Formation of Structure and Properties in Casting Processes on the Example of AZ91 Magnesium Alloy

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Abstract

Contemporary materials engineering requires the use of materials characterised by high mechanical properties, as these precisely properties determine the choice of material for parts of machinery and equipment. Owing to these properties it is possible to reduce the weight and, consequently, the consumption of both material and energy. Trying to meet these expectations, the designers are increasingly looking for solutions in the application of magnesium alloys as materials offering a very beneficial strength-to-weight ratio. However, besides alloying elements, the properties are to a great extent shaped by the solidification conditions and related structure. The process of structure formation depends on the choice of casting method forced by the specific properties of casting or by the specific intended use of final product. The article presents a comparison of AZ91 magnesium alloys processed by different casting technologies. A short characteristic was offered for materials processed by the traditional semi-continuous casting process, which uses the solidification rates comprised in a range of 5 - 20⁰C/s, and for materials made in the process of Rapid Solidification, where the solidification rate can reach 10⁶⁰C/s. As a result of the casting process, a feedstock in the form of billets and thin strips was obtained and was subjected next to the process of plastic forming. The article presents the results of structural analysis of the final product. The mechanical properties of the ø7 mm extruded rods were also evaluated and compared.

Keywords: Innovative foundry technologies and materials, Magnesium alloy, RS process, VDC process, Mechanical properties

1. Introduction

Particularly stringent quality requirements in the aerospace and automotive sectors of industry result in a continuous search for new solutions in terms of both materials and technology. Trying to meet these expectations, the designers are increasingly looking for solutions in the application of magnesium alloys. According to the data published in technical literature, the use of magnesium alloy components in cars in 2005 amounted to about 6 kg and made about 0.3% of the overall weight. Forecasts predict that by 2015 this proportion will change to about 11 kg, to reach in 2020 the level of as much as 100 kg [1,2]. Pure magnesium has low mechanical properties and that is why to increase the strength and corrosion resistance, various magnesium alloys are created [3-6]. The most valuable feature of magnesium alloys is their low density of about 1.8 g/cm³. The main advantages of magnesium alloys comprise a very favourable strength-to-weight ratio, relatively good electrical and thermal conductivity, high fluidity and machinability.

The most common magnesium alloys are now alloys composed of aluminium, zinc and manganese. Aluminium improves the tensile strength and hardness. Zinc in magnesium alloys is used...
toraise thealloy strength at room temperature, while manganese increases the corrosion resistance.

Yet, all these properties are to a large extentdependent on the resulting cast material microstructure. According to the well-known relationship between the grain size and mechanical properties, which can be described by Hall-Petch equation, the ultimate goal should be the reduction of grain size.

\[ \sigma = \sigma_0 + K_d d^{-1/2} \]  

(1)

where: 
- \( \sigma \) - materials constant, 
- \( K_d \) - stress intensity factor for plastic yielding (for Mg and Mg-based alloys the value is about 210 MPa*\( \mu \)m\(^{1/2} \)), 
- \( d \) - average grain diameter

Problems related with the grain size refinement in magnesium alloys are the subject of numerous studies conducted over the last decade [8-14]. Until now, many methods have been developed, which are applicable to alloys in both cast and wrought condition. The fine-grained microstructure has a beneficial effect on the mechanical properties and workability. The process of microstructure formation can be controlled through the use of modification treatment, e.g. superheating of alloy, the addition of carbon nucleating elements, Elfinal process (FeCl\(_3\)), etc., and also through the creation of appropriate conditions for alloy solidification. Rapid Solidification process, in which molten alloy is cast onto a spinning copper wheel ensuring high rate of heat extraction, is one of the techniques enabling the achievement of fine-grained structure [15-19]. The rate of solidification in this method is in a range of \( 10^2 - 10^5 \) C/s. As a result of the liquid metal entering in contact with the wheel, an immediate solidification follows and metal leaves the wheel in the form of a thin ribbon of ultrafine-grained structure. The ribbon is then cut into small flakes, and flakes are converted into the final product in the process of extrusion. The article compares the AZ91 alloy feedstock cast by two different techniques and subjected to plastic forming to obtain material for testing of the mechanical properties and structure examinations.

2. Methodology of the research

Tests were carried out on a magnesium alloy from the AZ91 family whose chemical composition determined with mass spectrometer is shown in Table 1. The alloy was used for the semi-continuous VDC casting process and melt-spinning. The resulting semi-finished products in the form of billets and thin strips were next subjected to plastic forming on an indirect-direct press, and as a result of this operation Ø 7 mm rods were obtained. The rods were used for the evaluation of product structure and testing of mechanical properties.

