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THE EFFECT OF EXTRUSION IN THE COMPLEX STRAIN STATE ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MgAlZn MAGNESIUM ALLOYS

The paper presents the results of tests concerning the effect of the extrusion process in the complex strain state on the microstructure and properties of one of magnesium alloy with aluminium, zinc and manganese, designated AZ61. Due to its specific gravity, it is increasingly being used in the automotive and aerospace industries to reduce the weight of structural elements. As a result of plastic deformation processes, rods with a diameter of 8, 6 and 4 mm were obtained from AZ61 magnesium alloy. The microstructure analysis was performed using light and electron microscopy (STEM) techniques in the initial state and after plastic deformation. Microstructure studies were supplemented with a quantitative analysis using the Metilo program. A number of stereological parameters were determined: average diameter of grain, shape factor. A static tensile test was carried out at 250ºC and 300ºC, at deformation rates of 0.01, 0.001 and 0.0001 $m \cdot s^{-1}$. Better plastic properties after deformation using KoBo method were obtained than with conventional extrusion.

Keywords: magnesium alloys, KoBo method, microstructure, electron microscope, shape factor

1. Introduction

Magnesium and its alloys are widely used in the metalworking industry. These materials combine density with mechanical and physical properties in such a way that makes them ideally suited for applications in light constructions. Therefore, they present a high potential for innovation [1-2]. Thanks to the application of magnesium with a density of 1.74 g/cm^3, a product's weight is reduced by 30%. The limited application of magnesium alloys, until now, is mostly due to their susceptibility to corrosion (their main flaw), flammability, low strength, high costs of processing by way of plastic working (they must be heated before forming), and poor mechanical workability [3-5]. Although magnesium alloys have many flaws, their application in various types of constructions may lead to a significant weight reduction, which is very desirable in certain cases. Many national and international research centres are currently conducting research on magnesium alloys, the main objective of which is to improve the properties and deformability of magnesium alloys using conventional and unconventional methods of deformation. There have been numerous efforts to process fine-grained or nano-structured Mg alloys, with the aim of obtaining specific mechanical properties, such as high strength and ductility. Among various approaches in grain refinement, severe plastic deformation (SPD) methods have attracted considerable research interest. These SPD methods include high-pressure torsion (HPT) [1], equal channel angular extrusion (ECAE) and equal channel angular pressing (ECAP), multiaxial forging (MAF) and multi-directional forging (MDF), accumulative roll bonding (ARB) as well as cyclic extrusion and compression (CEC), etc. [6-7]. However, the application of SPD methods in mass production remains challenging at present. Hence, it is necessary to provide a simple and efficient processing method suitable for industrial applications. Recently, one-step high-ratio extrusion has received attention due to its flexibility in use on Mg alloys with a great diversity of shapes, such as sheets, rods, tubes, etc. It has been confirmed that an intense deformation using one pass is more effective in obtaining ultrafine grains and a high fraction of high-angle grain boundaries than a high strain accumulated over multiple passes. Studies on high-ratio extrusion have been mainly concentrated on AZ31, AZ80 and AZ91 alloys. The article presents the research process conducted to assess the possibilities of plastic processing of alloy AZ61 with the application of Kobo extrusion method. The process of plastic processing conducted with the use of KoBo extrusion is one of the unconventional methods of plastic forming of metals

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and their alloys with the use of large deformations – SPD (Severe Plastic Deformation). KoBo method is based on the application of additional reversible rotation of the die by a given angle with the determined frequency. Deformation is conducted by rotation of the die until the viscoplastic material flow phenomenon occurs in the material [11]. It enables the replacement of high-temperature deformations of metals and their alloys with processes performed cold without the initial heating of the charge. It also enables the application of high speeds of processes and a significantly higher degree of deformation with smaller work of deformation. An additional advantage of the process is the achievement of beneficial mechanical properties which are not typical after application of other types of deformation [8-14]. Literature data show the possibility of plastic forming with the use of this method in case of conventional magnesium alloys WE43. The results achieved for conventional magnesium alloys were satisfactory. It is shown in the paper [3] that for alloy WE43 significant fineness of the structure was achieved together with beneficial mechanical and plastic properties when the KoBo deformation method was applied. For alloy WE43 there was an average grain diameter of about 0.9 μm [3]. That is why attempts were made to apply this method of deformation to alloy AZ61. After the extrusion process with KoBo method, an analysis of the microstructure and properties was conducted for alloys AZ61. Static tensile tests were performed at a temperature of 250ºC and 300ºC.

2. Material and Procedure

The materials for the extrusion process were ingots prepared from magnesium alloy AZ61 with a size of ϕ120 × 65 mm. The chemical composition of the tested alloys is presented in Table 1. Ingots underwent the process of extrusion to the diameter of 40 mm.

