Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 42
ISSUES 1-2
September-October
2010

Investigations of microstructure and dislocations of cast magnesium alloys

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Received 21.05.2010; published in revised form 01.09.2010

Materials

ABSTRACT

Purpose: The microstructures and the dislocation arrangements in the cast magnesium alloy have been investigated using transmission electron microscopy and high-resolution transmission electron microscopy. In this paper are presented also the results of phase morphology investigation of an new developed Mg alloy. Such studies are of great interest for the metal industry, mainly the automobile industry, were the improvement of cast elements quality is crucial for economic and quality reason and depends mainly on properly performed controlling process of the production parameters. There are presented especially the effect of heat treatment on the size and distribution of the precipitation occurred in the matrix.

Design/methodology/approach: The basic assumptions of this work are realised an Universal Metallurgical Simulator and Analyzer. The solidification process itself is analysed using the UMSA device by appliance of the Derivative Thermo Analysis. The thermal analysis was performed at a low but regulated cooling rate in a range of 0.2 ºC to ca. 3 ºC. Cooling curve for the thermal analysis was performed using a high sensitivity thermocouples of the K type, covered with a stainless steel sheath. The data were acquired by a high speed data acquisition system linked to a PC computer. Two different types of samples were used, bulk-cylindrical, and thin-walled cylindrical. Metallographic investigation were made on cross section samples of a engine bloc. Non-equilibrium heating and cooling process conditions were applied to achieve changes in shape and distribution of the phases such as Al_2Cu and Si.

Findings: During the investigation Dislocation networks are found to increase with deformation in all cases. The dislocation networks have been found in the g- Mg17Al12 phase as well as in the matrix in the investigation magnesium alloys. The crystallographic orientation relationship are: (1 01) α-Mg ‖ (10) Mg17Al12 and [11 0] α-Mg ‖ [111] Mg17Al12. Precipitation of the g-Mg17Al12 phase are mostly of the shape of roads, and the prevailing growing directions are the directions <110> α-Mg.

Research limitations/implications: The investigations were performed using standard metallographic investigation as optical, scanning and transmission electron microscopy methods, also electron diffraction methods were applied for phase identification.

Originality/value: The originality of this work is based on applying of regulated cooling rate of magnesium alloy for structure and mechanical properties changes. In this work the dependence between the regulated heat treatment, chemical composition and structure of the investigated magnesium cast alloy on the basis of the structure investigations was presented.

Keywords: Metallic alloys; Electron microscopy; Microstructure; Magnesium alloys; Aging treatment

Reference to this paper should be given in the following way:
T. Tański, L.A. Dobrzański, K. Labisz, Investigations of microstructure and dislocations of cast magnesium alloys, Journal of Achievements in Materials and Manufacturing Engineering 42/1-2 (2010) 94-102.

1. Introduction

Recent years dynamic development, present in the automobile industry, is mostly based on innovative constructional solutions as well modern materials, which directly influence on mass, performances and fuel consumption. The most often pioneer solutions in the field of material engineering are just present in the automobile industry market – there are applied light, strength materials with big projecting potentials [1-5].

One of the basic groups of metal alloys, which allows the realisation of the early mentioned tasks are the magnesium alloys. Magnesium alloys are distinguished by a very useful technological parameter, namely the strength (yield strength $R_{p0.2}$) to density ratio. For the reason of a relative low value of the elasticity modulus magnesium alloys are characterised by a very good vibration dumping ability. Also for this reason these alloys are mainly used for sport equipment as well as application elements in the automobile industry. A huge advantage of the magnesium alloys is the cast possibility of thin-walled large surface elements manufactured with high accuracy and dimensional stability. Moreover magnesium alloys are characterised by extraordinary machinability, even at a low speed rate [6-9].

The demand for the magnesium cast alloys is mainly connected with the development of the automotive industry (Fig. 1). For example, General Motors in their big cars (Savana & Express) use 26.3 kg of magnesium cast alloys, and in smaller cars (Safari, Astro) – 165 kg, Ford F – 150 – 14.5 kg, VW Passat and Audi A4 and A6 from 13.6 to 14.5 kg, Alfa Romeo – 9.3 kg. A further demand for magnesium casts is expected, of up to 50 kg per each car. It is mainly because of the fact that the magnesium casts have got a low density (1700-1900 kg/m^3), and at the same time, their mechanical properties are similar to the aluminium casting alloys [1-5]. Magnesium alloys have got good casting properties and the possible shrinkage porosities or hot micro-cracks can be counteracted by applying alloy additions. By choosing the alloy additions, the mechanical properties or corrosion resistance can be influenced.

