MICROSTRUCTURE CHARACTERISTICS CAST MAGNESIUM ALLOYS AFTER FORMING AND HEAT TREATMENT

CHARAKTERISTIKY HORCÍKOVÝCH SLITIN PO TVÁRENÍ A TEPELNÉM ZPRACOVÁNÍ

Cížek Lubomír, Greger Miroslav, Šustai Ondrej\textsuperscript{a}  
Kielbus Andrzej, Sozanska Marija\textsuperscript{b}

\textsuperscript{a} VSB-TU Ostrava, 708 33 Ostrava Czech Republic, lubomir.cizek@vsb.cz
\textsuperscript{b} Silesian University of Technology, Katowice, Poland

Abstract
The structure and microstructure characteristics of magnesium alloys are connected with microstructure that is influenced by metallurgical and technological aspects. The experimental part deals with structure determination of the phases presented in selected cast magnesium alloys as well as with the influence of the forming and selected heat treatment on modifying the initial structure. The methods of the colour and polarized light metallography and microstructure phases analyse on EDAX analysator were used.

Abstrakt
Struktura a mikrostruktura horcíkových slitin je závislá na metalurgických a technologických aspektech jejich výroby a zpracování. Experimentální část práce je venována rozboru mikrostruktury vyskytujících se fází v odlitcích vybraných horcíkových slitin. Rovněž je sledován vliv tvárení a vybraných způsobu tepelného zpracování na zmeny výchozí struktury a mikrostruktury. Ke studiu byly použity metody světelné mikroskopie s využitím technik barevného leptání a pozorování v polarizovaném svetle a mikrostrukturní fázové analýzy na analyzátoru EDAX.

1. INTRODUCTION
Magnesium alloys has been used for a wide variety of applications, namely from the reason of their low density and high strength–to–weight ratio. Due to its attractive properties, magnesium alloys has been successfully used in different spheres of life, including the aircraft and motor vehicle as well as metallurgical, chemical and electrical-chemical industries. Low inertia, which results from its low density, is advantageous in rapidly moving parts, for example automobile wheels and other automobile parts. Moreover, magnesium shows relatively good electric and thermal conductivity as well as high damping capacity [1,2].

The basic magnesium alloys include ones which contain manganese, aluminium, zinc, zirconium and rare-earth elements which allow obtaining suitable properties. Manganese does not cause any increase of tensile strength, however, it does slightly increase the yield point. It also brings about an increase of resistance to the action of sea water. The quantity of manganese in magnesium alloys is limited by its relatively low solubility in magnesium. Manganese content in alloys with an Al addition does not exceed 0.3% and 1.5% in alloys without Al addition. Aluminium enhances both tensile strength and hardness, and improves casting properties of an alloy. The best ratio of mechanical to plastic properties is obtained with a 6% Al content. An addition of zinc in combination with Al aims at improving tensile strength at a room temperature; however 1% of Zn with a 7÷10% Al content in an alloy enhances hot cracking. Zirconium is added to alloys which contain zinc, rare-earth elements,
Thorium and their combinations, for the purpose of structure refinement. It should not be used in alloys containing aluminium and manganese, since it forms stable compounds with them which are removed from the solid solution. Rare-earth elements are added to manganese alloys as a mischmetal or didymium. Mischmetal contains cerium, lanthan and neodymium, whereas didymium is a mixture of neodymium and praseodymium. An addition of rare-earth elements enhances magnesium alloys’ strength at a room temperature and what is more, it reduces porosity of casts [3].

