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Investigations of the Effect of Heat Treatment and Plastic Deformation Parameters on the Formability and Microstructure of AZ91 Alloy Castings

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ABSTRACT

The article discusses the influence of heat treatment and metal forming parameters on formability and the structure of the AZ91 cast magnesium alloy. The aim of the article is to determine the optimal parameters of homogenization and plastic deformation of sand castings made of the AZ91 alloy in order to improve their properties and structure. In this study, sand castings made from the AZ91 alloy were examined. In the first stage, the castings were homogenized at: 385 °C, 400 °C, 415 °C and 430 °C with argon as a shielding gas for 24 hours and then quenched. Subsequently the upsetting tests were conducted at 380 °C; 400 °C; 420 °C; 440 °C for two deformation values: $\varepsilon = 0.7$ and $\varepsilon = 1.1$. After upsetting, the samples were water- and air-cooled. At this stage, a visual assessment was made and samples without cracks were subjected supersaturation at 415 °C for 6 h, and artificial aging at 175 °C for 24 h. Vickers hardness tests and microstructure assessment were carried out, at individual stages of testing. Based on the results obtained from the upsetting, structure and hardness tests, the most favorable homogenisation and plastic deformation conditions were determined for AZ91 alloy sand castings. The best results are achieved by homogenizing sand castings at 415 °C for 24 h. Among the tested parameters for conducting metal forming processing in the range of 380–440 °C and deformation values: $\varepsilon = 0.7$ and $\varepsilon = 1.1$, forging of sand-cast AZ91 magnesium allow at 420 °C and deformation of $\varepsilon = 0.7$ with water cooling seems to be the most favourable. The final heat treatment applied after the deformation process consists of supersaturation at 415 °C for 6 hours water quenching as artificial aging at 175 °C for 24. This combination of heat and plastic treatment parameters of castings allows for improvement of the structure and properties of sand castings made of the AZ91 alloy.

Keywords: cast magnesium alloys, AZ91 alloy, heat treatment, microstructure, formability.

INTRODUCTION

Magnesium alloys have enjoyed considerable interest in aviation, automotive, and machine industries [1]. Characteristics enabling such a wide application of the alloys include low density, dimensional stability, vibration-damping capacity, good machining properties [2], and resistance to indentation [3,4]. The limitations of the alloys' application encompass low corrosion resistance, high price, low ductility, and tendency to self-ignite [5,6]. However, effective methods of surface protection are used to avoid corrosion problems [7,8]. Due to problems with the plastic metal forming of magnesium alloys, their full potential as a construction material has not been completely exploited [9]. The alloys manifest a high tendency for cracking. Therefore, they require slow hydraulic pressing and multi-stage and time-consuming heat treatment [10]. Currently, a lot of research attention is devoted to the possibility of improving the properties of magnesium alloy castings in thermoplastic processes [11].

In the traditional approach, a distinction is made between magnesium alloys used for castings and those intended for plastic processing [12]. Even though the AZ91 alloy is classified as a casting alloy, attempts are being made to improve its properties through heat and plastic treatment [13]. In work [14], the use of various heat treatment variants (T4, T5 and T6), the AZ91 alloy cast into metal and sand mold was analyzed. The work showed that the use of T6 treatment (supersaturation at 416 °C for 16 h., cooling in air and aging at 170 °C for 16 h.) results in the greatest increase in tensile strength and hardness. The authors of the paper [15] proposed the use of homogenization of the AZ91 alloy at a temperature of 380 °C for 15 h. in order to dissolve most of the β -Mg17Al12 phase while maintaining the average grain size of approximately 100 µm. further increasing the homogenization time and temperature results in significant grain growth, in their opinion. In the work [16] the possibilities of direct extrusion of the AZ91 alloy was examined. It has been shown that the use of homogenization at 430 °C for 12 h. immediately before the extrusion process significantly increases the plastic properties of the alloy and improves the quality of the products. Similarly, in the work [17], it was proposed to homogenize castings at a temperature of 420 °C for 12 hours. In turn, the authors of work [18] propose the use of homogenization of the AZ91 alloy at a temperature of 380 °C for 19 h., before extrusion. Then Supersaturation at 415°C for 4 h. and Aging 175°C for 16h. Attempts are also made to deform without homogenization. The authors of the [19] paper indicate the possibility of effective shaping of raw castings from the AZ91 alloy in the range of 250-350 °C with an extrusion ratio of 4:1 and extrusion speed of 0.3 mm/s. In the case of using the casting homogenization process, the literature reports indicated above are divergent, however, in the case

of post-deformation machining, the prevailing view is that the best improvement in mechanical properties is obtained for the AZ91 alloy when it is annealed for several hours above 400 °C and aged at a temperature of around 150–200°C for several hours (usually 8–24) to allow the precipitation of secondary phases such as Mg17Al12, which contribute to the alloy's strength and hardness [19,20].