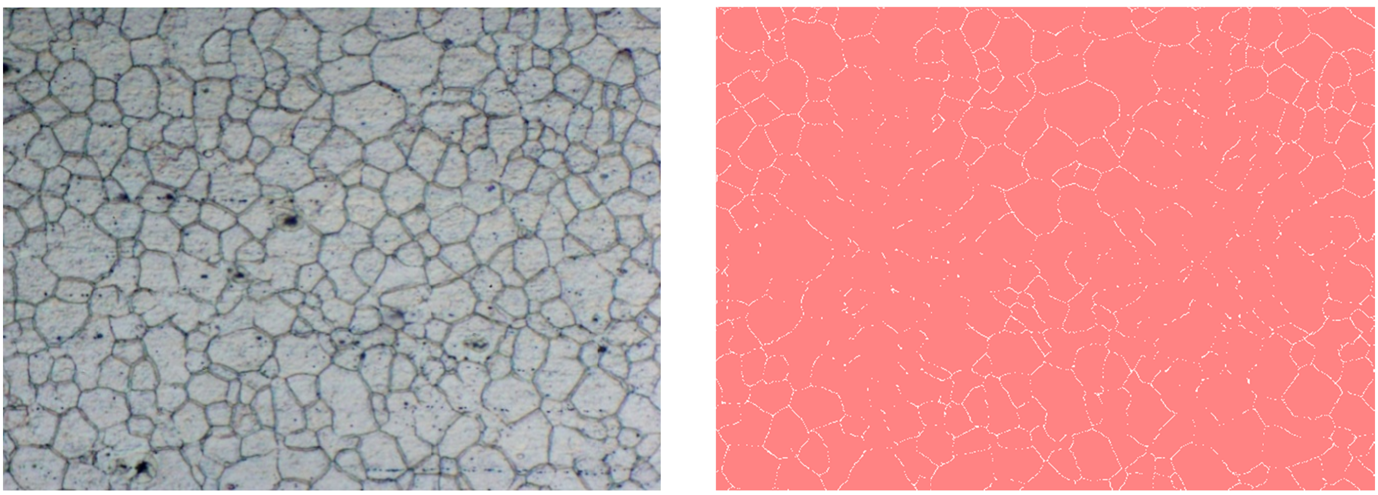
In the first stage of the research, ingots made of AZ61 alloy were subjected to the process of coextrusion and KoBo extrusion.

TABLE 1

Chemical compositions of magnesium alloy (mas.%)

	Al	Zn	Mn	Si	Fe	Mg	rest
AZ61	5,92	0,49	0,15	0,037	0,007	93,33	0,066

The coextrusion process was carried out at 400ºC with a pressure of 300 kN and a travel speed of 0.1 mm. As a result of this process a rod with a diameter of 8 mm was obtained. In the next stage, AZ61 alloy was directly extruded with the KoBo method at room temperature – without prior heating with the shift speed of the inverted die of 0.33 mm/s, die torsion angle of ±8° and frequency of 5 Hz. Rods with a diameter of ϕ 8, 6 mm and 4 mm were obtained as a result of extrusion and the processing degree λ was calculated for them, equalling 100. The material was intensively cooled with water at the press throat outlet. The static tensile test was conducted on a Zwick/Roll machine at a temperature of 250°C and 300°C, with a deformation rate of 0.01, 0.001 and 0.0001 $m \cdot s^{-1}$. Based on the results of the static tensile test, curves in the stress-elongation system were determined. The m index was determined to indicate the sensitivity to deformation rate $m = d\ln\sigma | d\ln\varepsilon$, where σ – plastic stress, ε – plastic deformation, [4]. Microstructure tests were conducted with the use of light and electron microscopy. The microstructure was analysed quantitatively with the use of Metilo program [15]. The grain size was measured with the use of the surface method based on the images recorded by light and scanning microscopes. Quantitative studies were conducted on properly prepared (polished and etched) metallographic specimens, recorded with the use of a light microscope in the bright field technique. Figure 1 shows an example of the microstructure of AZ61 alloy with grain boundaries marked red, which were the basis for the determination of stereological parameters. Images were recorded using a camera correlated with a PC and saved in graphic files. In order for the results to be considered statistically significant for one area of analysis, several images were recorded and loaded sequentially into the program. Results from several photographs were then averaged.



a) b)

Fig. 1. a) Microstructure of alloy AZ61, b) example of a microstructure with detection

An X-ray phase analysis was performed on an X'Pert[3] Powder diffractometer using a copper anode tube ($\lambda_{CuK}\alpha$ – 1.5406 Å) supplied with a 30 mA current at 40 kV. A PiXcel 1D strip detector with a monochromator was used. The data was recorded with a 0.02° step in the angle range from 10° to 90° 2θ. The tests were carried out on solid samples. Crystalline phases present in the material were identified using the database of the International Centre for Diffraction Data.

3. Results and Discussion

Figure 2 shows an example of the microstructure of AZ61 alloy and X-ray diffractogram in its initial state. AZ61 alloy in its initial state was characterised by a coarse grain microstructure with varied grain sizes (Fig. 2a). An average grain size was 14.3 µm. The phase composition of AZ61 alloy in its initial state was identified using an X-ray phase analysis. The presence of α-Mg solid solution was found (Fig. 2b).