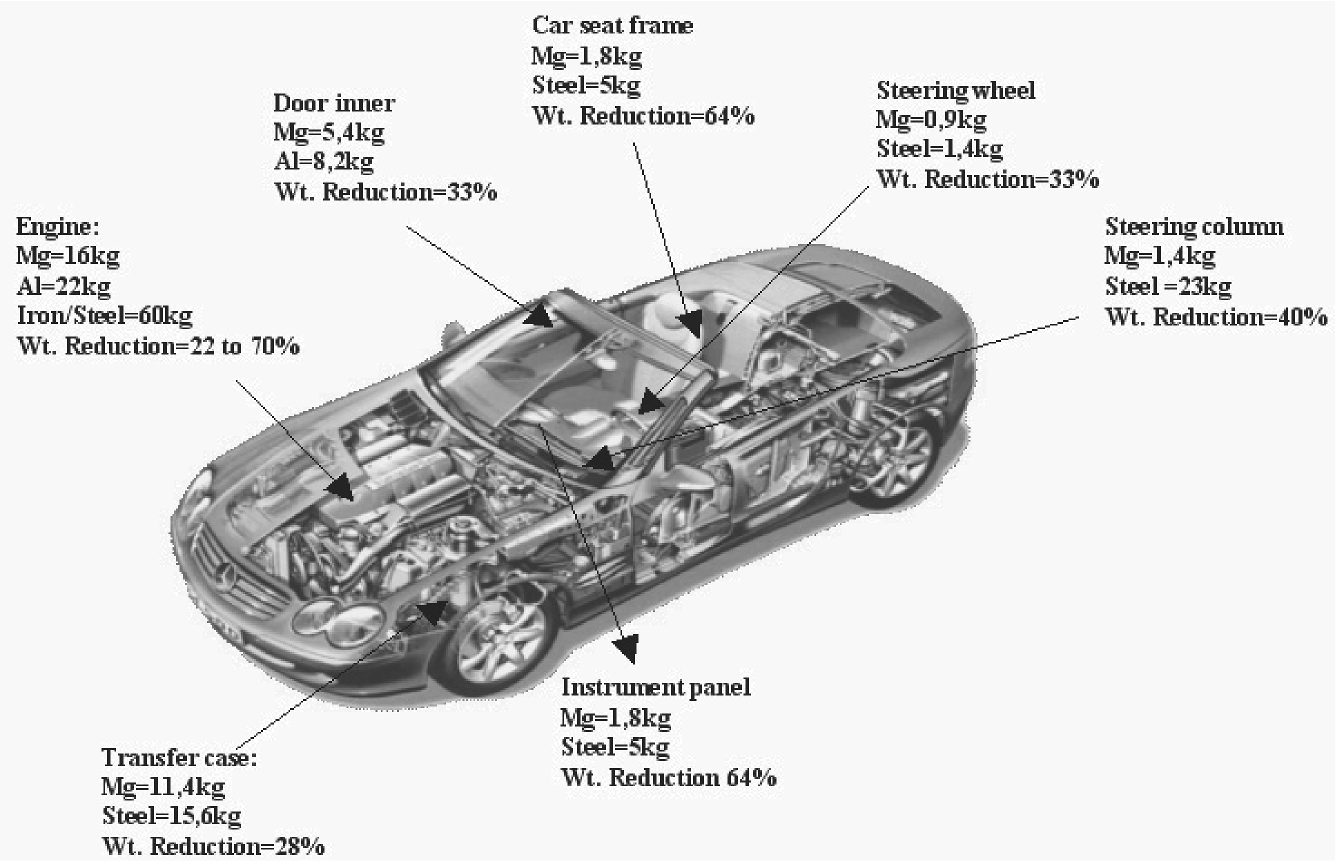

Fig. 1. Some automotive components made of Mg alloy and obtained weight reduction

