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MICROSTRUCTURE OF MAGNESIUM ALLOY AZ31 AFTER LOW-SPEED EXTRUSION

1. INDUSTRIAL MOTIVATION

Extrusion of magnesium alloys, in general, does not differ a lot from extrusion of other metals. Magnesium alloys are warm and hot extruded. Deformation dependence on temperature allow obtaining wide range of mechanical properties due to temperature variation from hot working range, where the material is entirely recrystallised to warm and cold working range, where deformation takes place below recrystallization temperature and so significant amount of deformed material remains in work-hardened condition. The latter makes it possible to control and influence the final properties of extrudates, therefore lowered temperatures are often used to attain higher strength, which is of great significance, as magnesium alloys, contrary to aluminium alloys or steel that undergo additional treatment after extrusion, are provided their final properties directly after extrusion [1]. Although higher loads are needed, lower extrusion temperatures make it also possible to increase extrusion rate preventing from surface defects formation in the aftermath of deformation heat generated.

In extrusion magnesium alloys working speeds are lower than with aluminium alloys. The highest speeds (50 m/min) are used for AZ31 magnesium alloy in hydrostatic extrusion processes. However, good surface quality can provide extrusion speed below 34 m/min [2, 3]. At 50 m/min so called “orange skin” effect occurs.

AZ31 magnesium alloy is the most ductile and the most popular amongst AZ wrought alloys (Mg-Al-Zn group). Soft as it is, this alloy offers good combination of strength and ductility for structutral application after deformation with severe reductions. Parameters of main groups of products made of this alloy are presented in Table 1.

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Table 1. Mechanical properties of AZ31B magnesium alloy [1]

Type of products	UTS	TYS	Elongation	Hardness	Shear strength	UCS
	MPa	MPa	%	HB	MPa	MPa
Rolled sheets	290	220	21	73	160	180
Extrudates	255	200	12	49	130	97
Forgings	260	170	16	50	130	–

Sometimes extrusion is carried out in order to prepare the material for further deformation, for instance, impression-die forging. In such cases, the extrusion process is not aimed at obtaining required level of strength, but appropriate quality of the forging stock. The criteria of determining extrusion process conditions, such as temperature and/or reduction ratio, is then providing proper size and shape of grains as well as microstructure uniformity and homogeneity, with care of β-phase precipitates, which are known to adversely affect plasticity [4, 5].

Generally, it is maintained that to complete breakdown of a cast structure throughout the whole cross-section, reduction ratio of at least 50 should be assumed [6]. Provision of such large deformations significantly affects production rate, as well as cross-sectional dimensions of produced extrudates due to limited capacities of available equipment. Also tool life is shorter. Lower reduction ratios in extrusion can improve process efficiency of both extrusion and die forging processes, if properties requirements are met.

As mentioned above, despite providing higher production rate, higher speeds are accompanied by significant temperature increase. Therefore, lower process temperatures are assumed so that the increase in temperature during extrusion due to deformation heat should not exceed the incipient melting point. In result of lower temperatures higher loads are observed. In addition, high speeds are found to influence the mechanical properties of extrudates in an adverse way. For that reason application of lower working speed to obtain higher strength is addressed in the paper with microstructural changes taken into consideration.

The effect of extrusion process conditions on the final properties and microstructure of profiles extruded of magnesium alloys has been a subject of numerous studies. However, results presented in papers most often concern problems of extrusion of pipes or profiles as a finished products or focus on the issues of producing fine-grained structures to employ superplastic flow conditions. Since extrudates of smaller dimensions are characterized by more uniform profile of deformation in a cross-section, process conditions used there may not be suitable for bigger cross-sections. As such, extrusion of bars designed for forging stock call for determination of conditions which will guarantee microstructure providing satisfactory plasticity and required level of final properties of final parts.

2. EXPERIMENTAL PROCEDURES

The goal of the study is an investigation on the influence of extrusion process conditions upon the microstructure changes in as-cast AZ31 magnesium alloy with a focus on changes in grain size and its homogeneity after extrusion in variable temperature. The results are to show the possibility of grain refinement in a low-speed extrusion process with small values of reduction ratio. Such conditions are meant to prevent from deformation heat generation, providing near constant temperature on the exit of the die orifice.