Figure 3 shows examples of microstructures of AZ61 alloy rods with a diameter of 8, 6 and 4 mm obtained by coextrusion and KoBo extrusion processes.

The analysis of the microstructure of the rods after coextrusion and KoBo extrusion processes showed that the microstructure of AZ61 alloy was refined due to the recrystallisation process (Fig. 3). The substructure of AZ61 alloy revealed the presence of recrystallized areas and subgrains (Fig. 4). As a result of the application of the KoBo method, in the microstructure of the obtained rods with a diameter of 8, 6 and 4 mm grain refinement was higher (Fig. 3b-d) compared to conventional extrusion (Fig. 3a). An average grain size below 7 µm was obtained. The highest grain refinement (6.1 µm) was obtained for a rod with a diameter of 6 mm (Table 2). The analysis of the microstructure of AZ61 alloy after deformation using the KoBo method was supplemented with a quantitative analysis. The results of the quantitative characterisation of AZ61 are presented in Table 2.

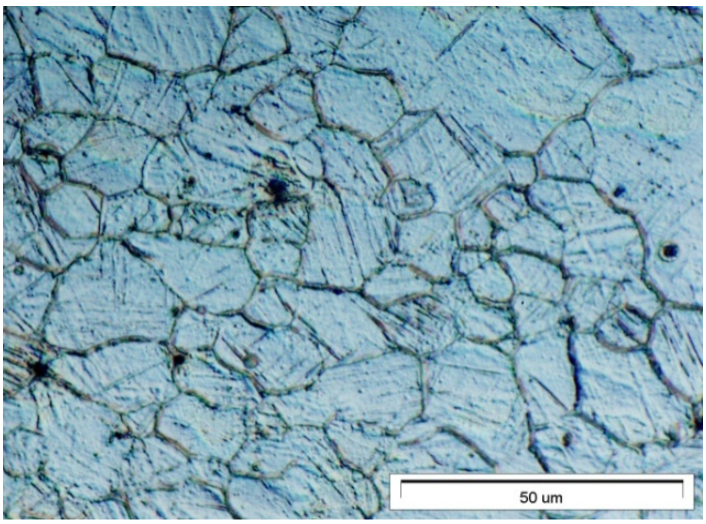
TABLE 2

Results of quantitative characterization AZ61

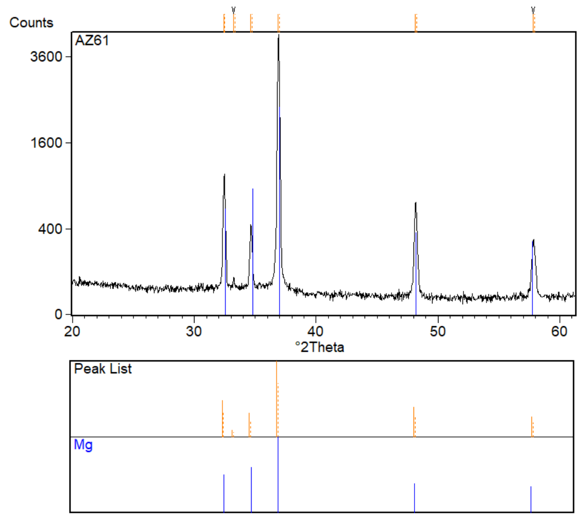
Alloy AZ61	Average equivalent diameter of grains [µm]	Average surface area [µm^2]	Shape factor
after extrusion-initial state	14,3	139,8	0,63
after hot extrusion rods with diameter of 8 mm	17,3	139,8	0,63
after cold extrusion by KoBo method rod with diameter of 8 mm	7,1	193,4	0,61
after cold extrusion by KoBo method rod with diameter of 6 mm	6,1	39,4	0,63
after cold extrusion by KoBo method rod with diameter of 4 mm	6,8	26,2	0,66

Fig. 5 shows examples of histograms of the surface share of grains after deformation using the KoBo method.

For the 8 mm rod diameter, on the basis of the analysis of the obtained distribution of the surface grain share, it was found that grains ranging from 0.06 µm to 0.24 µm, which constituted approximately 20% of the analysed surface, were present. Grains ranging from 0.96 µm to 15 µm constitute the largest share – approximately 70%. Grains ranging from 15 µm to 60 µm constitute a small share – approximately 10% (Fig. 5a). An average grain size is 17.3 µm. For the 6 mm rod diameter, grains ranging from 0.36 µm to 1.3 µm, which constituted approximately 20% of the analysed surface, were found in the microstructure. Grains ranging from 4.6 µm to 17 µm constitute the greatest share – approximately 70% of the analysed surface (Fig. 5b). An average grain size is 6.1 µm. Similar results were

