STRUCTURE AND PROPERTIES OF SELECTED MAGNESIUM ALLOYS PREPARED FOR SPD PROCESSING

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Abstract

At the contemporary stage of the development of the engineering thought, and the product technology itself, material engineering has entered the period of new possibilities of designing and manufacturing of elements, introducing new methods of melting, casting, forming, and heat treatment of the casting materials, finding wider and wider applications in many industry branches. Engineers whose employment calls for significant expenditure of labour and costs strive to reduce material consumption. Therefore the development of engineering aims at designs optimizing, reducing dimensions, weight, and extending. Increasing the share of light structural materials in structures of airplanes or vehicles leads to reduction of environmental load. Magnesium alloys also play an important role among these materials. Aluminium and zinc are used the most frequently for alloying of magnesium. Small concentrations of manganese in the Mg-Al-Zn alloys improve resistance of these alloys to corrosion. Alloys AZ91 have been investigated very well and they found many applications in automotive industry. Mg-Al-Si alloys improve resistance to creep at temperatures up to 150 °C, they have good plasticity, ultimate strength and yield strength. They are used in crank cases of air-cooled automobile engines, for production of clutch pistons and blade stators. Recently, however, increases also utilization of formed magnesium alloys namely application of SPD methods. From this reason the presented work deals results of metallographic analysis, microanalysis and hardness measurements above presented magnesium alloys. Knowledge of structure and mechanical properties is very important for complex evaluation of magnesium alloys for its next application in SPD processing. SPD methods application for magnesium alloys in as cast state was practically impossible so that the heat treatment before processing of those is advisable and it will be of aim of study in the next work.

Keywords:
Magnesium alloys; Metallographic analysis; Structure; Properties

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 centers 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) \cite{1-5}. Increasing the share of light structural materials in structures of airplanes or vehicles leads to reduction of environmental load. Magnesium alloys also play an important role among these materials.
Aluminium and zinc are used the most frequently for alloying of magnesium. Small concentrations of manganese in the Mg-Al-Zn alloys improve resistance of these alloys to corrosion. Alloys AZ91 have been investigated very well and they found many applications in automotive industry. Magnesium alloys are divided on:

- Casting alloys (Gravity and Low-pressure casting, Die-casting)
- Wrought alloys
- Composites
- Powder materials

Scope of utilization of foundry magnesium alloys is continuously being extended, so if we want to operate as competitive producers, it is necessary to investigate very actively properties of individual alloys, optimize their chemical composition, study issues of their metallurgical preparation, verify experimentally their casting properties and conditions of successful casting of castings by individual methods, including heat treatment.

Magnesium alloys are subjected to heat treatment mostly for the purpose of improvement of their mechanical properties or as an intermediary operation, to prepare the alloy to other specific treatment processes. The type of heat treatment depends namely on the chemical composition of the alloy.

SPD methods application for magnesium alloys with graduated aluminum content in as cast state was impossible so that the heat treatment before processing of those is advisable [1,2].

![Fig.1 Examples of elements from magnesium alloys](image)

The presented paper is focused on evaluation of structure and properties of the alloys AZ91, AS31 and on investigation of structure and properties of the newly developed alloy Mg-Zr.

### 2. USED MATERIALS AND EXPERIMENTAL METHODS

The model alloys AZ91 and AS31 (three sheets of the alloy AS31 with modified silicon contents), as well as Mg-Zr alloy made of high-purity magnesium with contents of added zirconium 0.7 %, AZ91 and pure magnesium – for reason of comparison hardness measurement were used for investigation of structure and properties in experimental part of the work. Chemical composition of alloys is given in the Tab.1.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Si</th>
<th>Fe</th>
<th>Be</th>
<th>Zr</th>
<th>Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>AS31-1</td>
<td>3.43</td>
<td>0.13</td>
<td>0.289</td>
<td>0.852</td>
<td>0.000</td>
<td>0.001</td>
<td>0.002</td>
<td>0.002</td>
</tr>
<tr>
<td>AS31-2</td>
<td>3.76</td>
<td>0.17</td>
<td>0.363</td>
<td>0.883</td>
<td>0.002</td>
<td>0.000</td>
<td>0.001</td>
<td>0.002</td>
</tr>
<tr>
<td>AS31-3</td>
<td>3.60</td>
<td>0.16</td>
<td>0.352</td>
<td>0.996</td>
<td>0.002</td>
<td>0.000</td>
<td>0.001</td>
<td>0.003</td>
</tr>
<tr>
<td>Mg-Zr</td>
<td>0.07</td>
<td>0.01</td>
<td>0.02</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.07</td>
<td>-</td>
</tr>
<tr>
<td>AZ91</td>
<td>8.95</td>
<td>0.76</td>
<td>0.21</td>
<td>0.041</td>
<td>0.008</td>
<td>0.001</td>
<td>0.003</td>
<td>0.01</td>
</tr>
</tbody>
</table>