In the authors' opinion, it is possible to reduce the costs of products and simplify the technological scheme for making products from magnesium alloys by using appropriate parameters of heat treatment and plastic deformation of castings, while maintaining functional parameters at an acceptable level [21]. The objective of the present study was to determine the impact of heat treatment and metal forming parameters on the structure of cast AZ91 magnesium alloy.

Research methodology

The present study surveyed the magnesium alloy whose chemical composition was outlined in Table 1. The chemical composition tests were carried out at the stage of delivery of castings for testing. Cylindrical samples were (\emptyset 20×30 mm) prepared from ingots cast into sand molds by mechanical processing. The set of samples prepared for testing is shown in Figure 1.

Samples from the AZ91 alloy were homogenized at 385 °C, 400 °C, 415 °C and 430 °C with argon as a shielding gas for 24 hours and then quenched in water. An electric furnace LAC (Czech Republic) was used for this purpose. After homogenization, the microstructure was assessed and hardness tests were performed. On this basis, the material was qualified for further tests. In

Table 1. Chemical composition of the AZ91C magnesium alloy

Tuble II chemical composition of the fills for magnesiant anoy									
AI	Zn	Mn	Si	Cu	Ni	Mg			
8.1-9.3	0.40-1.0	0.13-0.35	0.3	0.1	0.01	balance			



Figure 1. Example cylindrical samples used in the study (Ø20×30 mm)

the next stage, the cylindrical samples underwent upsetting tests (Fig. 2). The following sample temperature conditions were adopted for the study: 380 °C, 400 °C, 420 °C and 440 °C. Upsetting dies were heated to 200 °C. The samples were upset to two final heights for each temperature, i.e. 15 and 10 mm. As a consequence, two logarithmic deformation values were obtained: $\varepsilon = 0.7$ and 1.1. After upsetting, the samples were water- and aircooled. The criterion for failure during upsetting was cracks on the side surfaces of the samples, which were qualified based on visual assessment (Fig. 3). Samples without cracks were heat treated.

Subsequently, samples without cracks underwent supersaturation at 415 °C for 6 h with argon as the shielding gas, and artificial aging at 175 °C for 24 h. After these thermo-plastic processing, samples were again subjected to metallographic tests and hardness tests were also performed. Metallographic tests were performed on cross-sections of cylinders and samples after upsetting. The central area of the samples (area marked 1) was analyzed. Preparation of metallographic sections was carried out by grinding on abrasive papers of various grains and polishing using diamond suspensions. At the final stage of polishing was used an OPS silica suspension $(0.05 \ \mu m)$. After the above steps, the samples were etched by immersion and gentle stirring for 5-15 seconds in a solution of the following composition: 100 ml ethanol, 10 ml distilled water, 10 ml acetic acid, and 5 g picric acid etchant. Then, the samples microstructure was examined using Nikon MA200 microscope. Vickers hardness was also tested with the Future-tech FM800 hardness tester. The measurements were made on the HV 0.5 scale in accordance with the PN-EN ISO 6507-1:2006 standard. The tests were performed in the central section of the samples by making 6 impressions on each of the tested samples.

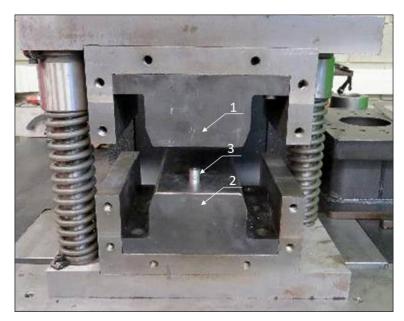


Figure 2. Device for upsetting samples made from the cast AZ91 alloy: 1 - upper die, 2 - lower die, 3 - sample

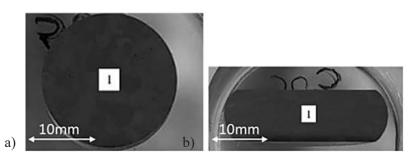


Figure 3. An example of the metallographic specimen from the cylindrical AZ91 magnesium alloy samples before upsetting (a) and after upsetting (b) with the area marked for structural examination