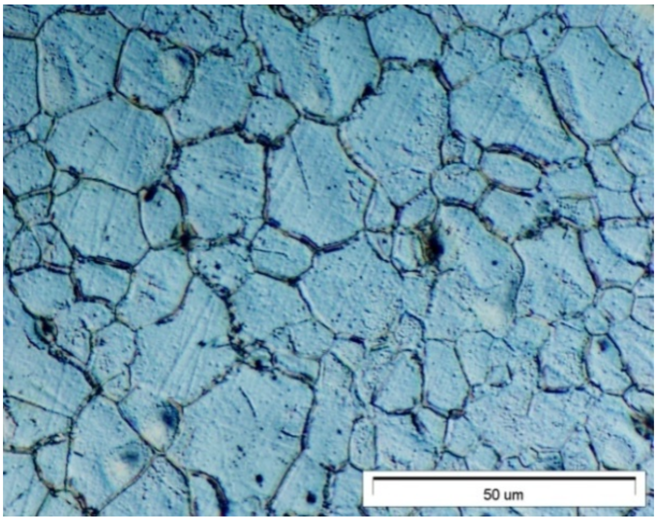


a)

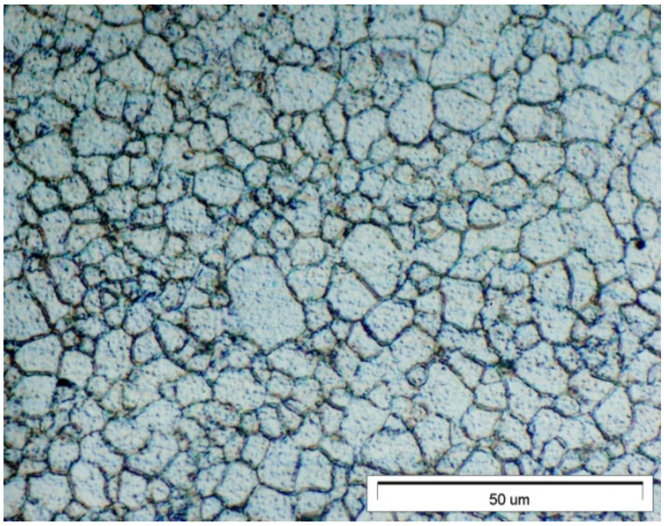


b)

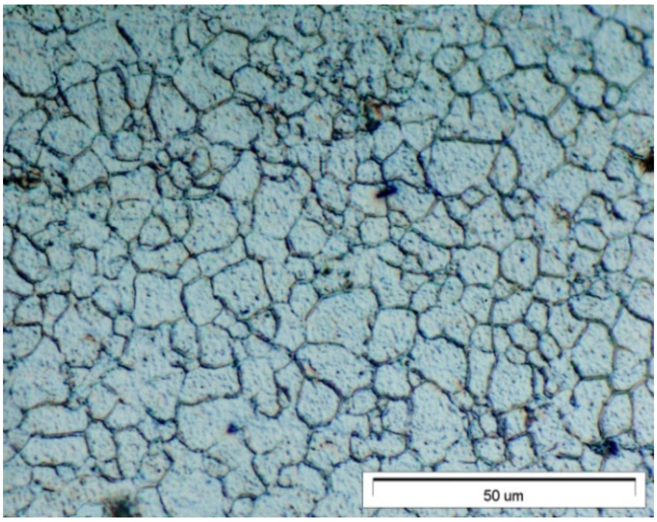
Fig. 2. a) Microstructure of alloy AZ61, b) X-ray diffraction pattern after extrusion – initial state