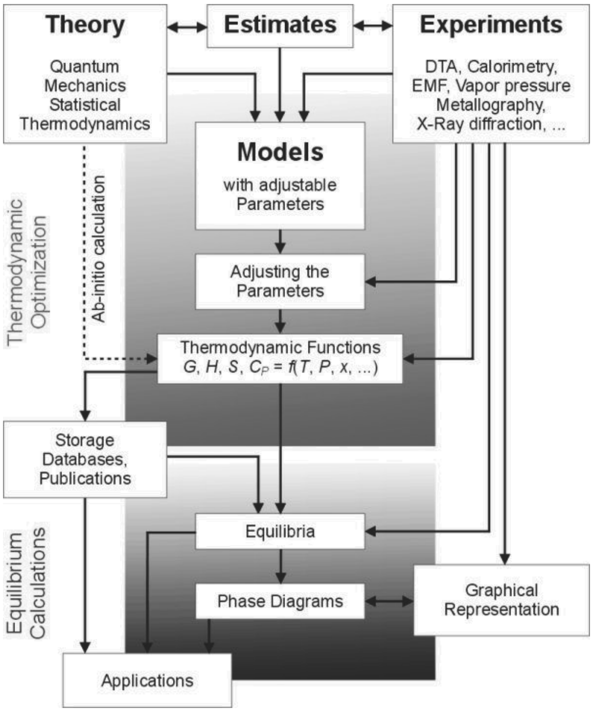

Fig. 2. Thermodynamic relation in theory and praxis

Theoretical relations needed for proper heat treatment conditions are getting from the phase diagrams providing the graphical presentation of the equilibrium state of a material as a function of temperature, pressure, and composition of the components. This is why they are frequently used as roadmaps for alloy design or a better understanding of the processing of materials. The thermodynamic properties of materials, such as the heat of solidification or the chemical activities of components, are also frequently used to understand, for example, metallurgical reactions of materials (Fig. 2). These two aspects, phase diagrams and thermodynamic properties, have been treated separately for a very long time despite the fact that their fundamental interrelations had been established more than a century ago by J. W. Gibbs [11]. A predictive capability allows the extrapolation of thermodynamic descriptions and phase equilibrium calculation from assessed binary systems to ternary, quaternary and higher order systems. Identification of key experiments drastically reduces the necessary experimental effort in multicomponent systems. So also the experiments performed in this work should help to understand the processes occurred during different heat treatment in the studied magnesium alloys.

2. Experimental conditions

The investigations have been carried out on test pieces of MCMgAl9Zn and MCMgAl6Zn magnesium alloys in as-cast and after heat treatment states (Table 1). The chemical composition of the investigated materials is given in Table 2.

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 precipitations in magnesium alloys as an etching reagent a 5% molybdenic acid has been used. The observations of the investigated cast materials have been made on the light microscope LEICA MEF4A as well as on the electron scanning microscope Opton DSM-940 and ZEISS SUPRA 25 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 ZEISS SUPRA 25 scanning microscope. Observations of thin foil structure were carried out in the transmission electron microscope JEM 3010 supplied by JEOL using an accelerating voltage of 300 kV. Thin foils were prepared on the basis of 1 mm thick thin plates cutting out using the electro-erosion method, following by mechanical thinning down to a thickness of. about 0.2 mm, wherefrom after that discs were cut out with a diameter of 3 mm. So prepared samples were again thinned mechanically using the Disc Grinder device to a thickness of ca. 80 µm and finally polished using the PIPS ion milling system from Gatan. Diffraction pattern from Transmission Electron Microscope were solved using the "Eldyf" software. Crystallographic dependence occurred between the phases identified on the thin foil diffraction pattern was analysed using stereographic projection.