Analysis of the results

The microstructure of the AZ91 alloy after casting is presented in Figure 4 (a-b). Its morphology revealed the emergence of a base (α -Mg) and numerous secretions of networks of intermetallics, probably γ (Mg17A112). Non-equilibrium eutectics are also present around the secretions. In the case of such inhomogeneities, the deformation cannot be uniform and hard particles of precipitates constitute possible nodes for crack development. Figure 5 (a-d) shows selected structures of the AZ91 magnesium alloy after homogenization under different temperature conditions. It can be observed that the application of a temperature of 385 °C did not cause significant structural changes (Fig. 5a). Most of the eutectic areas were dissolved in the solid solution, but the vast majority

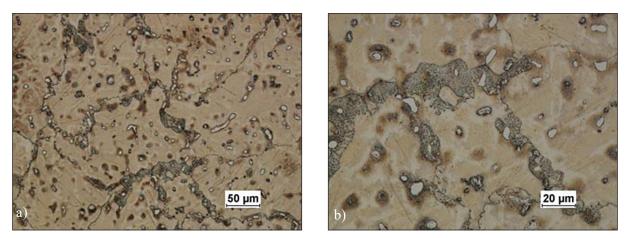


Figure 4. Microstructure of the AZ91 alloy after casting

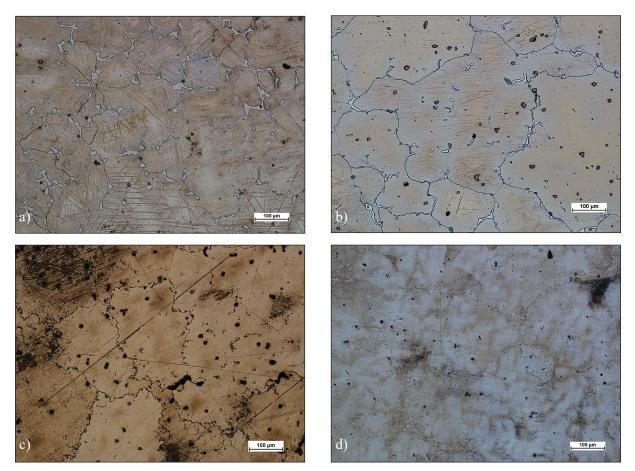


Figure 5. Microstructure of the AZ91 alloy after homogenisation at: (a) 385 °C, (b) 400 °C, (c) 415 °C, (d) 430 °C

of the second phase precipitates remained. A similar effect was obtained for a temperature of 400 °C (Fig. 5b). The volume fraction of the second phase is slightly lower, but complete dissolution has not been achieved. The best results were achieved at a temperature of 415 °C (Fig. 5c). A single solid solution was obtained in the entire studied area, without any second phase precipitation. In this case, there was no significant grain growth, which was observed after homogenization at a temperature of 430 °C (Fig. 5d). Due to the favorable structural effects, samples homogenized at 415 °C for 24 hours and cooled in water were selected for the forging tests. The analysis of the capacity for deformation of the cast AZ91 magnesium alloy was conducted based on the visual inspection of the upset samples developed under varying process parameters such as forging temperature and deformation. The emergence of defects in the form of cracks disqualified samples. Table 2 outlines the results of upsetting tests. Based upon Table 2, crack-free samples were obtained for a deformation value equal to 0.7 for the upsetting test conducted at 420 °C. Examples of samples after the upsetting test carried out at various temperatures are presented in Figure 6. For the deformation value of 0.7, cracks in samples emerged in each of the surveyed temperatures. The microstructures of the AZ91 alloy after the upsetting test are presented in Figure 7 (a-f). In the case of a temperature of 380 °C, significant heterogeneity is visible both for cooling in water (Fig. 7a) and

 Table 2. Results of upsetting tests of AZ91 magnesium alloy

Cracking							
Cooling medium	Forging temperature (°C)						
after forging	380	400	420	440			
Deformation value: ε = 0.7							
Air	yes	yes	no	yes			
Water	yes	yes	no	yes			
Deformation value: ε = 1.1							
Air	yes	yes	yes	yes			
Water	yes	yes	yes	yes			