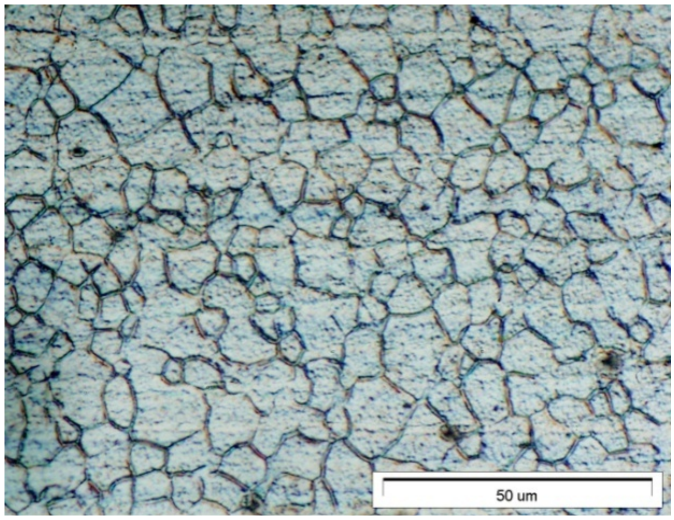
a) rod with diameter of 8 mm



b) rod with diameter of 8 mm

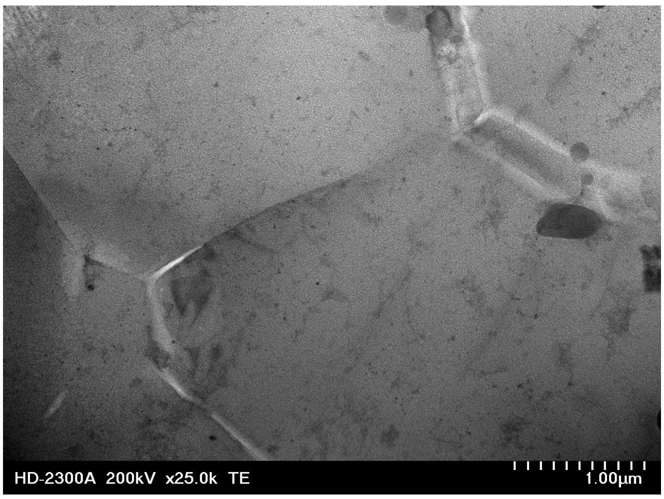


c) rod with diameter 6 mm

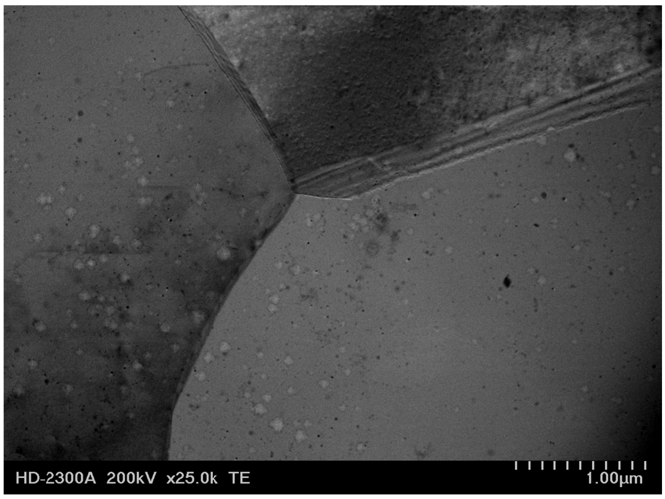


d) rod with diameter of 4 mm

Fig. 3. Microstructure of alloy AZ61 after plastic deformation a) conventional extrusion, b-d) KoBo method extrusion



a)



b)

Fig. 4. Substructure of alloy AZ61 after KoBo extrusion a) rod with a diameter of 6 mm, b) rod with a diameter of 4 mm; recrystallised areas; new subgrains/grains

obtained for a rod with a diameter of 4 mm. On the basis of the histogram of the grain surface share, it can be concluded that approximately 10% of the grains range from 0.36 μm to 1.3 μm. Grains ranging from 4.6 μm to 17 μm constitute the greatest share – approximately 80% of the analysed surface. This share is greater than for a rod with a 6 mm diameter. An average grain size is 6.8 μm (Fig. 5c). No grains between 0.1 μm and 0.36 μm were found in the microstructure of rods with diameters of 6 mm and 4 mm. The shape factor presented in Table 2 calculated for the AZ61 alloy microstructure after KoBo extrusion and conventional extrusion amounts to 0.63, which indicates the presence of grains with a shape similar to the equiaxial shape. Figures 6÷7 show the appearance of specimens after static tensile tests for different deformation rates of 0.01, 0.001, 0.0001 $m \cdot s^{-1}$, after conventional extrusion and KoBo extrusion.

For all the analysed deformation rates, higher elongation values after KoBo deformation (275%) were obtained in comparison to conventional extrusion, where only 185% elongation was obtained. On the basis of the results obtained from the static tensile test, curves (Fig. 8) in the stress-elongation system and parameter m – sensitivity to the deformation rate – were determined (Fig. 9).

This index makes it possible to identify the state of the deformed material as a superplastic state. A correlation was