Table 1.
Parameters of heat treatment of investigation alloy

Sing the state of heat treatment	Conditions of solution heat treatment		
	Temperature, °C	Time of warming, h	Cooling types
0	As-cast		
	Solution treatment		
1	430	10	Water
	Aging treatment		
2	190	15	Air

Table 2.
Chemical composition of investigation alloy

The mass concentration of main elements, %						
Al	Zn	Mn	Si	Fe	Mg	Rest
9.09	0.77	0.21	0.037	0.011	89.79	0.0915
5.92	0.49	0.15	0.037	0.007	93.3347	0.0613

3. Results and discussion

As a result of metallographic investigations (Figs. 3, 4) made on the optical microscope it has been confirmed that the magnesium cast alloys MCMgAl9Zn1 and MCMgAl6Zn1 in the cast state are characterised by a microstructure of the solid solution α (Fig. 3) (#1) constituting the alloy matrix as well as the $\gamma - Mg_{17}Al_{12}$ discontinuous intermetallic phase in the forms of plates located mostly at grain boundaries (Fig. 3) (#2). Moreover, in the vicinity of the γ intermetallic phase precipitations the presence of the needle eutectics (α + γ) has been revealed (Fig. 3) (#3). The applied ageing process after the solution heat treatment with cooling in the air has caused the release of the γ phase at grain boundaries as well as in the form of pseudo eutectic locations (Fig. 4) (#4) (pseudo-eutectic - γ phase precipitation from solid solution after ageing was determining as similar structure to an eutectic). There have been revealed, in the structure of the material, the parallel twinned crystals extending along the whole grain (Fig. 4).

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 has been confirmed. Both in the alloy matrix as well in the eutectic area or near big precipitates present on phase boundaries identified as $Mg_{17}Al_{12}$ a big concentration of magnesium and aluminium was state. Also and small concentration of Zn was measured, what is presented in Fig. 5.

The chemical analysis of the surface element decomposition made on the transverse microsections of the magnesium alloys have also confirmed the evident concentrations of magnesium, silicon, aluminium and manganese 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 (Fig. 5).

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 $\alpha - Mg$ (matrix) and an intermetallic secondary phase $\gamma - Mg_{17}Al_{12}$ in the form of bulk precipitations (Figs. 6-9).