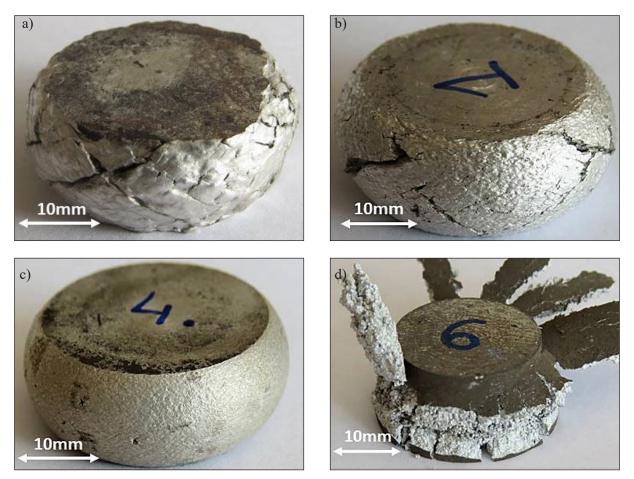
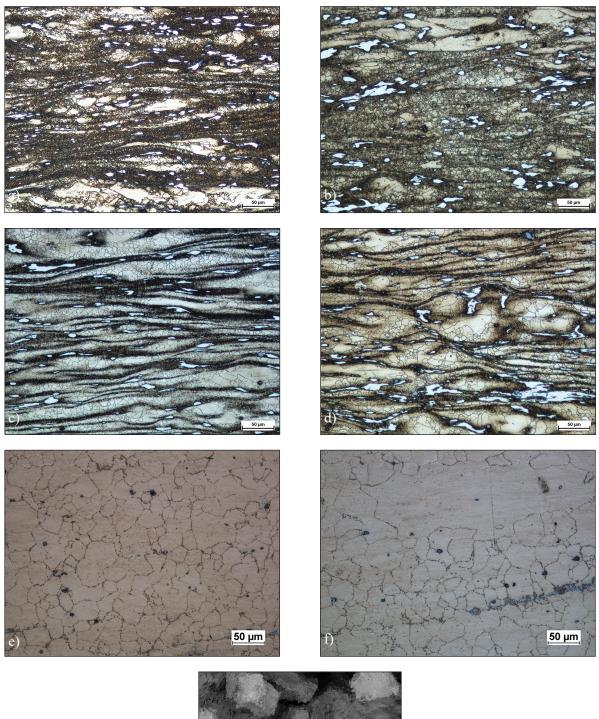


Figure 6. Photographs of selected samples from the AZ91 magnesium alloy after upsetting in temperature: (a) 380 °C, (b) 400 °C, (c) 420 °C, (d) 440 °C



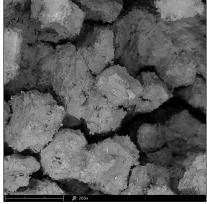


Figure 7. Microstructures of the AZ91 alloy after homogenization and upsetting in: a) 380 °C and water-cooling; b) 380 °C and air-cooling; c) 400 °C and water-cooling; d) 400 °C and air-cooling; e) 420 °C and water-cooling; f) 420 °C and air-cooling, g) 440°C (SEM)

g)

in air (Fig. 7b). Significant banding of the microstructure was observed, indicating the heterogeneity of the strain distribution. In the slip bands, small subgrains resulting from the deformation mechanism and large grains with signs of recrystallization are visible. The bright spots of secondary precipitates are an undesirable phenomenon and indicate an incorrect course of the process during heating to the forging temperature. Most likely, these are secondary precipitates that appeared as a result of slow heating of the charge for the forging process. Long-term exposure to temperatures below the solubility limit could lead to the release and coagulation of secondary precipitates. No significant structural differences were identified in relation to the cooling method, only the size of the finest recrystallized grains is larger in relation to those visible in the water-cooled sample. The microstructures obtained for samples forged at 400 °C are analogous to those discussed earlier. banding is slightly smaller and the strain distribution is more uniform. Again, there are no significant structural differences in relation to the cooling method used: (Fig. 7c - water), (Fig. 7d – air). The samples after forging at 420 °C have significantly different structural morphology than the others. Traces of banding are marginal. Grains on the entire analyzed surface were subjected to the process of dynamic recrystallization and have regular, equiaxed shapes. Structural heterogeneity in terms of grain size is the smallest in this case for both the water-cooled sample (Fig. 7e) and the air-cooled sample (Fig. 7f). Detailed fractographic observations of damaged samples after forging at 440 °C (Fig. 7g) revealed that the dominant mechanism of destruction was the

formation of many intergranular cracks as a result of weakening the boundaries of the grown grains. As a result of the carried out tests, samples were selected for the precipitation hardening process after homogenization at a temperature of 415 °C and forging at a temperature of 420 °C with different cooling methods. Process included supersaturation at 415 °C for 6 h with argon as the shielding gas, and artificial aging at 175 °C for 24 h. Figure 8 (a,b) shows the microstructures after precipitation hardening.