2.1. Research material

Microstructure of the as-cast AZ31 alloy specimens used in the studies are shown in Figure 1. Average content of alloying elements in the alloy was: 2,4–3,6% Al, 0,8–1,4% Zn, 0,35% Mn, 0,1% Si, 0,05% Cu. Grain size estimated with a comparative method suggested coarse-grained structure, ranging from 1 to – 1 ASTM.

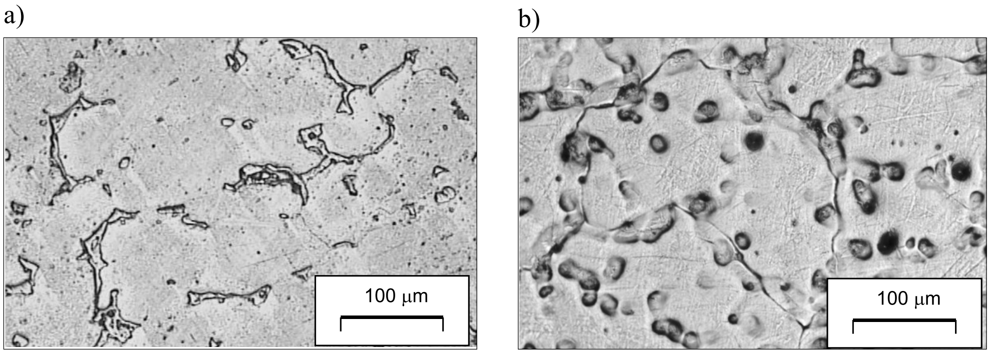

Fig. 1. *Microstructure of AZ31 magnesium alloy: a) as-cast; b) after homogenization*

Before extrusion the specimens cut out of an ingot were subjected to 2 hours annealing at 400°C. Both, before and after homogenization, large precipitates, identified as $Mg_{17}Al_{12}$, were present in the structure. However, after annealing they have partially dissolved, reaching eventually up to 50 μm and, as it can be seen in Fig. 1b, underwent spheroidization process.

2.2. Extrusion tests

The tests were carried out on a hydraulic press of 100 ton capacity. Assumed elongation coefficient defined as

$$\lambda = \frac{S_0}{S_1} \tag{1}$$

where:

S_0 – cross-section of a billet,
S_1 – cross-section of an extrudate,

was equal $\lambda = 3$. A scheme of the extrusion rig is shown in Figure 2. On account of small reduction ratio, to reduce unfavorable effect of friction on uniformity of strain in the cross-section of extruded bar and in result – microstructure, as well as, the hazard of surface cracking occurrence [7], generous lubrication with MoS_2 was applied.

Prior to extrusion die, container and punch were heated to 150°C. Temperature measurement was realized with independent thermocouples digitally controlled. To avoid excessive heat generation and/or surface defects formation, low velocity was established, equal to $v = 1$ mm/s.

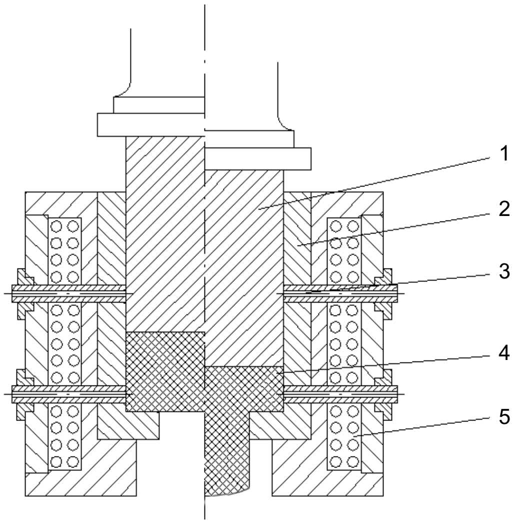

Fig. 2. *Extrusion rig: 1 – punch, 2 – die, 3 – thermocouples, 4 – billet, 5 – heating system*

2.3. Metallographic work

Microstructure examination was performed on transverse relieves of extruded bars. The specimens were mechanically polished with 1 µm and ¼ µm diamond paste and etched. To reveal grain microstructure etchant composition: 20 ml CH_3COOH + 3 g $C_6H_2(NO_2)OH$ + 20 ml H_2O + 50 ml C_2H_5OH was used. For observation of the microstructure optical microscope NU2 was used.

