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Influence of Deformation Process Parameters on Changes in Microstructure and Properties of Mg-8Li-2Ca Alloy

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Abstract

Currently, the aviation and automotive industries are seeing increasing interest in magnesium alloys. The manufacture of semi-finished and finished magnesium alloy products is mainly based on casting technology, which results from the good casting properties of these materials. There are some difficulties using plastic processing for magnesium alloys but it offers better mechanical properties. For these reasons, alternative ways of plastic forming are being sought for magnesium alloys. Research has also been undertaken into the development of production and forming technology of a new group of ultralight Mg-Li-Ca magnesium alloys, using conventional (classical extrusion) and unconventional plastic forming methods (KoBo extrusion). The paper presents the results of the study on plastic forming of Mg-8Li-2Ca alloy. The research material consisted of ingots with the dimensions ϕ 40 x 90 mm. Before the deformation process, the ingots were subjected to heat treatment. Classical extrusion tests and extrusion by SPD methods (KoBo) were performed. Using light and electron microscopy, the changes formed in the microstructure of the Mg-8Li-2Ca alloy in the initial state and after plastic deformation processes (classical extrusion, KoBo extrusion) are presented. Quantitative analysis of the microstructure of the Mg-8Li-2Ca alloy was performed using Metilo software after the deformation process. The mechanical properties were evaluated based on the results from the conducted HV0.2 hardness measurements and the static tensile test performed at room temperature.

Keywords: Magnesium Mg-Li-Ca alloy, KoBo method, Microstructure, Grain size

1. Introduction

Magnesium alloys are widely used as structural materials in the automotive and aerospace industries due to their low specific gravity and high specific strength [1-3]. As a metal, magnesium exhibits a hexagonal structure (HCP), which affects the limited plastic deformability and results in a wider application of magnesium alloys [4]. With regard to their manufacturing technology, magnesium alloys can be divided into magnesium casting alloys and alloys intended for plastic processing [5]. The shaping of the structure and properties of magnesium alloys for plastic forming can be carried out using conventional and unconventional deformation methods. Conventional methods include rolling, stamping, forging and drawing processes. These processes result in products/semi-finished products in the form of sheets, bars or wires [6-7]. Literature data [1÷3] provide many research results on the properties and microstructure formation of magnesium alloys after conventional deformation methods. These often provide an informative basis for further research into improving their mechanical properties, especially ductility.

Among the alloys intended for plastic processing, the ultralight alloys containing lithium in their chemical composition should be distinguished. Magnesium-lithium alloys have the lowest specific gravity of engineering materials, 1.35-1.65g/cm^3. They are characterized by high specific strength and specific rigidity. Mg-Li alloys are widely used in the construction of modern, ultralight metal structures. Medicine is an important future application area for magnesium alloys. Implants, surgical clips for connecting bones, and surgical threads are manufactured from magnesium alloys. Mg-Li alloys can also be used in these areas of the medical industry once the requirements are met. The presence of lithium favourably influences plasticity and reduces the density of the alloy, yet it deteriorates strength properties [8-10]. It is estimated that 1% by weight of lithium in a magnesium alloy reduces its density by about 3%. Additionally, it reduces corrosion resistance due to the high reactivity of lithium [9], resulting in limited technical applications for these alloys. Mg-Li alloys, in terms of phase composition, can be divided into three groups: single-phase with α structure, two-phase α+β and single-phase with β-Li structure [11]. They display very good plastic and even superplastic properties at relatively low temperatures and low deformation rates. The authors of this study demonstrated that the rolled two-phase Mg-Li α+β alloy manifested a superplasticity effect between 523 and 623 K [8]. New ultralight Mg-Li-Ca alloys are promising materials of the future. Due to their low density, good resistance, plasticity and biocompatibility, they can be used in medicine in biodegradable implants and surgical stitches [12]. Calcium in the chemical composition can result in reduction of density of Mg-Li alloys and improvement of strength through the formation of a second phase, as well as reduction of the ignition of molten magnesium and improvement of oxidation resistance [13]. The introduction of calcium into the Mg-Li alloy is expected to result in the formation of the compound Mg_2Ca, which will improve the strength of Mg-Li-Ca alloys [14-15]. The literature evidence suggests that, in addition to modifying the chemical composition, another way to improve the strength of Mg-Li-Ca alloys is to attempt plastic forming using unconventional SPD deformation methods. The use of SPD methods allows significant grain size reduction in metallic materials to the submicrometric scale [16-17]. The use of unconventional deformation methods for magnesium alloys results in their increased plasticity [18-19]. This method exploits the phenomenon of a change in the plastic deformation path [20-21]. The results obtained for conventional magnesium alloys confirmed the achievement of microstructure refinement and improved mechanical properties. The scientific literature on the implemented non-covalent plastic deformation processes (KoBo method) of magnesium alloys containing lithium and calcium in their chemical composition is still incomplete, so this article presents the results of a study to determine the effect of the parameters of the applied plastic deformation process on the changes in the microstructure and properties of the Mg-8Li-2Ca alloy.