The microstructure of the AZ91 alloy after precipitation hardening is characterized by the presence of numerous lenticular secondary precipitates of a hardening nature. The precipitates are distributed dispersively in the grain space. It forms a needlelike microstructure with an overall regular distribution except in certain areas where the secretions are densely packed. As a result of heat treatment, proper distribution of reinforcing particles was obtained. It can be observed that in the case of samples after cooling in water (Fig. 8a), the precipitates are finer and their distribution is more uniform compared to those after cooling in air (Fig. 8b). The results of hardness tests are outlined in Table 3. It can be observed that the hardness of the homogenized material declined in relation to the raw state. It stems from the dissolution of secretions of the reinforcing phase. After forging, a significant growth of hardness was observed in relation to the material after homogenization. It is a consequence of compression. After precipitation hardening, hardness increased in relation to that of the alloy after forging. The study revealed that it arises from fine secretions of the reinforcing intermetallics. The analysis of

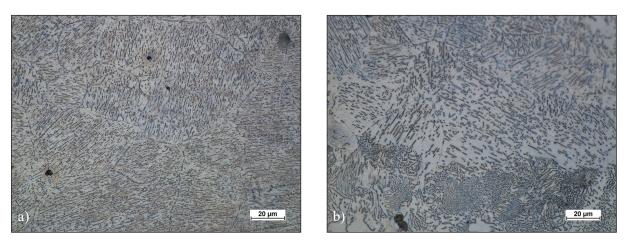


Figure 8. Microstructure of the AZ91 alloy after homogenization, upsetting at 420 °C: (a) water-cooling; (b) air-cooling, supersaturation, and artificial aging

No.	State of the alloy	Average from microhardness tests (HV05)	Standard deviation
1.	Cast	78.5	4.0
2.	Cast after homogenization at 415 °C for 24h	73.3	3.7
3.	Cast after homogenization and forging at 420 °C and after water-cooling	77.9	1.6
4.	Cast after homogenization and forging at 420 °C and after air-cooling	78.9	2.3
5.	Cast after homogenization and forging at 420 °C and after water-cooling, supersaturation, and artificial aging	93.1	2.8
6.	Cast after homogenization and forging at 420 °C and after air-cooling, supersaturation, and artificial aging	87.2	2.9

Table 3. Hardness of samples from cast AZ91 magnesium alloy at different stages of thermo-plastic processing

the results suggests that the thermo-plastic processing is valid. The precipitation-hardened alloy (after homogenization at 415 °C) improved its hardness by 15% in relation to the cast material. The highest hardness was obtained for samples homogenized at 415 °C, forged at 420 °C, watercooled, supersaturated, and artificially aged.

CONCLUSION

The article outlines the results of experimental studies concerning the impact of heat treatment and metal forming parameters on the structure of sand-cast AZ91 magnesium alloy. The study aimed to determine the most favorable processing conditions for the cast alloy. Based upon upsetting tests, the deformation capacity of the sandcast AZ91 magnesium alloy was determined in view of variable processing parameters such as temperature, degree of deformation, and means of cooling immediately after forging. On the basis of the results of homogenisation, upsetting tests and the examination of the structure and hardness, the most favorable thermo-plastic conditions for the AZ91 alloy were established. Preferably, before deformation, heat treatment of the alloy should be conducted in the form of homogenization at 415 °C for 24 h with argon as the shielding gas. As far as the structure and hardness are concerned, among the studied thermo-plastic processing parameters in the 380-420 °C range, the forging of the sand-cast AZ91 magnesium alloy at 420 °C with water-cooling and 0.7 deformation seems the most favorable. After thermo-plastic processing of the alloy, further heat treatment in the form of supersaturation at 415 °C for 6 h with argon as the shielding gas as well as artificial aging at 175 °C for 24 h are recommended. When analyzing the

structure of the AZ91 alloy after forging and cooling, flow strands with considerable fragmentation of grains due to dynamic recrystallization are visible. In between the flow strands, dynamically recrystallized grains of larger size are visible. Due to the heat treatment after deformation, full recrystallization occurs. Additionally, the size of grains across the cross-section becomes homogeneous. Hardness analysis of the cast and deformed AZ91 magnesium alloy suggests that the material becomes strengthened due to the heat treatment. The highest hardness emerges after forging at 420 °C and water-cooling and in the course of further heat treatment (supersaturation and artificial aging). Despite the use of the homogenization process, it was not possible to obtain such a wide range of the forging process window as in some of the publications discussed, e.g. [19]. This may be due to the lower strain rate used. Additionally, the use of a homogenization or supersaturation temperature below 400 °C [15] does not allow for full homogenization of the Az91 alloy.

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