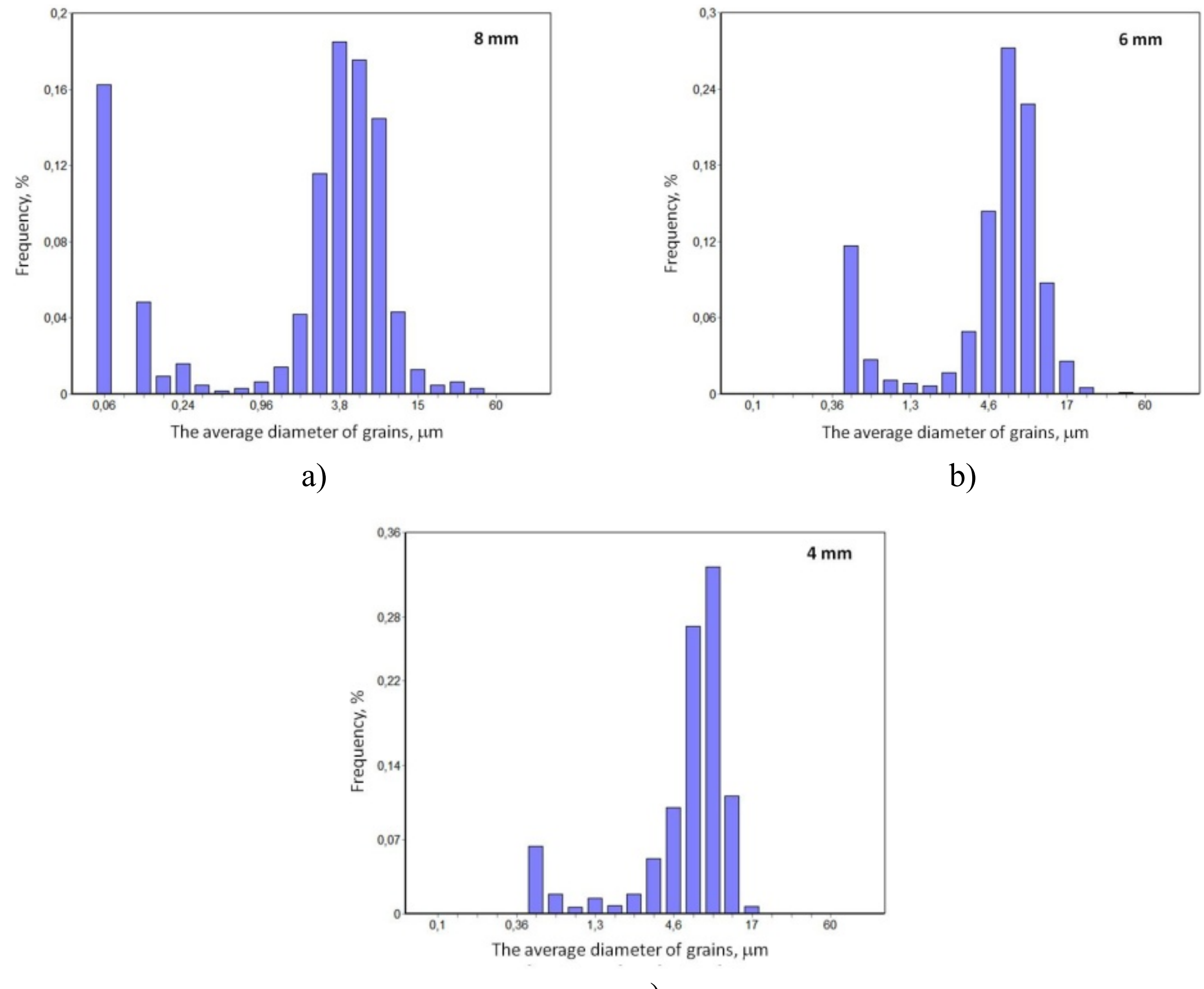


Fig. 5. Results of the quantitative analysis for AZ61 alloy after KoBo extrusion for rods with a diameter of: a) 8 mm, b) 6 mm, c) 4 mm

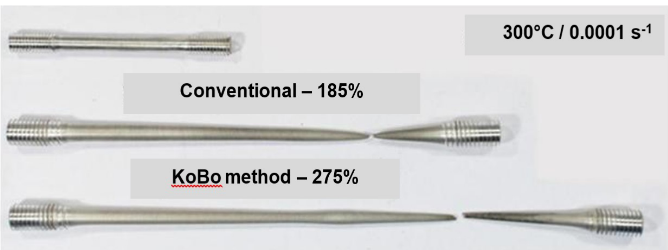


Fig. 6. View of samples for a static tensile test after conventional extrusion and extrusion by KoBo method