2. Materials fabrication

The test material consisted of 40 mm diameter magnesium alloy ingots with an addition of lithium and calcium. The chemical composition of the Mg-8Li-2Ca alloy is presented in Table 1.

Table 1.
Chemical composition of the Mg-8Li-2Ca alloy [mas %]

Alloy	Li	Ca	Mg
Mg-Li-Ca	8	2	rest

In order to improve the mechanical properties of the tested Mg-8Li-2Ca alloy, Ca and Li additives were introduced into the chemical composition. The casting process of the investigated alloy was realized at the Department of Materials Engineering in the Department of Materials Technology. Graphite molds were used. Ingots with a diameter of 40 mm were obtained. The parameters of the casting process were used as for the Mg-4Li-1Ca alloy [3]. The Mg-8Li-2Ca alloy ingots were heat-treated at 300℃ for a period of 3h with cooling in air. The purpose of implementing the heat treatment process was to homogenize the microstructure of the Mg-8Li-2Ca alloy.

3. Methodology

3.1. KoBo extrusion

The Mg-8Li-2Ca alloy was subjected to extrusion using the KoBo method and conventional extrusion was performed for comparison. Both processes of plastic deformation were carried out at the AGH University of Science and Technology. Figure 1 shows the dependence of the extrusion force as a function of punch travel speed (0.1 mm/s, 0.5 mm/s) for the conventional and KoBo extrusion processes. The charge was heated to 350°C before the conventional extrusion process, after which the deformation process was conducted. The KoBo method was used in a cold extrusion process. The parameters of the implemented deformation process (KoBo method, conventional extrusion) are shown in Table 2.

Table 2.
The parameters of the implemented deformation process (KoBo method, conventional extrusion)

Process parameters:	
Torsion amplitude	$\alpha = \pm\ 8°$
Torsion frequency	$f =$ 5 Hz
Extrusion velocity	$v =$ 0.1, 0,5 mm/s
Diameter reduction	from 40 mm to 4 mm (rods)

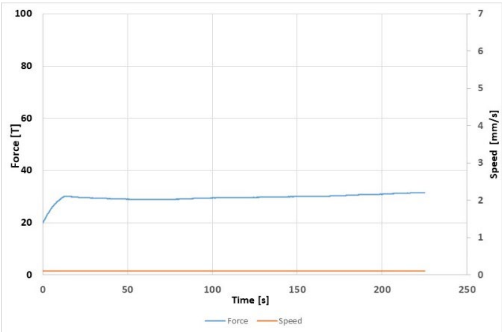
The material was intensively cooled with water at the press throat outlet

3.2. Microstructural characterization

The microstructure of the Mg-8Li-2Ca alloy was analysed in the initial state after deformation using an Olympus GX71 light microscope in bright field mode. Structures of both samples underwent quantitative analysis and the parameters of grain were determined with the use of program Metilo programme [22]. Stereological parameters were determined: average grain diameter [μm], shape factor, average surface area [μm^2] and coefficient of variation [%]. Figure 2 shows an example of the microstructure of the Mg-8Li-2Ca alloy with grain boundaries marked red, which were the basis for the determination of stereological parameters. In order to identify the phases in the studied alloy, a phase analysis was carried out for the alloy in its initial state. An X-ray phase analysis was conducted on an X-ray diffractometer, JeOL JdX-7S. An X-ray phase analysis was conducted on an X-ray diffractometer, JeOL JdX-7S, equipped with a copper anode tube ($\lambda cuK\alpha$ = 1.54178 Å) and supplied with a current of 20 ma and voltage of 40 kV, and a graphite monochromator.