In general, magnesium alloys can be divided into two groups [1,3]:

I – those containing 2-10 % Al with minute quantities of Zn and Mn. They cost of production is low and they are characterised by good corrosion resistance as well as a considerable decrease of mechanical properties with an increase of temperature. The following alloys are rated among this group:

?? Mg-Al-Mn alloys;
?? Mg-Al-Zn-Mn alloys;

II – those containing a wide range of elements (mostly Mn, Zn, Th, Ag and Si instead of Al), but always with an adequate content of Zr which has a considerable influence on the increase of mechanical properties. These alloys can work at elevated temperatures, however, the price of alloying additions along with a special production technology considerably raises the cost of their fabrication. Within this group of alloys, those most often applied are:

?? Mg-Zr alloys;
?? Mg-Zn-Zr alloys;
?? Mg-rare-earth elements-Zr alloys;
?? Mg-Ag-rare-earth elements-Zr alloys;
?? Mg-Y-rare-earth elements-Zr alloys;

Casting magnesium alloys make up about 85÷90% of all products fabricated in Europe with the application of Mg. Those most frequently applied are AZ91 and AZ31 alloys which contain 9 and 3% Al respectively, and ca. 1% Zn; next are AM50 and AM60 alloys with 5 and 6% Al contents and with a Mn addition. These alloys are characterised by good casting properties, in particular during high-pressure casting, as well as good mechanical properties. They are used for casting such structural components of cars as: wheel rims (Fig.1a), instrument panels, steering wheels (Fig.1b) and seat frames (Fig.1c), as well as components of equipment in other fields, e.g. telephones (Fig.2a), camcorders (Fig.2b) and gardening tools (Fig.2c).

Fig.1. Car components made of magnesium alloys:
a) Ford wheel rim of AZ91D alloy;
b) steering wheel of AM50A alloy;
c) seat frame of AM60B alloy.
The maximum solubility of aluminium in magnesium at an eutectic temperature (437°C) is 14%, whereas an eutectic mixture (\(? + \text{Mg}_{17}\text{Al}_{12}\) intermetallic phase) occurs at ca. 33% Al content (Fig. 1). The content of aluminium in all industrial alloys of Mg with Al is not higher than the boundary solubility of Al in Mg. The equilibrium structure of these alloys is characterised by 100% presence of a solid solution, whereas the unbalanced structure, additionally metastable in casting alloys, shows the presence of an eutectic already at a 2% Al content [4].

![Fig. 2. Components used in different sectors of economy, made of magnesium alloys:](image)
a) fragment of a cell phone made of AZ91D alloy;  
b) camcorder casing;  
c) garden shears made of AZ91D alloy.

![Fig. 3. Mg-Al binary phase diagram [3]](image)

### 2. EXPERIMENTAL PROCEDURE

#### 2.1 Material for research

The object of the research was the AZ91D casting alloy intended for wheel rims, cast into a sand mould. The casting practice included melting in a 250 kg induction furnace with the application of modification with SPEFINAL T 200 as well as refining with Emgesal Flux 200. The casting temperature was 740°C. The chemical composition of the analysed alloy is presented in Table 1.
Forming process was made with use of cast plates (size 10x20x150 mm)

Table 1. Chemical composition of AZ91D alloy in wt.-%

<table>
<thead>
<tr>
<th>Mg</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>Fe</th>
<th>Be</th>
</tr>
</thead>
<tbody>
<tr>
<td>Remainder</td>
<td>9,15</td>
<td>0,6</td>
<td>0,24</td>
<td>0,03</td>
<td>0,01</td>
<td>0,01</td>
<td>0,0001</td>
</tr>
</tbody>
</table>

2.2 Research methodology of cast samples

The microsections for structure examination were subjected to grinding with sandpaper of 250 to 1200 granulation. Next, they were mechanically polished with the use of diamond pastes and chemically, in a solution of 90 ml CH₃OH + 10 ml HNO₃.

For etching, two reagents were used with the following chemical compositions:
1. 10 ml fluoric acid (48%) + 90 ml water;
2. 2 ml fluoric acid + 2 ml nitric acid + 96 ml water;

The observation was performed with:
1. REICHERT light microscope of MeF2 type; the results of the observation were recorded with a NIKON digital apparatus, COOLPIX 990 model;
2. HITACHI S-4200 scanning microscope with a cold cathode, equipped with an X-radiation detector EDS-VOYAGER of NORAN INSTRUMENTS.