The investigation included specimens extruded with a constant reduction ratio λ, at constant velocity and variable temperature. Obtained micrographs of as-extuded alloy AZ31, shown in Figures 3a–d, indicate increasing inhomogeneity of microstructure, evinced in variation in grain size. All the grain structures shown in Figure 3 are generally fine-grained, without any distinct orientation in a microscopic scale. However there are significant differences between various extrusion temperatures. As seen in Figures 3a and 3b, grain structure of specimens taken from bars extruded at 300÷350°C is composed of eqiaxial grains uniform in size with scarce protruding, non-recrystallized ones. In turn, in specimens obtained at temperature 250°C two kinds of areas can be distinguished: fine-grained and intersecting them zones of coarser grains (Fig. 3c). In temperature 200°C the inhomogeneity increase and in the structure numerous deformation twins are observed (Fig. 3d).

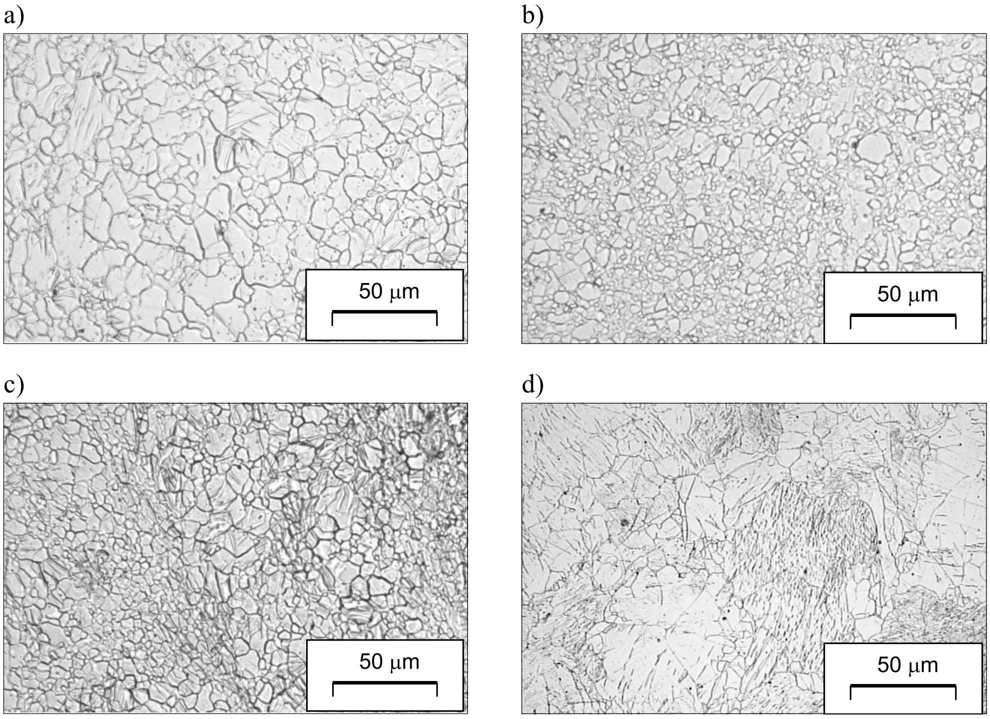

Fig. 3. *AZ31 magnesium alloy extruded at: a) 350; b) 300; c) 250; d) 200°C*

3. ESTIMATION OF RELATIVE SURFACE OF GRAIN BOUNDARIES

For quantitative interpretation of the microstructure observation a stereological parameter S_V that is, specific surface area of grain boundaries was used. Estimation of this parameter was carried out by means of linear intercept [8], which is based on assumption

$$S_V = 2P_L \tag{2}$$

where P_L – relative number of intersection points of a secant with grain boundaries.