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Cu</th>
<th>Fe</th>
<th>Si</th>
<th>Be</th>
</tr>
</thead>
<tbody>
<tr>
<td>(%)</td>
<td>8.78</td>
<td>0.51</td>
<td>0.24</td>
<td>0.002</td>
<td>0.002</td>
<td>0.037</td>
<td>0.001</td>
</tr>
</tbody>
</table>

2.1. Casting by the VDC Process

Billetswere cast on a vertical semi-continuous casting machine operating at the IMNOMLSkawina. This is the only one in Poland installation for professional casting of billets in magnesium alloys. A diagram of the whole installation is shown in Figure 1. The alloy was melted in a resistance furnace cooperating with the casting equipment. Melting and casting processes were carried out under a protective gas atmosphere: comprising a mixture of nitrogen and SF\(_6\). The following parameters of the casting process were observed:

- the casting temperature of 690-700° C, the casting speed of 210 mm/s, the cooling water flow rate of 50 l/min.
- The billets were cast using a Hot-Toptype crystalliser with gas-oil lubrication system. The billet diameter was Ø100mm. From the cashtubes, after the removal of outer skin, the feedstock for extrusion was prepared.

![Figure 1. A diagram of the VDC installation for casting of billets in magnesium alloys](image)

2.2. Casting by the melt - spinning process

The alloy was melted in an atmosphere of protective gas (argon + SF\(_6\)) in a resistance furnace of the melt-spinning device. The casting process was carried out at a temperature of 680°C by pouring the metal directly onto a rapidly spinning water-cooled copper wheel. Owing to the contact of molten alloy with the rapidly dissipating wheel surfacel, an immediate solidification took place and the cast material left the wheel in the form of a thin strip with an average width of 1.9 mm and an average thickness of 50-90µm. The cast strip linear speed was 26 mm/s. The strip cast by rapid solidification was next subjected to the process of fragmentation. This operation was performed in a high-speed mill operating according to the principle of scissor cutting. Additionally, the mill chamber was equipped with a 1 mm mesh classifying sieve, which enabled producing the material with a grain size of less than 1 mm. Figure 2 shows a view of the cast strip before and after the process of fragmentation. Thus prepared material was used as a feedstock in the preliminary process of cold consolidation, followed by hot extrusion. Schematically, the method is shown in Figure 3.
2.3. The process of plastic forming

The extrusion tests were carried out in a 500T capacity direct-indirect extrusion press. The process was conducted by direct route, the feedstock before the test was preheated in an oven. The feedstock was composed of billets from the VDC process and fragmented ribbons from the RS process. An important problem has proved to be the technique of feeding the feedstock in the form of pellets (formed in a “cold” consolidation process) into the recipient chamber. To determine the optimal feedstock charging mode, two variants were examined:

- direct insertion of individual pellets into the recipient,
- making packets of 3-6 pellets each and pre-moulding them in an aluminium alloy tube (sleeve).

The no-sleeve variant eliminated the problem of a thin Al alloy film formed on the extruded items, which in some cases had to be removed mechanically. Unfortunately, feedstock of this type also raised some problems related with the fact that the “cold” consolidated pellets were not fully bound with each other. This has made the pellet loading process much more time-consuming as it was necessary to take care and avoid scattering of the product. The extrusion test parameters were as follows: the ram speed of 0.5 mm/s, the feedstock preheating temperature of up to 320 and 380⁰C. The small cross-section of the final product prompted the use of a 5-hole die to ensure the high extrusion ratio of \( \lambda = 41 \). As an end result of the extrusion process, ø7 mm rods with a smooth surface and absence of any visible cracks and tears were made.

3. Results and Discussion

On the samples of the extruded material, metallographic examinations were performed showing changes that have occurred in the microstructure under the effect of casting and plastic forming. Samples were examined under an OLYMPUS GX71 optical microscope. The structure was examined by the scanning electron microscopy (SEM). From rods extruded in the AZ91 alloys, samples were cut out and were used for the preparation of thin films made by grinding and electropolishing. Examinations were carried out under a Tecnai G2 transmission electron microscope using EDS and STEM HAADF attachments.