3.3. Mechanical properties

Tensile tests were carried out on a ZWICK testing machine (Zwick Roell AG, Ulm, Germany) with a maximum force of 250 kN. Results were recorded on a computer using TestXpert 2 software (AG, Ulm, Germany). The tests were performed at ambient temperature (RT) with a constant strain rate of 1×10^{-4} s^{-1} and three repetitions for each sample. The microhardness test was performed using the Vickers method with a load of 0.2 kg (HV0.2) using the Zwick hardness tester. Measurements of HV0.2 microhardness were tester in the initial state and after plastic deformation processes. Six measurements each were taken on the test samples in the initial state and after extrusion using the Kobo method and after conventional extrusion. The results obtained were used to determine the average value.



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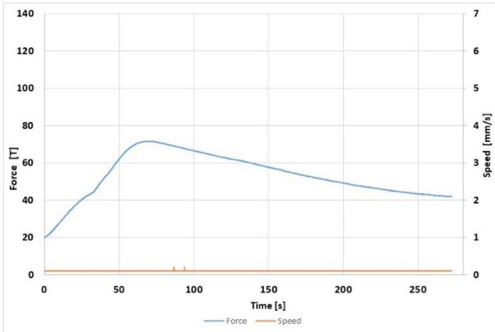

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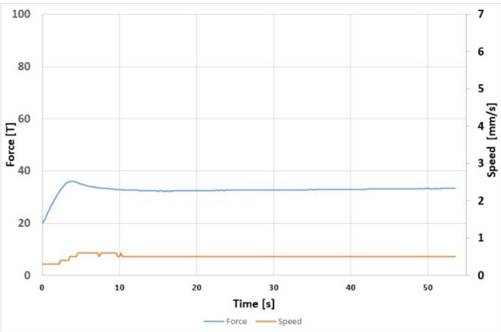

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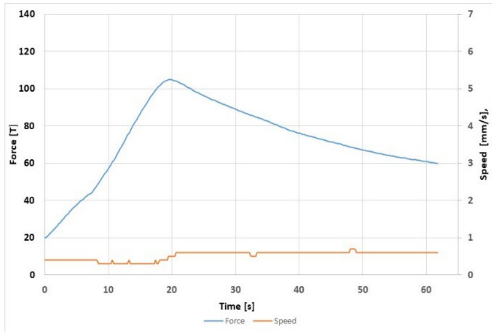
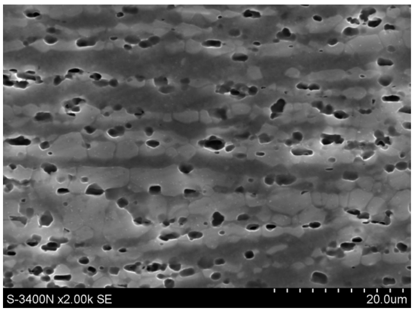
a)



0.5 mm/s

b)

Fig. 1. Dependence of extrusion force as a function of punch travel speed (0.1 mm/s, 0.5 mm/s) for: a) conventional extrusion, b) KoBo method

a) 