An elementary measurement used in the method pedends on the number of intersections of a specified-length secant with grain boundaries. The measurements were conducted on the micrographs (specimens 3 and 4) or directly on a relief (specimens 1, 2, 5 and 6) with help of micro-eyepiece with a special gauge insert. Linear enlargement applied was dependent on grain size.

Absolute limit error of S_V estimation was determined as follows:

$$\delta_{S_V} = \frac{2u_\alpha s_{P(L)}}{L_{rz}\sqrt{n}} \tag{3}$$

and the relative limit error γ_{Sv} with a formula

$$\gamma_{Sv} = \frac{\delta_{Sv}}{S_V} \tag{4}$$

where:

$u\alpha$ – quartile of normal distribution for level of confidence $1 - \alpha = 0{,}95$, $u\alpha = 1{,}96$,

$s_{P(L)}$ – standard deviation of intersects number in the secant of length L,

L_{rz} – actual length of the secant,

n – number of secants.

The presented results of the quantitative analysis (Tab. 2) are confirmation of the above-mentioned qualitative observations. There is a distinct discrepancy between S_V value estimated for specimens couple 1 and 2 or couple 3 and 4.

Table 2. Quantitative characteristics of microstructure of extruded AZ31 magnesium alloy

Specimen number	Extrusion temperature, °C	S_V, mm^{-1}	δ_{Sv}, mm^{-1}	γ_{Sv}, %	ASTM	HV5
1	ingot	6,76	0,85	12	–1	42
2	ingot	17,8	0,77	8,6	1	45
3	350	289	15,7	5,4	5÷5,5	53
4	300	338	24,0	7	5,5	61
5	250	Method inapplicable			5,5÷6	74
6	200				5÷6	78

Values of relative surface of grain boundaries calculated for specimens 3 and 4 (380 and 300°C, respectively) are 338 mm^{-1} and 289 mm^{-1}, which indicates significant grain refinement after extrusion of as-cast structure which prior to extrusion was characterized by relative surface of grain boundaries equal to 6,76 mm^{-1} and 17,8 mm^{-1} that stands for coarse-grained structure. Value of relative error γ_{Sv} (Tab. 2) allow consideration the accuracy satisfactory. On account of significant in homogeneity and non-uniform shape of grains, grain size for specimens 5 and 6 was determined with a comparative method (Tab. 2).

4. HARDNESS MEASUREMENT

In order to represent an influence of the microstructure conditions, presented in the form of calculated estimators, hardness measurements were carried out. Vickers method was used, with a loading of 5 kg. The measurement was done both for as-cast and extruded material. Obtained results are presented in table 2 along with values of calculated stereological parameters and corresponding ASTM grain size.

5. SUMMARY AND CONCLUSIONS

The results of the investigation go along with the results of similar studies, which indicate decreasing grain size with temperature of deformation of AZ31 magnesium alloy. Linear dimensions of analyzed grain structure depend a great deal on extrusion temperature. Despite rather small reduction ratio, changes in the microstructure of extrudates, determined by value of relative surface of grain boundaries S_V are significant, which is reflected by increase of this parameter over a several dozen times as compared to initial grain size.

With a decrees in temperature a fraction of *eqiaxial* grains is decreasing, with increasing amount of deformation twins. In the structure also appear non-existing in specimens extruded in higher temperatures (300, 350^{o}C) β-phase precipitates.

Hardness measurements tend to illustrate the microstructural changes after extrusion. Despite significantly smaller reductions on the cross-section, the obtained hardness level exceeding 70 HV, are comparable to those obtained in extrusion in industrial practice [1, 2, 3]. High values of HV which speak for improvement of strength properties of the alloy can be attributed to more favorable cooling conditions during low-speed extrusion. Low speeds reduce the amount of generated deformation heat, decreasing temperature of extrudates, compensating for its adverse effect on the final properties. In case of large cross-sections, for example, forging billet for impression-die forging, it allows performing the deformation in higher temperatures, reducing the pressures and extrusion load with attained good surface quality, fine-grained and uniform microstructure.

Acknowledgements

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