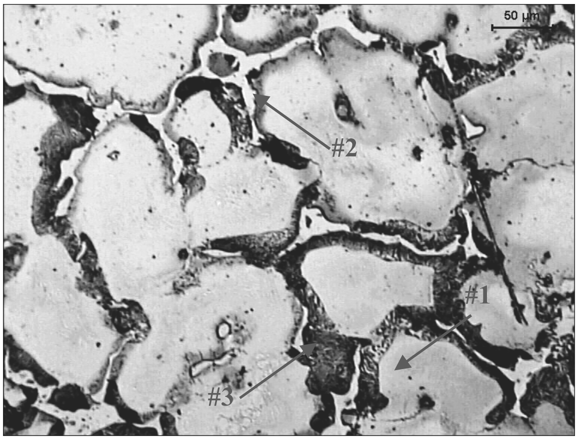

Fig. 3. Microstructure alloy MCMgAl9Zn1 - without heat treatment

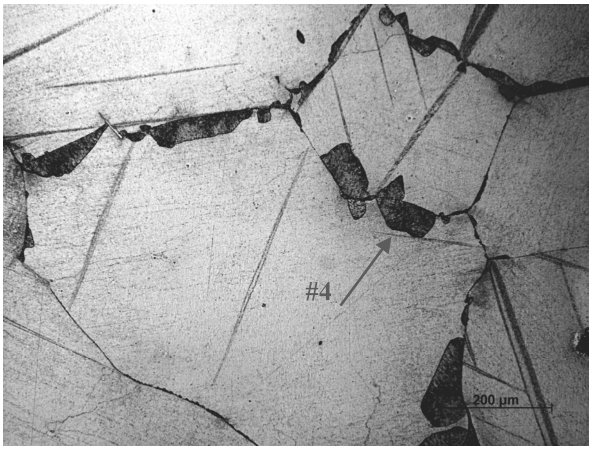

Fig. 4. Microstructure alloy MCMgAl9Zn1 - after aging treatment

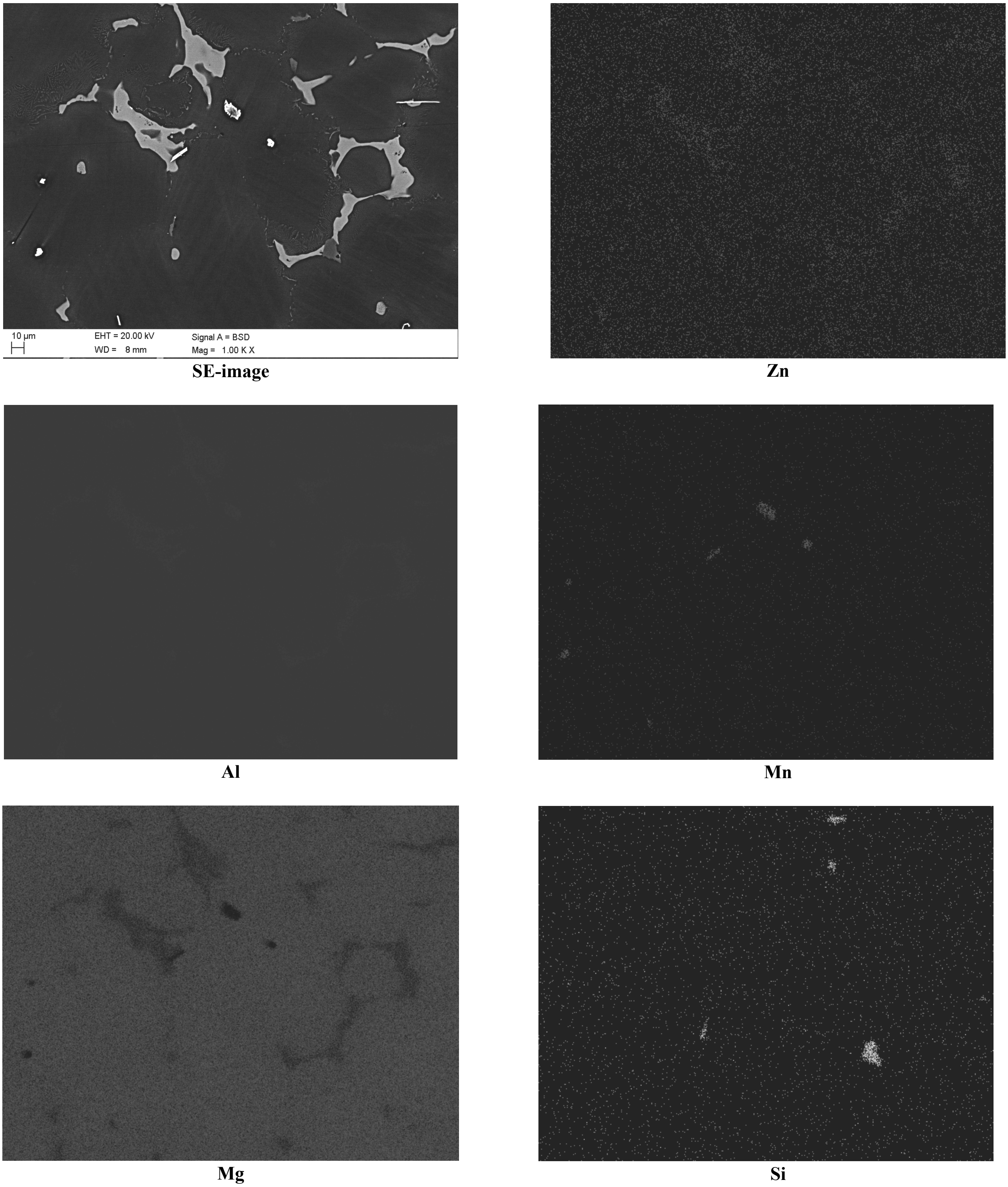

Fig. 5. The area analysis of chemical elements alloy MCMgAl6Zn1 in as-cast state: image of secondary electrons (A) and maps of elements' distribution

