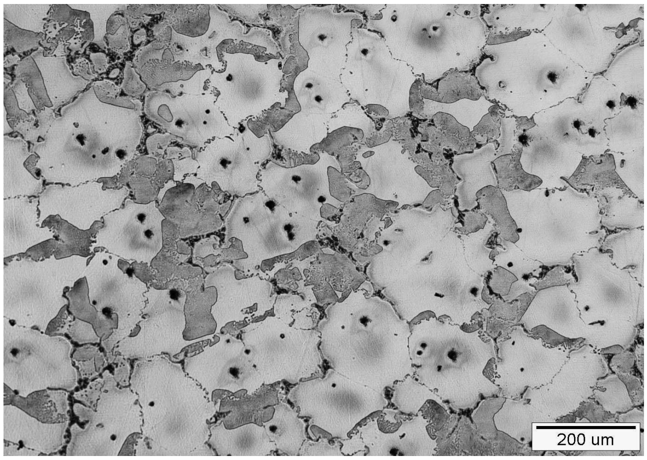
a)



b)



c)



d)

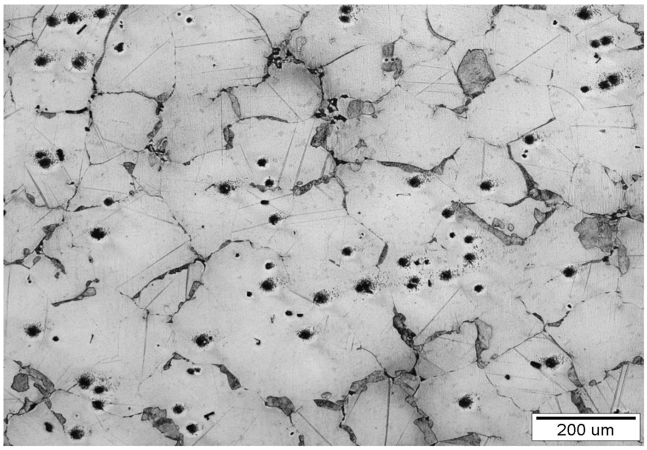


Fig. 4. Microstructure alloy MCMgAl9Zn1: a) without heat treatment – 0, b) after heat treatment – 2, c) after heat treatment – 3, d) after heat treatment – 4, magnification - 200x