Compared to the structure of the sample made by Rapid Solidification (RS) and consolidation by extrusion, the structure of AZ91 alloy sample prepared in a conventional manner by billet casting and extrusion differed in the morphology of \( \text{Al}_{12}\text{Mg}_{17} \) phase precipitates (Figs. 4 and 5). The structure of the sample after RS and consolidation showed fine-grained \( \text{Al}_{12}\text{Mg}_{17} \) phase. The scanning electron microscopy (SEM) did not show the presence of fine Mn-containing phases present in the sample produced by conventional methods.
was cast by Rapid Solidification followed by consolidation and extrusion in a 500 T capacity press (sample 2) showed little differences in the average grain size. The average grain size in sample 1 was 3 µm and in sample 2 it was 2.7 µm. Samples 1 and 2 hot-extruded at 320°C were characterised by the structure typical of a dynamic recovery process. The structure of sample 1 showed the equiaxed grains with the slip bands inside and precipitates inside and along the grain boundaries (Fig. 6). From the chemical analysis in microregions it follows that there are two types of precipitates in the structure, i.e. the first type, relatively numerous, containing Mg and Al, a small amount of Zn, and Cu as well as Fe, and the second type, rarely present in the structure containing mainly Mn (Fig. 8). The structure of sample 2 differed somewhat from the structure of sample 1. The grains, though equiaxed, showed a large scatter in size. Clusters of both fine and coarse grains were visible and occasional slip bands occurring in their interior (Fig. 7). The grains, which in most cases revealed no visible traces of deformation, contained numerous fine precipitates with the size ranging from 50 to 300 nm. In addition to these fine precipitates, there was a large group of the precipitates with the size of about 2 µm located mostly within the grain boundaries. The results of the chemical analysis of the fine precipitates (50 - 300 nm) showed the presence of a phase containing Mg, Al and Mn (Fig. 9), the precipitates slightly coarser (about 500 nm) contained only Mg, Al and Zn, while the precipitates largest in size (about 2 µm), besides Mg, Al, Zn, also contained trace amounts of Cu and Fe.

Static tensile tests were performed on an INSTRON testing machine under a load of 100 kN in accordance with PN-EN ISO
Brinell hardness was measured with a Duramin 2500E hardness tester, using the indenter with a diameter of 2.5 mm operating under a load of 306.5 N in accordance with PN-EN ISO 6506-1:2008. Three indentations were made in each sample.

The results of measurements of the mechanical properties carried out on samples prepared from rods extruded at two different temperatures are compared in Table 2 and shown graphically in Figures 10 and 11.

Table 2. Mechanical properties extruded bars

<table>
<thead>
<tr>
<th>Material</th>
<th>Temp. extrusion [°C]</th>
<th>$R_{p0.2}$ [MPa]</th>
<th>$R_m$ [MPa]</th>
<th>$A_5$ [%]</th>
<th>HB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Billet</td>
<td>380°C</td>
<td>254</td>
<td>342</td>
<td>11.2</td>
<td>86</td>
</tr>
<tr>
<td>RS</td>
<td></td>
<td>275</td>
<td>362</td>
<td>9.7</td>
<td>80</td>
</tr>
<tr>
<td>Billet</td>
<td>320°C</td>
<td>262</td>
<td>361</td>
<td>8.4</td>
<td>93</td>
</tr>
<tr>
<td>RS</td>
<td></td>
<td>268</td>
<td>370</td>
<td>12.2</td>
<td>92</td>
</tr>
</tbody>
</table>

Fig. 10. The mechanical properties of rods obtained in the extrusion process carried out at 380°C

Fig. 11. The mechanical properties of rods obtained in the extrusion process carried out at 320°C

4. Conclusions

The AZ91 alloy feedstock made by two different casting techniques subjected next to plastic forming was used as a base material in studies of the possibilities of the structure formation control and, consequently, shaping of the mechanical properties. The conducted examinations of the structure of final products, which were the extruded $\varnothing 7$ mm rods, revealed some differences in the morphology of the $\text{Al}_{12}\text{Mg}_17$ phase precipitates resulting from the different cast product solidification rates (strips and billets). Finer precipitates occurred in the RS material. High solidification rate also affected the behaviour of Mn-containing phases which, due to a high degree of refinement, were not observed in the examined material processed by RS. Detailed TEM analysis showed slight differences in the average grain size of the compared materials. In the RS material, a heterogeneity in the size of particles was observed, ranging from 50 to 300 nm. Slight variations in the grain size in both materials were reflected in the results of mechanical properties. For thus conducted process of the plastic forming, only a very insignificant improvement in the above mentioned properties can be expected in favour of the material processed by RS, where higher values of $R_m$ and $R_{p0.2}$ were obtained in the material extruded at higher temperatures.

Close analysis of the obtained results clearly shows that the process of plastic forming performed at a temperature of 320 and 380°C has undoubtedly contributed to the grain growth in the material processed by RS, causing only slight improvement in the mechanical properties, compared to the material from a conventional process. Unfortunately, due to technological reasons, at this stage of the study, it was not possible to carry out the plastic forming at lower temperatures, which would not induce the recrystallisation process.

The search for alternative materials and methods for their production leads to the conclusion that the method of RS offers some potential the full use of which is dependent on the selection of process parameters and also on the possibility of using non-standard chemical composition that will yield the materials with non-standard properties. The study was performed on a typical AZ91 alloy, focusing attention rather on how to fabricate this material than on how to achieve its non-standard properties. However, in the next step, the authors intend to focus attention on the modification of chemical composition of magnesium alloys to get by means of the RS process the material unattainable by traditional methods.

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References