The samples for metallographic evaluation were prepared in usual manner: there were wet-ground on disc sanders with use of emery papers with roughness: 600, 800, 1000 and 1200. Special care was paid during
grinding to prevention of possible deformation of the surface layer. That's why the samples were cooled with water.

Polishing of samples was made in two stages. In the first stage the samples were polished on cloth with use of the Al₂O₃ based polishing suspension. Discs of the polishing machines rotated by speed of 200 rev./min.

In the second stage the polishing was made on very fine velvet cloth with short fibres. Diamond powder with grain size of 1 µm was used as polishing material. Diamond was applied by spraying and cloth was regularly wetted by alcohol-based liquid. The samples were moved against the direction of disc rotation with application of slight thrust. The samples were finally flushed with water and spirit and dried by stream of hot air.

The samples were then etched by Nital. Duration of etching varied from 5 to 10 seconds.

Light microscope NEOPHOT 2 was used for evaluation of microstructure of alloys. Investigation of microstructure was completed also by determination of hardness or micro-hardness by Vickers. In the case of alloy AZ91 SEM microanalysis method was used.

### 3. EVALUATION OF STRUCTURE, MICROANALYSIS AND HARDNESS OF USED ALLOYS

Microstructures of samples of the alloy AS31 are shown in the Figs.2 – 4. It follows from these figures that microstructure of all the samples of alloys with silicon show dendritic character. They are formed by coarse-grained basic solid solution α with partly segregated phase Mg₁₇Al₁₂ along the grain boundaries and together with areas of „Chinese characters“ which is Mg₂Si based compound. It is also possible to assume from these figures that areas with “Chinese characters” are distributed mostly in inter-dendritic spaces in the scope corresponding to composition of the alloy. In case of the alloy AS31-2 the Fig.2 shows a smaller extent of the area with segregated Mg₁₇Al₁₂ due to more distinct change of chemical composition.

![Fig.2 Microstructure of the alloy AS31-1](image1)

![Fig.3 Microstructure of the alloy AS31-2](image2)

![Fig.4 Microstructure of the alloy AS31-3](image3)

Examples of microstructure of the alloy containing zirconium are shown in Figs.5,6 and that of pure magnesium are in the Fig.7.

![Fig.5 Microstructure of the Mg-Zr alloy](image4)

![Fig.6 The detail of microstructure of the Mg-Zr alloy](image5)

![Fig.7 Microstructure of pure magnesium](image6)
Microstructure of the alloy with zirconium is formed mostly by equi-axed grains of various sizes, which contain oblong particles. Due to the fact that the alloy with this composition has been developed recently and its structure is not described in available literature, it can be assumed that these can be grains of magnesium based solid solution, in which a precipitation of fine minority phases could have occurred during solidification due to influence of positive solubility coefficient [6]. However, due to orientation of these etch patterns it is impossible to completely exclude the possibility that these are so called artefacts caused by imperfect removal of the deformed surface layer on the cut. It is also possible to take into consideration forming of a relief at surface deformation during preparation of the sample, or decoration of possible twins or glide bands enriched by dissolved zirconium. It was verified by thorough repeated preparation of the sample surface realised with maximum care, whether this effect disappears, but this phenomenon repeatedly re-appeared even in these cases of extra-careful preparation. Detailed explanation of this will require application of chemical or electrolytic methods of polishing or possibly even etching of the sample surface (provided that etching effect is achieved), or by methods of electron microscopy.