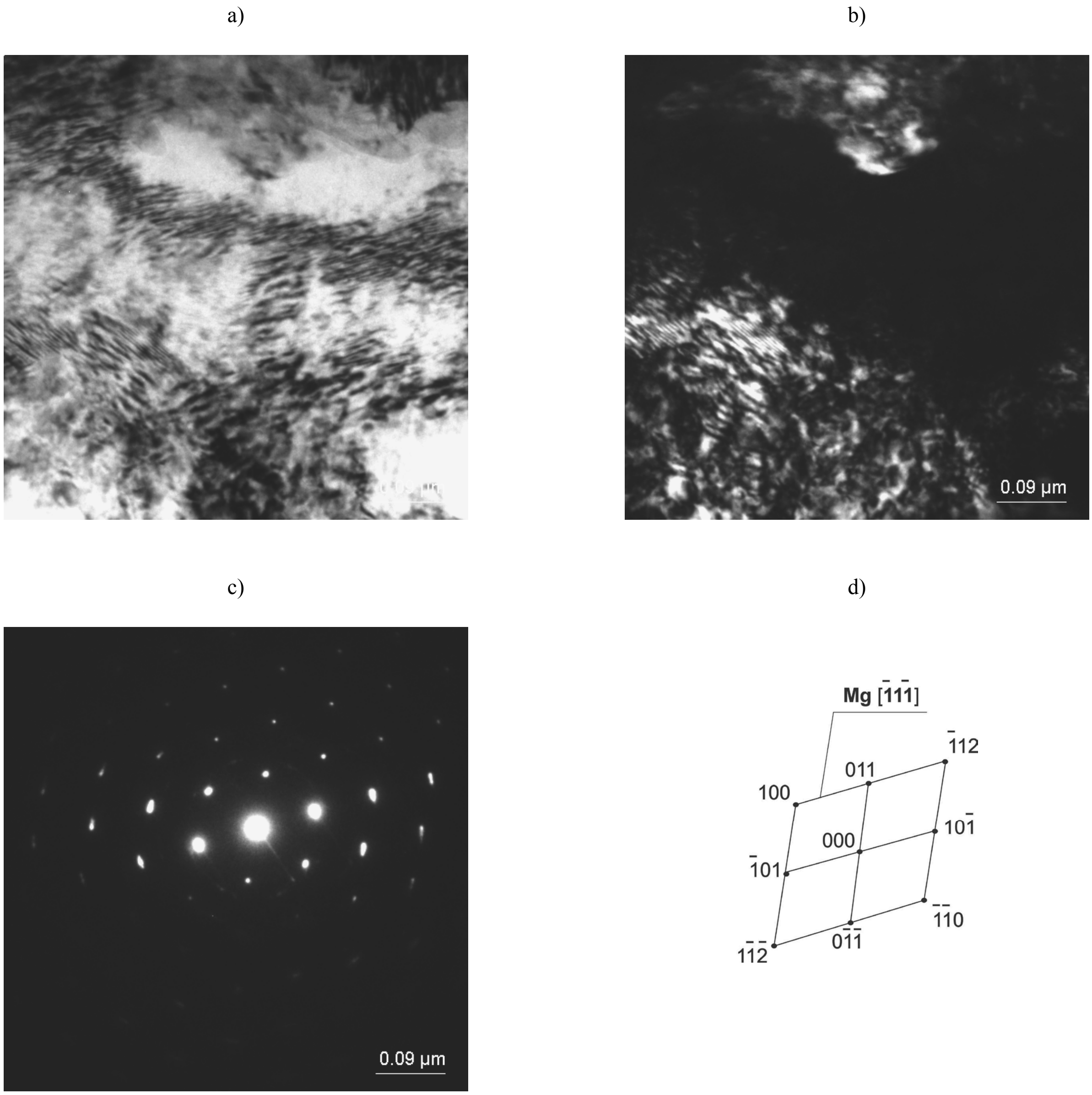

Fig. 6. a) TEM bright field image of the MCMgAl9Zn1 alloy after aging treatment with solid solution α – Mg (matrix), b) TEM dark field image of the area in Fig. 6a, c) diffraction pattern of area shown in a, d) part of solution for diffraction pattern shown in c

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 γ-$Mg_{17}Al_{12}$, explicitly indicate a big defect and micro deformations of lattice caused by the heat treatment (Fig. 7).

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 (Figs. 6, 7). The ageing process has caused the precipitation of evenly distributed dispersive g secondary phase in the needle form that has in the major performed investigations a preferred crystallographic orientation in the matrix (Fig. 9) [6-10].