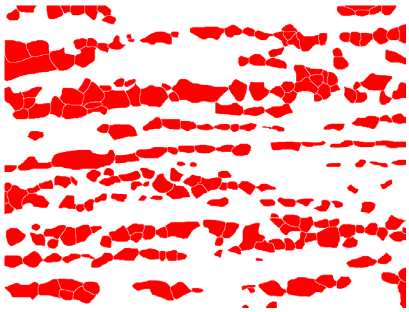
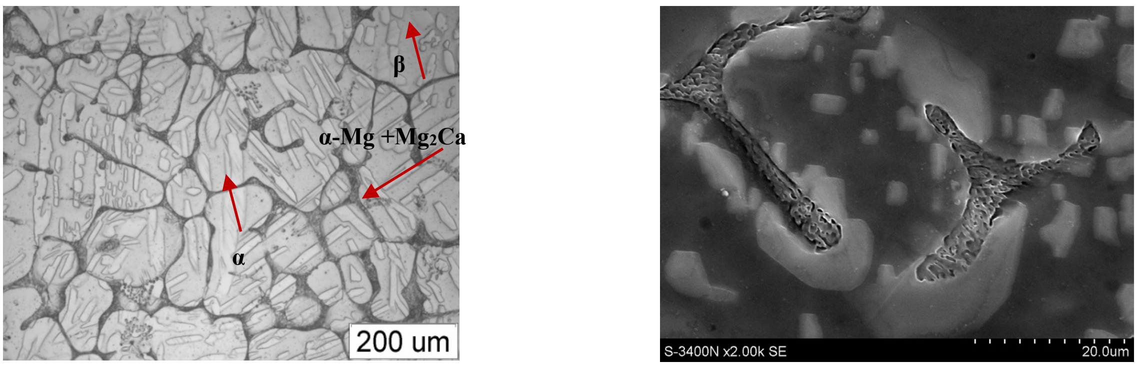
b) 

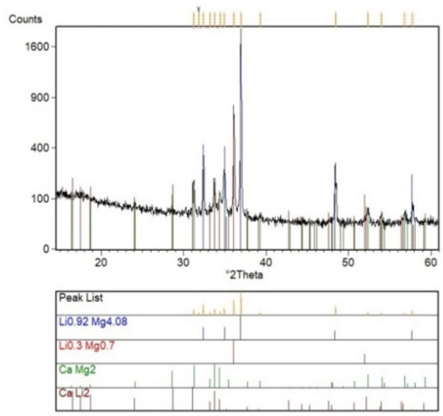
Fig. 2. a) Optical microstructure of alloy Mg-8Li-2Ca, b) example of a microstructure with detection

4. Results

Fig. 3a presents microstructure of alloy Mg-8Li-2Ca after the process of casting and heat treatment. The Mg-8Li-2Ca alloy is a biphasic α+β alloy according to the phase equilibrium system Mg-Li. In the microstructure of the studied Mg-8Li-2Ca alloy, the α phase was observed, probably having an elongated shape, distributed over the entire analyzed surface. Eutectics located at grain boundaries having a lamerall shape. Similar structural effects were observed for the Mg-4Li-1Ca alloy as presented in the paper [3]. Occurring singly or in clusters (Fig. 3a-b). The authors of the paper [6] for a magnesium alloy with a similar chemical composition, i.e. 9 Li wt% and 15 Al wt%, rest Mg, showed the presence of α+β phases in the alloy. In addition, XRD analysis conducted revealed the presence of lithium-rich phases (Li_2Ca, $Mg_{0.7}Li_{0.3}$). The presence of these Li-containing phases in the alloy improves ductility and deformability. The identification of phase composition after casting was conducted with the use of an X-ray phase analysis. An example view of the X-ray diffraction pattern is shown in Fig. 3b. Phase α-Mg (solid solution of lithium in magnesium) and phase Li_2Ca, $Mg_{0,7}Li_{0,3}$ were identified. The microstructure of the Mg-8Li-2Ca alloy after conventional and KoBo extrusion for 0.1 mm/s and 0.5 mm/s punch displacement is shown in Figure 4. The analysis of the microstructure revealed, a deformed microstructure and refinement due to the recrystallisation process (Fig. 4b-d). A deformed area was observed in the microstructure of the Mg-8Li-2Ca alloy after conventional deformation consistent with the direction of extrusion. The eutectics and precipitates found were refined and are present throughout the entire analysed surface of the alloy. The α-Mg phase has expanded and the boundaries of the new grains formed by the applied plastic deformation are visible inside (Fig. 4 a-c). Comparable changes were observed in the microstructure of the investigated alloy after conventional extrusion for a punch displacement of 0.1 mm/s, 0.5 mm/s (Fig. 4 a-c). The analysis of the microstructure after KoBo deformation revealed the deformed microstructure of the tested alloy. For a punch travel speed of 0.1 mm/s, similar effects of microstructure changes were found as after conventional extrusion. Undulating of α-Mg phase boundaries and areas of eutectic/precipitates placement at their boundaries were observed. For a speed of 0.5 mm/s, refinement of both phases was observed after KoBo deformation. The α-Mg and β phase have been deformed, which was not observed after conventional extrusion. The analysed eutectics are spherical in shape, located at grain boundaries and form a band structure (Fig. 4d). The results of quantitative analysis of the microstructure of the Mg-8Li-2Ca alloy after conventional extrusion and KoBo extrusion are shown in Table 3. A grain with an average equivalent diameter of about 2.52 μm was obtained after conventional extrusion, while the average equivalent diameter of the grain after KoBo deformation was 2.28 μm (Table 3). Fig. 5 shows histograms of the grain size distribution for the Mg-8Li-2Ca alloy after conventional and KoBo extrusion. After conventional extrusion, grains of 1-3 μm comprise the largest share of approximately 70%. The remaining 30% are made up of grains of varying sizes above 3 μm (Fig. 5a).