Fig. 7 shows microstructure of the sample of pure magnesium. Under the given conditions area etching of grains occurred. Structure is formed predominantly by equi-axed grains of various sizes, similarly as in case of the alloy with zirconium.

Fig. 8 shows microstructure of the AZ91 sample. As a result of the microscopic examination performed, it was found out that the casting AZ91 alloy is characterised by a solid solution structure $\alpha$ with $\alpha+\beta$ eutectic and $\beta$ phase (Mg$_{17}$Al$_{12}$) at grain boundaries [7].

Examinations of the chemical composition of the casting magnesium alloys using the EDX spectrometer (area analysis) confirmed the presence of the main alloying elements: magnesium, aluminium, manganese, and zinc. One can clearly observe in the structure of the casting magnesium alloys, not only the Mg$_{17}$Al$_{12}$ phase precipitations, the distinct aluminium, manganese and silicon concentrations, which indicate the presence of the MnAl$_4$ and Mg$_2$Si type precipitations in the alloy structure (Figs 10-13).

The above mentioned alloys were then subjected to determination of hardness according to Vickers HV30 or HV5 with use of HPO 250 hardness tester.

Determination of hardness for the Mg-Si was done without any problem and the values HV30 and HV5 practically did not differ. These results are given in the Tab. 2.

| Table 2 Values of hardness for individual samples of the alloys AS31 and Mg-Zr |
|---|---|---|
| Alloy | HV30 | HV5 |
| AS31-1 | 47.48 | 45.65 |
| AS31-2 | 45.82 | 44.94 |
| AS31-3 | 47.14 | 46.84 |
| Mg-Zr | - | 27.77 |
| AZ91 | 61.20 | 60.32 |

Nevertheless, it was impossible to read the values of diagonals at determination of the hardness HV30 of the alloy Mg-Zr due to heavy deformation of these diagonals. Hardness HV5 was determined for Mg-Zr without any problem, average hardness was 27.77 and standard deviation was 2.55. Under heavier load it became impossible to determine the size of diagonals, since the indentation was strongly deformed due to large grains and high anisotropy.
4. CONCLUSIONS

On the basis of obtained results it is possible to draw the following conclusions:

- Microstructure of the alloy Mg-Al-Si is of dendritic character, it is formed by comparatively coarse-grained basic solid solution α with partly dendritically segregated phase Mg\textsubscript{17}Al\textsubscript{12} and „Chinese characters”, which is Mg\textsubscript{2}Si based compound.

It was impossible to determine the hardness value of pure magnesium both at HV30 and HV5. In proximity of indentation distinctive slip lines were observed caused by plastic deformation on slip planes.

For this reason a possibility of determination of micro-hardness of pure magnesium was verified at lower loads. In this case it was possible to determine these values of micro-hardness without any problem. The micro-hardness indentation on pure magnesium gives this value – 32 HV0,01.
• Determination of hardness in the samples of the alloy Mg-Si was done without any problem and the values HV30 and HV5 practically did not differ.

• Microstructure of the alloys Mg-Zr is formed by polyedric grains of Mg based solid solution with dimensions 100 – 500 μm.

• It is characterised by occurrence of numerous etching artefacts, the origin of which cannot be determined unequivocally.

• Microstructure of pure magnesium is formed by coarse, uneven grains, the dimensions of which reach even several millimetres. Etching artefacts were not observed, however, area etching occurred due to influence of their orientation on intensity of their etching.

• In case of pure magnesium a dependence of the micro-hardness values on magnitude of the load was observed. It was found that the micro-hardness value slightly decreases with increasing load.

• The AZ91 alloy as cast is characterised by a solid solution structure α with α+β eutectic and β phase (Mg17Al12) at grain boundaries.

• The results of the EDX chemical composition analysis confirm the presence of magnesium, aluminum, manganese, and zinc, constituting the structure of α solid solution with the Mg17Al12 phase mainly on the grain order.

• Also the phase MnAl4 with irregular shape occurred often in the shape of blocks or needles and the Laves phase Mg2Si.

• From the reason of intermetallic phases the SPD methods application for magnesium alloys in as cast state was practically impossible so that the heat treatment before processing of those is advisable and it will be of aim of study in the next work.

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LITERATURE