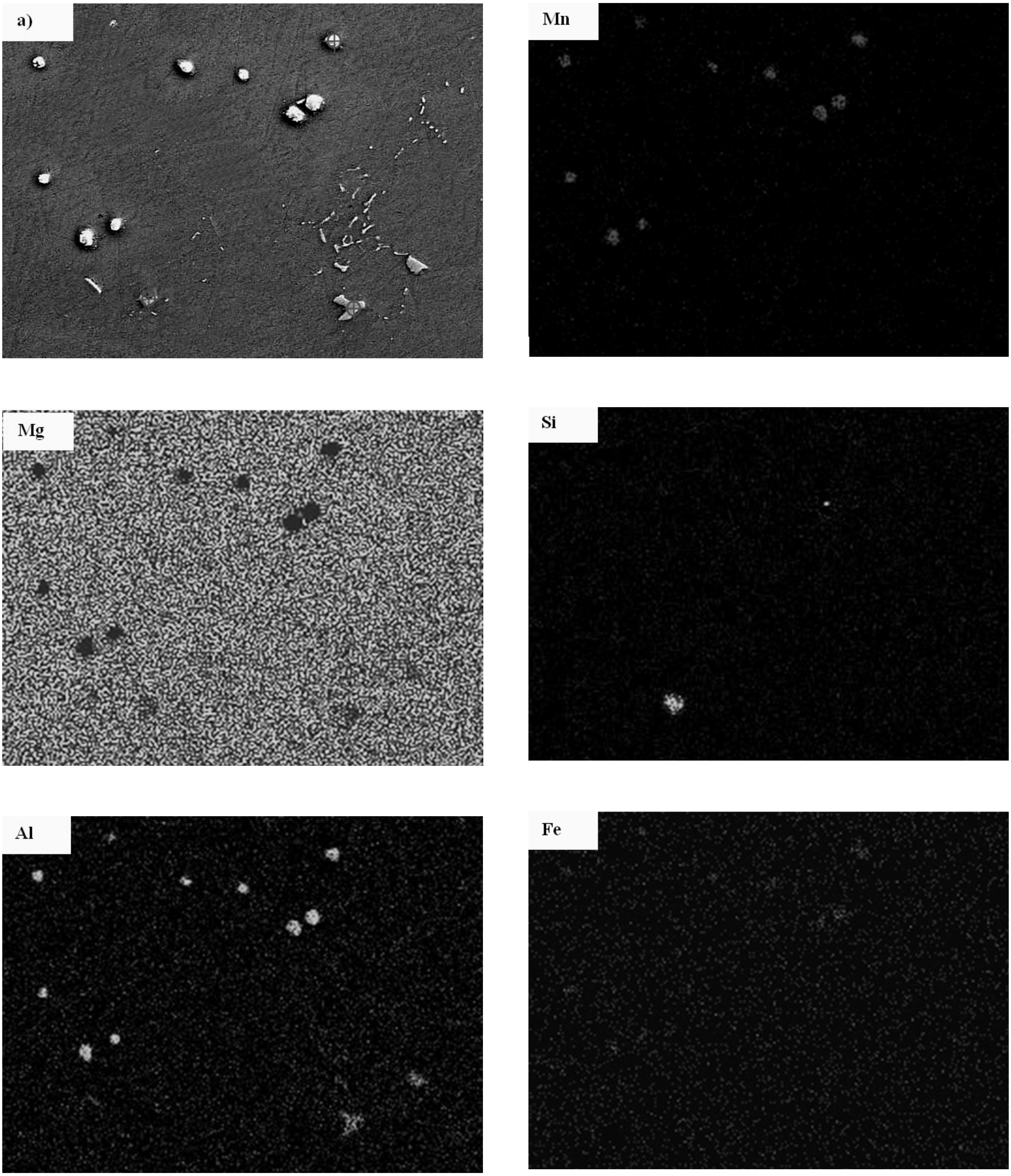


Fig. 5. The area analysis of chemical elements alloy MCMgAl9Zn1 after cooling in the air: image of secondary electrons (a) and maps of elements' distribution

There have appeared, after the process of solutioning with cooling in water and in the air, trace quantities of the β ($Mg_{17}Al_{12}$) phase
and single precipitations of a light grey phase in the structure of the alloy (Fig. 4b). There have not been noticed any locations of eutectic occurrences in the structure. After the cooling bell annealing the structure of the solid solution α with many precipitations of the secondary phase β has been revealed (locations resembling eutectics). The precipitations of the β ($Mg_{17}Al_{12}$) phase, located at grain boundaries and the light grey phase located mostly at the phase β boundary have also been observed. The structure of this alloy is similar to the structure of the as-cast alloy (Fig. 4c). The applied ageing process after the solution heat treatment with cooling in the air has caused the release of the β phase at grain boundaries as well as in the form of pseudo eutectic locations. There have been revealed, in the structure of the material, the parallel twinned crystals extending along the whole grain (Fig. 4d).

As a result of the surface decomposition of elements and the x-ray, quantitative micro analysis made using the EDS energy dispersive radiation spectrometer, the presence of the main alloy additions Mg, Al, Mn, Zn and also Fe and Si included in the magnesium cast alloys in as-cast and after the heat treatment has been confirmed. The information about a mass and atom concentration of particular elements in the pointwise examined micro locations of matrix and precipitations (Fig. 5-8). The chemical analysis of the surface element decomposition and the quantitative micro analysis made on the transverse microsections of the magnesium alloys using the EDS system have also confirmed the evident concentrations of magnesium, silicon, aluminium, manganese and iron what suggests the occurrence of precipitations containing Mg and Si with angular contours in the alloy structure as well as phases with high Mn and Al concentrations that are irregular with a non plain surface, often occurring in the forms of blocks or needles.

Table 3.
Pointwise chemical composition analysis from Fig 5a

Chemical elements	The mass concentration of main elements, %	
	element	atomic
	Analysis 1 (point 1)	
Mg	64,40	67,72
Si	35,29	32,13
Rest	0,31	0,15
	Analysis 2 (point 2)	
Mg	62,95	63,3
Al	35,0	35,10
Zn	1,62	0,69
Rest	0,43	0,91
	Analysis 3 (point 3)	
Al	39,02	53,75
Mn	45,13	34,28
Fe	15,70	11,02
Rest	0,17	0,95