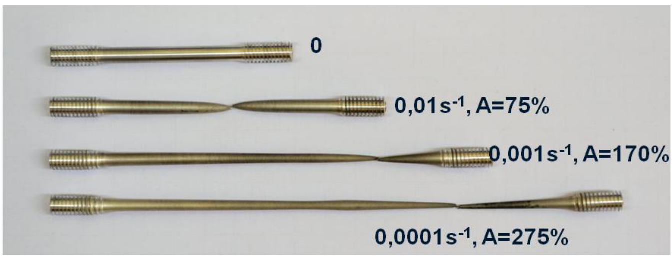


Fig. 7. View of samples for a static tensile test after extrusion by KoBo method

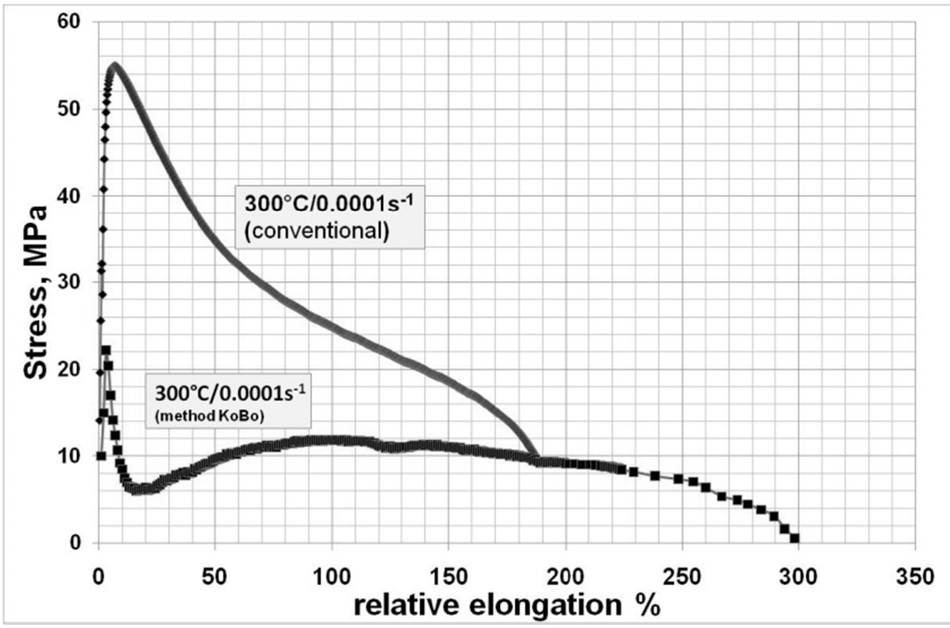


Fig. 8. Results of static tensile tests at 300°C, strain rate of 0.0001 $m \cdot s^{-1}$