2.3 Research results of cast samples

As a result of the microscopic examination performed, it was found out that the casting AZ91D alloy is characterised by a solid solution structure with eutectic and phase (Mg₁₇Al₁₂) at grain boundaries (Figs 4÷6). The eutectic obtained assumes the form of a so-called abnormal eutectic, where the initiating particles dissolved in the solid solution precipitate during the casting process at the boundaries of the initiating solid solution. Furthermore, the occurrence of Laves’ phase in the form of Mg₂Si was proved as well as in the form of precipitations of probably a MnAl₄ phase (Fig. 6). The Mg₂Si phase has a light blue colour and is characterised by a regular, multilateral shape with smooth edges. The precipitations containing manganese and aluminium are characterised by an irregular shape with a rough surface; they very often take the form of spines. The closer the precipitation surface, the higher the ratio of Al to Mn content. The microanalysis results of the chemical composition of individual phases are shown in Table 2 (Figs 5, 6).

a)

Fig. 4. Microstructure of the AZ91 alloy cast: solid solution + eutectic (?) + intermetallic phase Mg₁₇Al₁₂ and Mg₂Si precipitations

a) LM 150x; b) LM 400x;
**Fig. 5.** Microstructure of AZ91 alloy. a+b+??

- a) SEM magn. 1500×;
- b) spectrum of ??solution chemical composition
- c) spectrum of ??-phase chemical composition - Mg_{17}Al_{12}

**Table 2.** Chemical composition of identified AZ91 alloy phases

<table>
<thead>
<tr>
<th>Phase</th>
<th>Chemical element [at.- %]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mg</td>
</tr>
<tr>
<td>? ?phase</td>
<td>89,41</td>
</tr>
<tr>
<td>? phase</td>
<td>63,39</td>
</tr>
<tr>
<td>Mg_{2}Si phase</td>
<td>62,39</td>
</tr>
<tr>
<td>MnAl_{4} phase</td>
<td>1,37</td>
</tr>
</tbody>
</table>

### 2.4 Experimental technique of forging samples

Marking of samples for forging: A- initial state as cast, B- state after heat treatment: pre-heating 375°C/3h ? 415°C/18h, cooling in air (T4-signed after ASTM Standard).

Both groups of samples were rolling with graduated 6-degree deformation with step of 10-15 % (up to approx. 80 % of the original thickness of 10 mm) at temperatures of 380°C and 420°C, on the rolling mill stand with diameter of rolls 70 mm. The speed of rolling was 66mm.s⁻¹ and strain rate of was 1,23.s⁻¹.

Cooling of these samples after individual operations was always made in air.

The samples were subjected to a metallographic analysis with use of light microscopy. As well the hardness HV10 was determined.
Fig. 6. Precipitations of Mg$_2$Si and AlMn$_4$ phases in the AZ91 alloy matrix
   a) SEM 500×, b) SEM 1000×
   c) spectrum of Mg$_2$Si phase chemical composition
   d) spectrum of AlMn$_4$ phase chemical composition

2.5 Results and discussion of formed samples

Alloy micro-structure in initial as cast state A contains a majority phase – solid solution of aluminium and or other alloying elements in magnesium basis and two types of minority phases structures (Fig. 7).

The first type of this structure is formed by comparatively massive phase particles Mg$_{17}$Al$_{12}$, or Mg$_{17}$(Al,Zn)$_{12}$, the second type is represented by fine acicular or spherical particles of the same phase, occurring in the close vicinity of grain boundaries of the matrix phase ?1?2. This type of micro-structure does not quite correspond with binary equilibrium diagram Al-Mg.
After application of heat treatment (B), the precipitate and compact phase largely dissolves, during cooling on air there does not occur a repeated precipitation from solid solution (Fig. 8) in more significant extent. Obtained structure is formed by hyper-saturated magnesium based solid solution and un-dissolved rests of the massive phase $\text{Mg}_7\text{Al}_2\text{Zn}_{12}$.