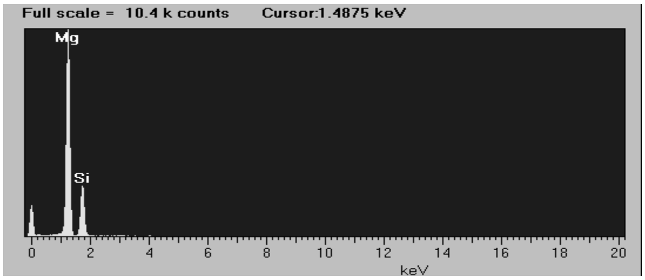


Fig. 6. Spectrum of the pointwise chemical composition analysis analysis 1 (point 1) from Fig 5a

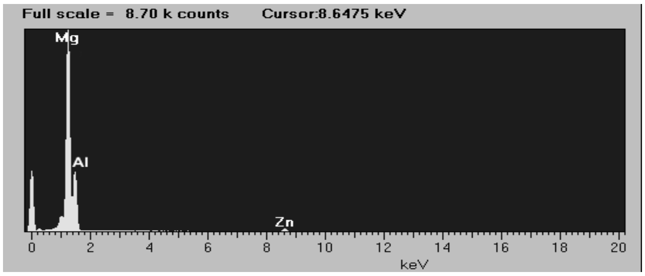


Fig. 7. Spectrum of the pointwise chemical composition analysis analysis 2 (point 2) from Fig 5a

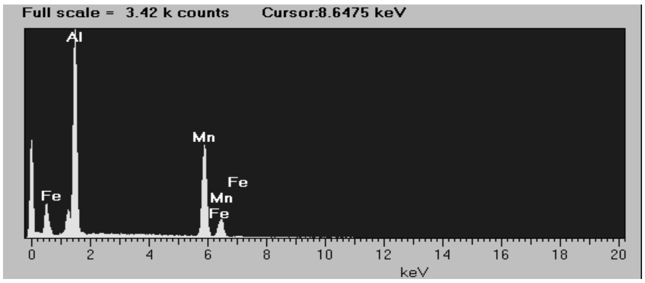


Fig. 8. Spectrum of the pointwise chemical composition analysis analysis 3 (point 3) from Fig 5a

Because the size of particular elements of the structure is, in a prevailing measure, smaller than the diameter of the analyzing beam, the obtained at the quantitative analysis chemical composition may be averaged as a result of which some values of element concentrations may be overestimated. A prevailing participation of magnesium and aluminium and a slight concentration of Zn has been ascertained in the alloy matrix as well as in the location of eutectics and big precipitations that arouse at phase boundaries identified as $Mg_{17}Al_{12}$.

4. Summary

As a result of the examinations of the thin foils made on the transmission electron microscope one has stated that the structure of the magnesium cast alloy MCMgAl9Zn1 after the annealing constitutes a solid solution α – Mg with visible dislocation ranges. The analysis of the thin foils after the ageing process has confirmed that the structure of the magnesium cast alloy consists of the solid solution α – Mg (matrix) of the secondary phase β – $Mg_{17}Al_{12}$ evenly located in the structure. The structure creates agglomerates in the form of needle precipitations, partially coherent with the matrix placed mostly at the grain boundaries. Furthermore, the examinations of the thin foils of magnesium cast alloys after the ageing confirm the appearance of a high density of defects of the crystal structure in the material (Fig.3).

The results of the metallographic examinations made on the light and scanning microscopes confirm the fact that the magnesium cast alloy MCMgAl9Zn1 is characterized by a microstructure of the solid solution α constituting the alloy matrix as well as the $\beta - Mg_{17}Al_{12}$ discontinuous intermetallic phase in the forms of plates located mostly at grain boundaries. Moreover, in the vicinity of the β intermetallic phase precipitations the presence of the needle eutectics (α + β) has been revealed. The applied ageing process after the solution heat treatment has caused the release of the β phase at grain boundaries as well as in the form of pseudo eutectic locations. In the structure of the material, the parallel twinned crystals extending along the whole grain have been revealed (Fig. 4).

The results of the analysis of the EDS chemical composition confirm the presence of the main alloy additions Mg, Al, Mn, Zn and also Fe and Si included in the magnesium cast alloys in as-cast and after the heat treatment**.** The chemical analysis of the surface element decomposition and the quantitative micro analysis made on the transverse microsections have also confirmed the evident concentrations of magnesium, silicon, aluminium, manganese and iron what suggests the occurrence of precipitations containing Mg and Si with angular contours, as well as phases with high Mn and Al concentrations that are irregular, with a non plain surface, often occurring in the forms of blocks or needles (Fig. 5-8)**.**

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