a)





b)

Fig. 3. a) Optical microstructure of alloy Mg-8Li-2Ca after casting, b) X-ray diffraction pattern after casting processes and heat treatment

Table 3.
Results of quantitative characterisation after deformation of the Mg-8Li-2Ca alloy, punch travel speeds 0.5 mm/s

Mg-8Li-2Ca	Average equivalent diameter of grains [µm]	Average surface area [μm^2]	Shape factor	Coefficient of variation [%]
after conventional extrusion	2,52	745	0,77	40
after KoBo method	2,28	872	0,79	44

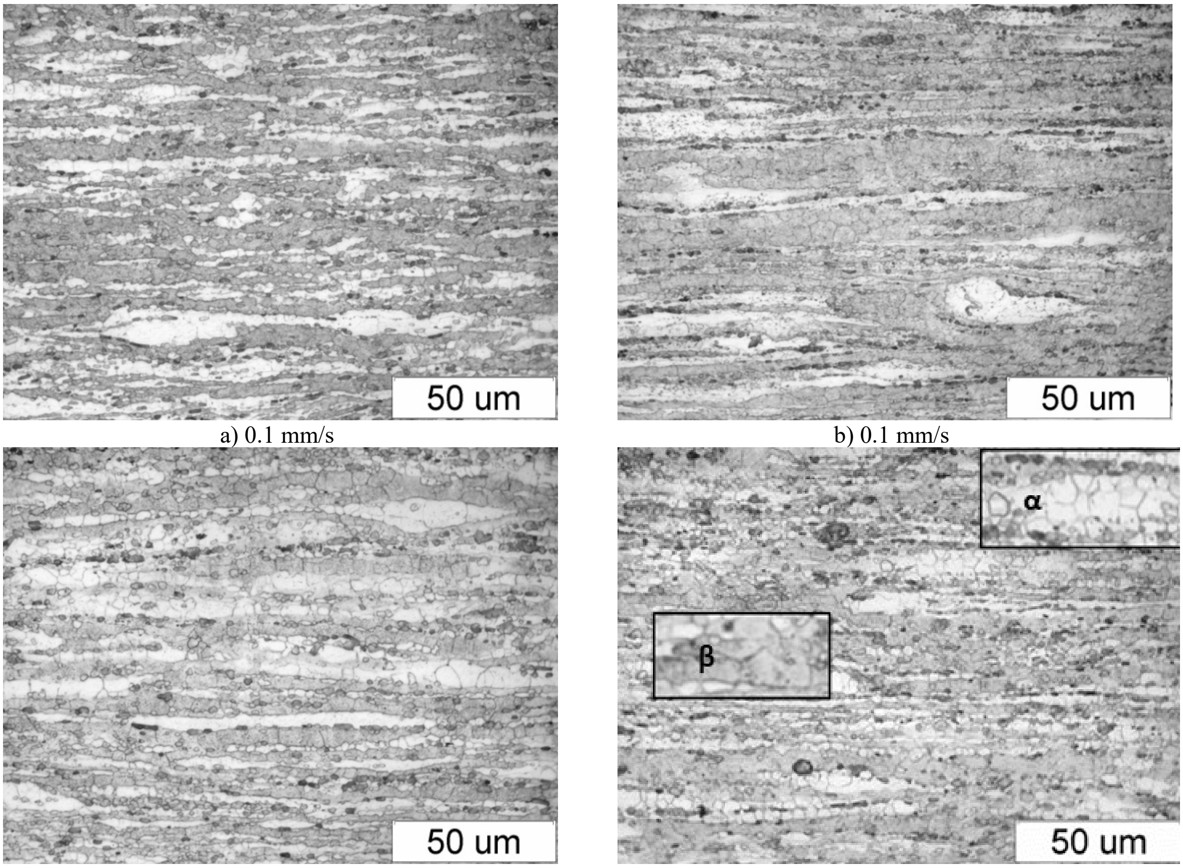
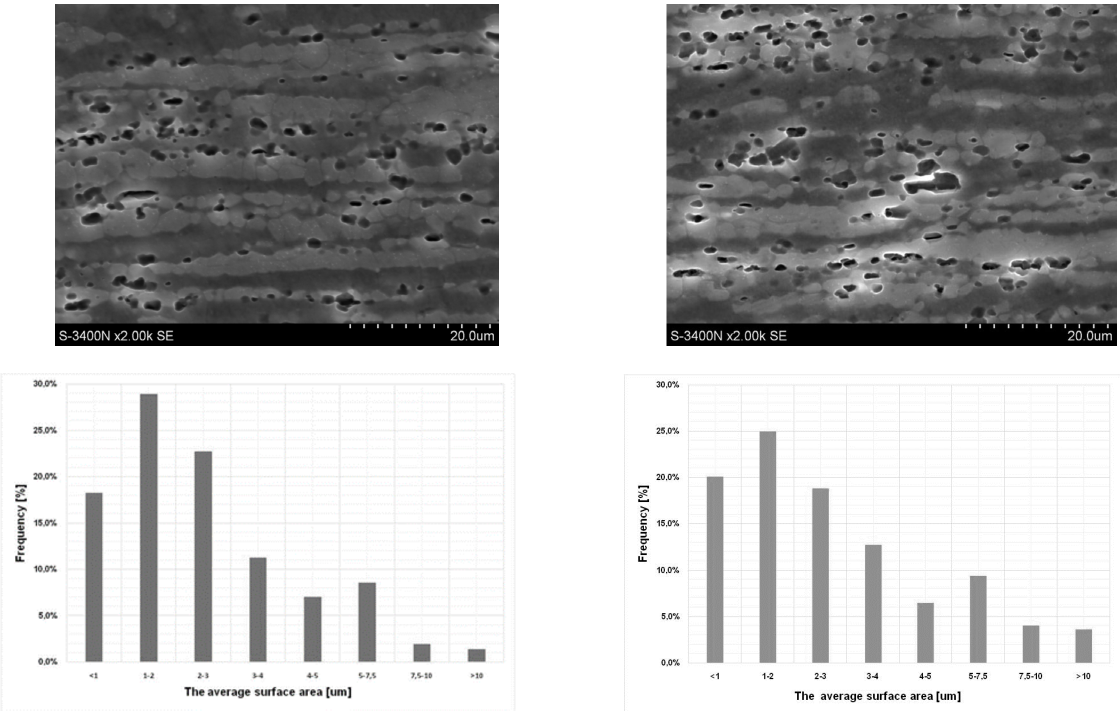


Fig. 4. Optical microstructure of alloy Mg-8Li-2Ca: a-c) conventional extrusion, b-d) KoBo method

After extrusion using the KoBo method, 1 to 3 µm grains constituted the largest share of 63%. 31% are grains from 4 µm to 10 µm. The smallest share of 6% is represented by grains up to 1 µm in size (Fig. 5b). A punch travel speed of 0.5 mm/s was chosen to assess the mechanical properties for both deformation methods used. The static tensile test was performed at room temperature. Table 4 and Figure 6 show the results of static tensile tests for the Mg-8Li-2Ca magnesium alloy after conventional extrusion and KoBo extrusion at room temperature. The rods, obtained at a punch travel speed of 0.5 mm/s, of 4 mm in diameter were selected to evaluate the mechanical properties for both deformation methods used. Better mechanical properties were obtained for rods deformed using the KoBo method. Lower Young's modulus values were observed in comparison with conventional extrusion. In addition, the KoBo method produced higher values for tensile strength and yield stress. After conventional extrusion, the microhardness was 60 HV0.2, while after KoBo extrusion it reached 69 HV0.2 (Table 4). In the initial state, a microhardness of 46 HV0.2 was obtained. This demonstrates the significant strengthening of the Mg-8Li-2Ca alloy.