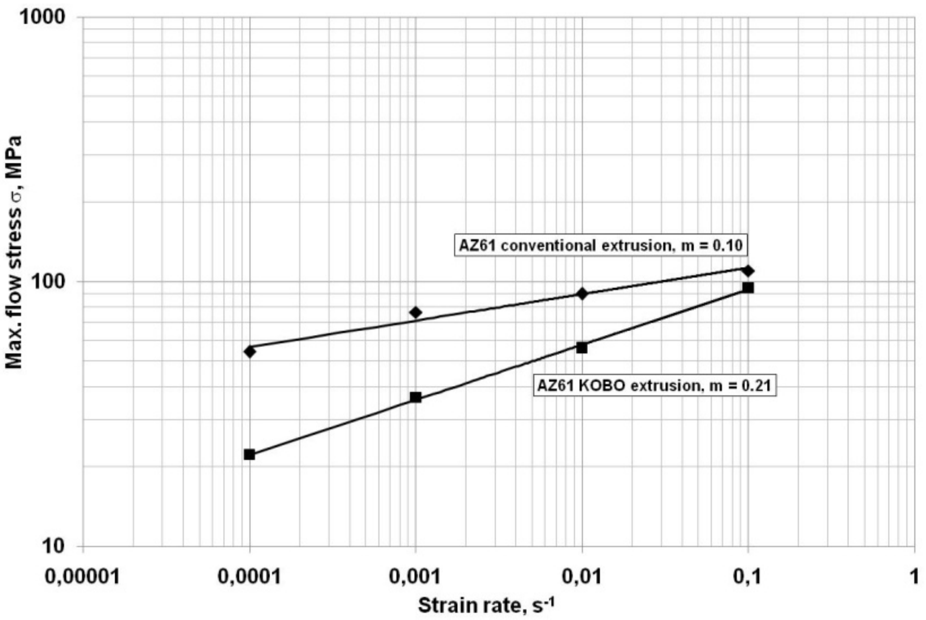


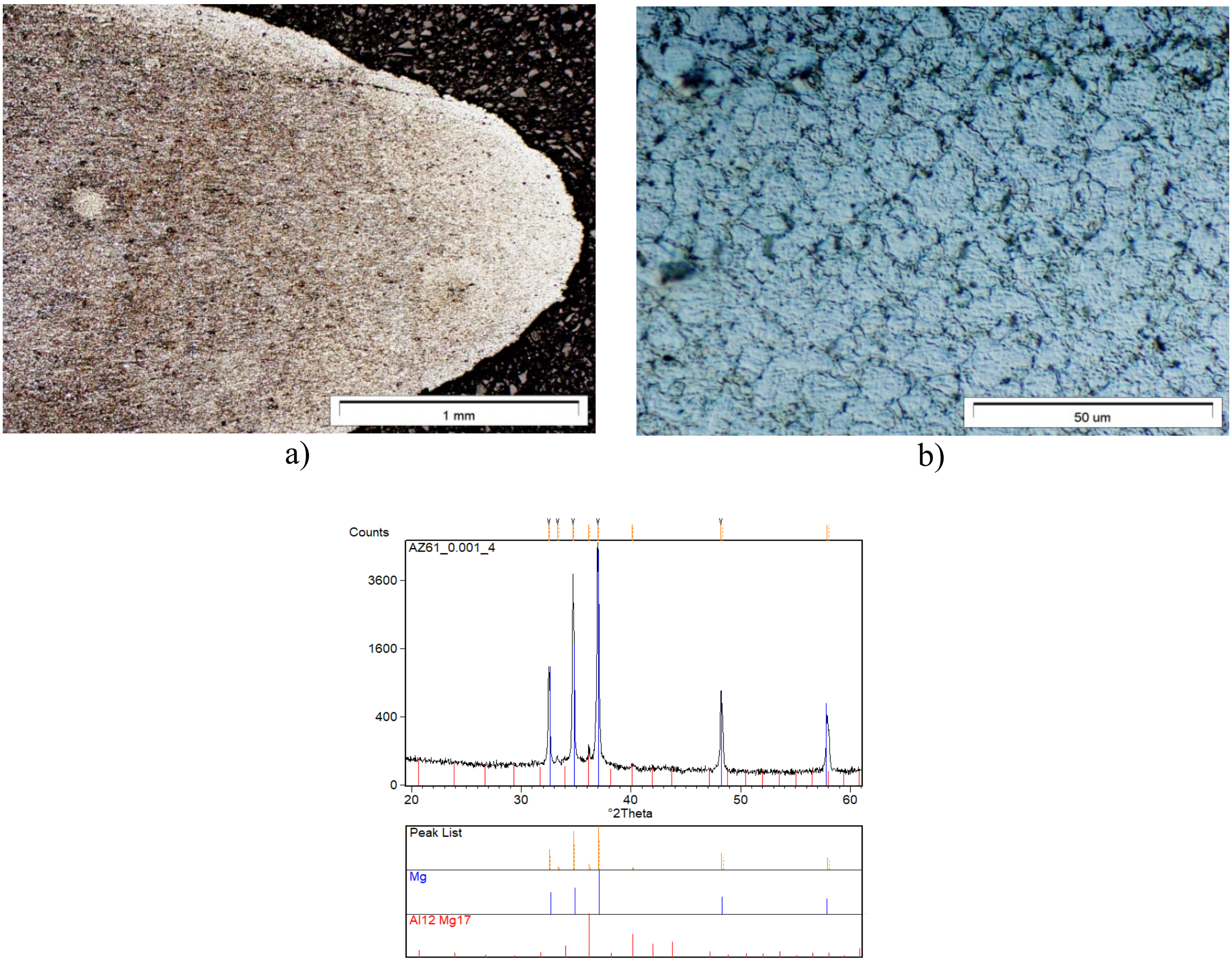
Fig. 9. M deformation rate sensitivity coefficient of AZ61 alloy after a static tensile test

found between the sensitivity to the deformation rate m and total elongation of the stretched specimen. The value of this coefficient for conventional extrusion is 0.10, while for KoBo extrusion it is higher and amounts to 0.21. The obtained value of the determined m coefficient may indicate susceptibility to superplastic flow for alloy AZ61 deformed by KoBo method (Fig. 9). The elongation value of 275% was obtained. Figure 10 shows the microstructure after a static tensile test for a rate of 0.0001 $m \cdot s^{-1}$.

In the microstructure of alloy AZ61, grain growth resulting from the elevated temperature was observed. Additionally, the $M_{17}Al_{12}$ phase (Fig. 10c) forming a band structure (Fig. 10b) was found at grain boundaries.

4. Conclusions

The achieved test results show good susceptibility to cold shaping of alloys AZ61 in the extrusion process with the use of KoBo method, which is not possible in conventional processes of plastic shaping which require an elevated temperature in the range of 300°C-400°C. As a result of the applied extrusion process, the refinement of the microstructure of the tested alloys was achieved. In the conducted test of deformation with the use of KoBo method there were rods achieved with diameters of 8, 6 and 4 mm. In microstructure of rod with diameter of 8 mm there was an average grain size of 7.1 μm achieved. There are grains which constitute 10% of the analysed surface ranging from 0.06 μm to



c)

Fig. 10. a-b) Microstructure of alloy AZ61 after static tensile test; c) X-ray diffraction pattern after static tensile test

0.24 μm. Grains ranging from 3.8 μm to 15 μm make up about 50% of the analysed surface in the analysed microstructure. In the microstructure of rod with diameter of 6 mm there was average grain size of 6.1 μm achieved. Grains ranging from 4.6 to 17 μm which make up 50% of the analysed surface in a variety of shapes, dominate in this surface. Average grain sizes for rod with diameter of 4 mm, however, were bigger in comparison to rod with diameter of 6 mm and equalled 6.8 μm. Grain sizes and their shapes achieved in microstructure of rod with 4 mm diameter are similar to the grain sizes and shapes in case of rod with diameter of 6 mm. It can be observed on the histogram of the surface share of grains that the grains ranging from 4.6 to 17 μm make up over 50% of the analysed surface. A small percentage is made up of grains ranging from 0.36 μm to 4.6 μm. It can be concluded, on the basis of results of quantitative analysis, that the analysed microstructures are heterogeneous with varied grain sizes and shapes. The microstructure obtained in the tested alloys after the extrusion process should provide good mechanical properties during their further shaping at an elevated temperature. The superplastic flow effect was demonstrated based on the results from the static tensile test. The results of the conducted tension tests at an elevated temperature of 300°C show that the tested alloy AZ61 has beneficial resistance properties and plastic properties.

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