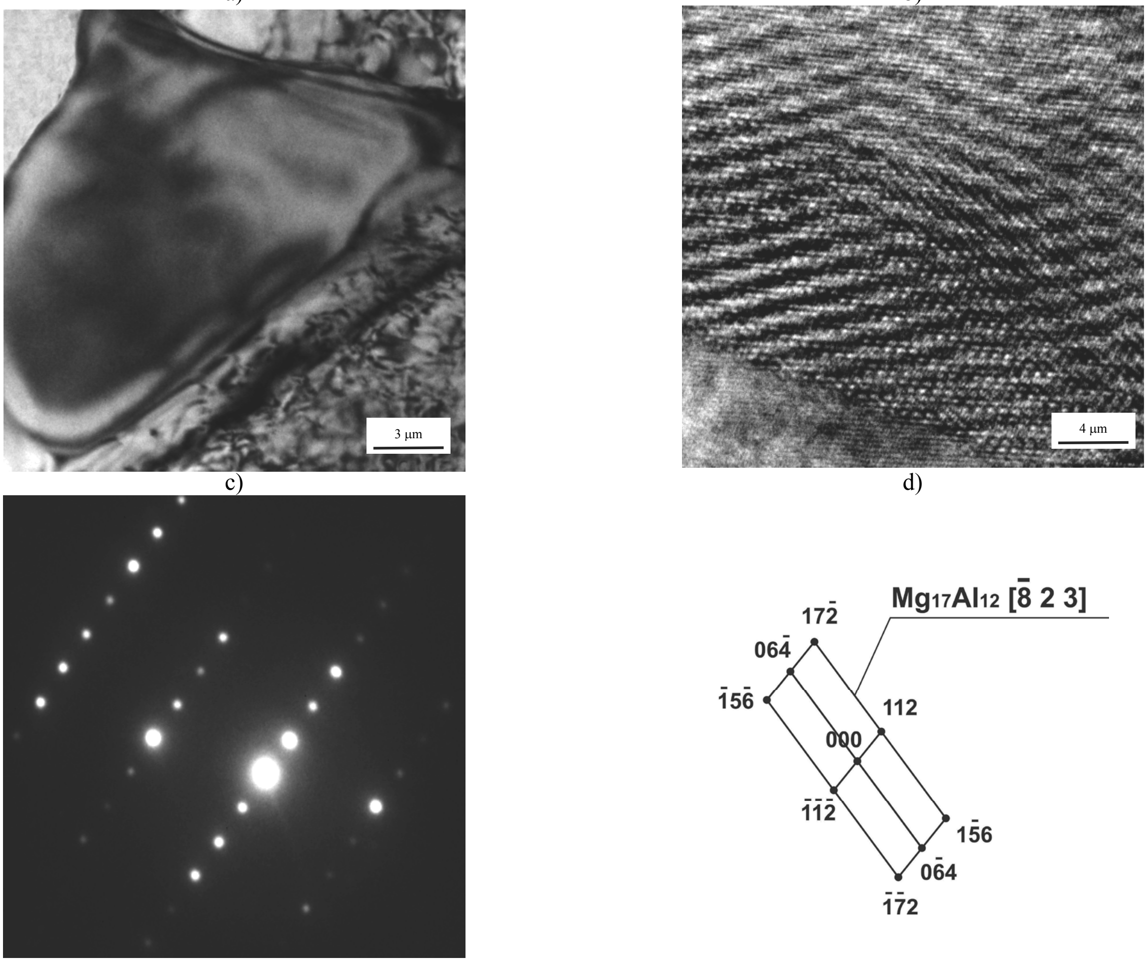

Fig. 7. a) TEM bright field image of the MCMgAl9Zn1 alloy after aging treatment with solid solution α – Mg (matrix) and an intermetallic secondary phase γ – $Mg_{17}Al_{12}$, b) high resolution image of the α-Mg matrix and precipitations, c) diffraction pattern of area shown in a, d) part of solution for diffraction pattern shown in c

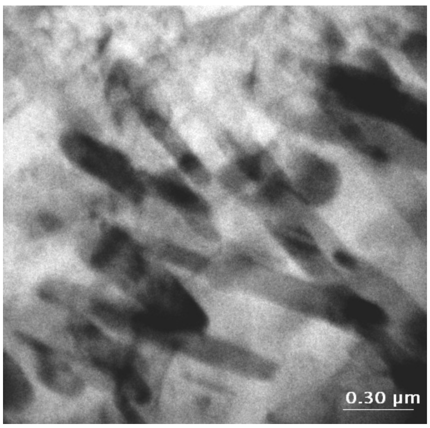

Fig. 8. TEM image examples of the intermetallic secondary phase g – Mg17Al12 in the form of needle precipitations from the MCMgAl9Zn1 alloy after aging treatment