a) b)

Fig. 5. Optical microstructure of alloy Mg-8Li-2Ca and grain size distributions: a) conventional extrusion, b) KoBo method

Table 4.
Results of mechanical properties after deformation of the Mg-8Li-2Ca alloy, punch travel speeds 0.5 mm/s

Alloy Mg-8Li-2Ca	conventional extrusion	extrusion KoBo method
R_m [MPa]	182	204
A [%]	4	9
$Rp_{0,2}$/Re	159	176
E [GPa]	42	39
HV0,2	60	69

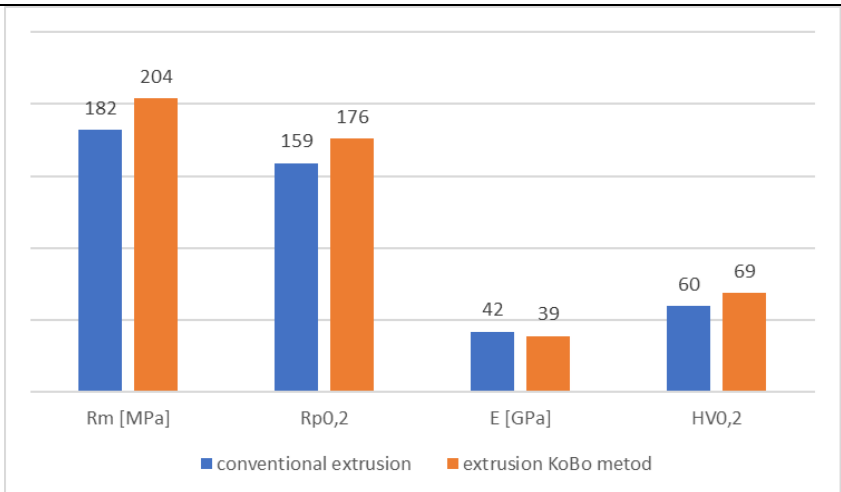


Fig. 6. Diagram of mechanical properties after deformation of the Mg-8Li-2Ca alloy, punch travel speeds 0.5 mm/s

5. Conclusions

The paper presents the results of a study on the effect of the plastic deformation parameters on the changes in the microstructure and properties of the alloy. A plastic deformation process was carried out using the KoBo method and, as a comparison, a conventional extrusion was performed with the same parameters. Microstructure analysis after conventional and KoBo extrusion revealed the presence of a recrystallised microstructure with eutectics forming a band structure. Greater microstructure refinement was demonstrated after KoBo deformation. The α-Mg and β phases were refined (Figure 5). An equivalent mean grain diameter of 2.52 μm was obtained, which is lower compared to conventional extrusion (2.28 μm). The mechanical properties were evaluated based on hardness measurements and static tensile test. In the case of the plastic properties of the tested alloy, an improvement was revealed after deformation using the KoBo method, where an elongation value of 9% was obtained. Microhardness in the initial state of the tested alloy was 46 HV0.2. After the application of deformation processes, the microhardness values changed. After conventional extrusion, an HV0.2 value of 60 was obtained, while after KoBo extrusion the value was 69 HV0.2. The increase in microhardness after KoBo extrusion may be attributed to the strengthening of the alloy due to the increase in the amount of grain boundaries. Moreover, the tested alloy may have become stronger as a result of the applied plastic deformation parameters, the presence of defects and the precipitates/phases present in the microstructure of the tested alloy. Based on the results obtained from the static tensile test, the Young's modulus value was 42 GPa after conventional extrusion and 39 GPa after KoBo extrusion. The application of the KoBo method as a deformation process for the Mg-8Li-2Ca alloy significantly altered the microstructure and properties. The presence of lithium in the chemical composition of the investigated alloy results in the formation of an α+β biphasic structure according to the Mg-Li system, which improves the properties of the investigated alloy. Obtaining a larger grain size distribution after deformation by the KoBo method probably contributed to the more favourable properties of the investigated alloy for the applied deformation parameters.

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