At rolling under temperatures of 420°C and 380°C there took place phenomena of plastic deformation and re-crystallization differently for both initial states A and B, as it is also indicated by course of hardness (Fig. 9).

![Fig. 9. Hardness HV10 in dependence on the step of deformation at 380°C and 420°C](image)

At rolling of samples in as cast-state A, there has been observed at lower degrees of deformation a spheroidization of fine phases in the area near the grain boundaries and their gradual dissolving (Fig. 10a). In these areas, a re-crystallization started at higher degrees of deformation (Fig. 10b).

Similar phenomena were observed for both forming temperatures. Even at the highest amount of deformation the extent of re-crystallization did not exceed approx. 50 volume %.

At rolling of annealed samples with more homogenous micro-structure (B) structural changes were driven by different mechanism. Re-crystallization, beginning also in matrix near the grain boundaries, occurred already on the lowest deformation degree (10 %) (Fig. 11a). At the grade of 40 % of deformation the volume of re-crystallized grains was almost 100%. At the same time there was observed intensive precipitation of fine phases in the area near the grain boundaries, as well as in the area of slip bands in grains (Fig. 11b). The highest degrees of deformation were related to formation of elongated grains and occurrence of slip bands.

![Fig. 11a. Sample B - 10% def. at 420°C](image)  ![Fig. 11b. Sample B - 40% def. at 420°C](image)  ![Fig. 12a. Sample A - 50% def. at 380°C](image)  ![Fig. 12b. Sample B - 40% def. at 420°C](image)

In both types of samples there was observed formation of micro-cracks in peripheral areas of higher degrees of deformation.

Comparison of the course of structural changes with changes of hardness in dependence on extent of deformation is interesting. Lower values of hardness of annealed samples (B) up to 40 % of deformation evened up the values of samples in as cast (A) and subsequent course of hardness with increasing amount of deformation was comparable (see Fig. 9), although mechanisms of deformation and extents of re-crystallisation and precipitation processes differed. It is possible to compare different extent of re-crystallisation on Figures 12a (A) and 12 (B).
3. CONCLUSIONS

The research conducted was aimed at determining the microstructure of the casting AZ91D magnesium alloy which was cast into a sand mould.

- It has been shown that the analysed alloy has a solid solution structure with an abnormal α+β eutectic formed as a result of precipitation, during casting, of β phase particles, with the β phase dissolved primarily in the solid solution.

- The alloy is also characterised by massive precipitations of β phase (Mg₁₇Al₁₂) at the solid solution grain boundaries. In addition, precipitations (light blue) of Mg₃Si phase were observed, characterised by a regular, multilateral shape with smooth edges. Some acicular precipitations, of an uneven surface, of MnAl₆ phase were found as well.

It is possible to draw the following conclusions from observation of structural changes of samples made of alloy of the type AZ91 at rolling under temperatures of 420°C and 380°C:

- Mechanism of deformation and extent of re-crystallization in investigated samples depends on the types of initial structure and particularly on its homogeneity.

- In less homogeneous samples in as cast state A there occurs in the area near the grain boundaries dissolution of minority phases and partial re-crystallization. Deformation of re-crystallized grains is limited; even at the highest grades of deformation there was not observed formation of slip bands or fibrous structure. Extent of re-crystallization is smaller than in samples in the state B.

- In more homogeneous, annealed samples in the state B there occurs re-crystallization in greater extent?at higher steps of deformation there are formed slip bands, in which intensive precipitation of fine phases occurs. Fibrous micro-structure is also formed.

- In spite of different mechanisms employed in both types of initial processing of samples, the obtained values of hardness HV10 at deformations of 40 – 80 % are comparable.

REFERENCES


* The work was financed by aim of GACR of the Czech Republic No. GA-106/01/1247, GA-106/04/1346 and sponzored by Project MSM 273600001 and CEEPUS CZ-13 and PL-13.