According to the relations given by S. Guldberga and N. Ryuma [10] which occured in the eutectic microstructure in the Mg alloys containing 33% Al. A part of them shows the following relation:

(1 01) α-Mg ‖ (10) Mg17Al12
[11 0] α-Mg ‖ [111] Mg17Al12

Some of the precipitations in the srudied magnesium alloys after solution heat treatment and ageing show an orientation, where the plains of the family {110} Mg17Al12 are rotated about 10° compared to the plains of the {1 01} family of the α-Mg solid solution, moreover other plains show bigger rotation value as given by S. Guldberga and N. Ryuma [10]. Precipitation of the γ-$Mg_{17}Al_{12}$ phase are mostly of the shape of roads, and the prevailing growing directions are the directions <110> α-Mg.

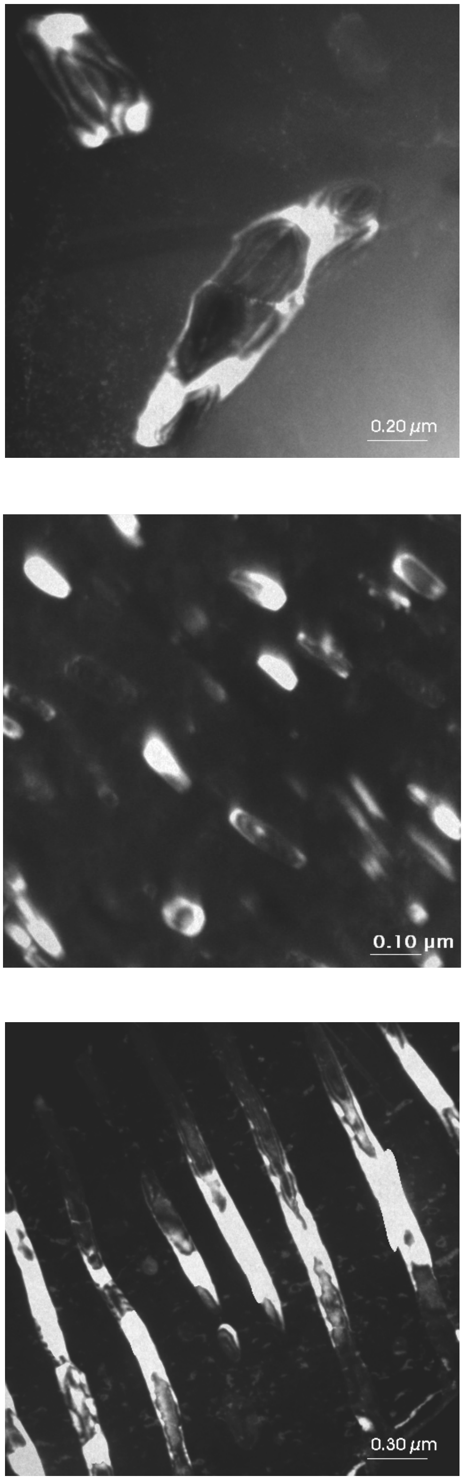

Fig. 9. TEM image examples of the intermetallic secondary phase $\gamma - Mg_{17}Al_{12}$ in the form of needle precipitations from the MCMgAl9Zn1 alloy after aging treatment

4. Conclusions

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 $\gamma - Mg_{17}Al_{12}$ 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 ageing confirm the appearance of a high density of defects of the crystal structure in the material.

Acknowledgements

Research was financed partially within the framework of the Polish State Committee for Scientific Research Project No. 4688/T02/2009/37 headed by Dr Tomasz Tański

Additional information

Selected issues related to this paper are planned to be presented at the 16th International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM3S'2010 celebrating 65 years of the tradition of Materials Engineering in Silesia, Poland and the 13th International Symposium Materials IMSP'2010, Denizli, Turkey.

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