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CHARACTERISTICS OF THE MICROSTRUCTURE AND PROPERTIES OF Mg-Li MAGNESIUM ALLOY AFTER DEFORMATION BY THE KOBO METHOD

The article presents the results of tests of plastic shaping of magnesium alloy Mg-Li. Magnesium alloy for an extrusion process was obtained with the method of vacuum smelting and casting into graphite moulds. The materials for tests were slabs cast from magnesium alloys with symbols: Mg-4%Li-1%Ca (LX41). Before the process of deformation the castings were subject to homogenization. Conventional extrusion tests were conducted in a complex state of deformation (KoBo method). An assessment was performed of the influence of the deformation process parameters on the structure and properties of the tested alloy. Results of mechanical tests were presented both for static compression test in room temperature. On the basis of the achieved tests results, the susceptibility to plastic working for the Mg-4%Li-1%Ca alloy was determined. An analysis of the microstructure was conducted both in the initial condition and after plastic deformation with the use of light and scanning microscopy techniques. The applied deformation methods allowed the determination of the influence of process parameters on changes in the microstructure and properties of the Mg-4%Li-1%Ca alloy.

Keywords: Mg-Li alloys; metod SPD; static compression test; microstructure

1. Introduction

Magnesium and its alloys are among the lightest of all structural metals, significantly lighter than steel, titanium and aluminum alloys [1,2]. Therefore, hey have become potential engineering materials for the automobile and aeronautical industries because of their high strength-to-weight ratio and low density [3-4]. The interest in magnesium alloys with respect to their applications as structural elements in the aerospace industry goes back to the 50s of the last century . Mg-Li based alloys are the lightest metallic structural materials with a density of 1.35-1.65 g/cm^3. They can be progressively used in the aerospace and aircraft structures as well as in ultraweight communications systems in the future due to their lightweight and strength. The density of magnesium is approximately of that of aluminium, one quarter of zinc, and one fifth of steel [5,6]. Lithium addition to magnesium bears an important effect on the crystal structure, in particular the lattice parameters. They also possess many other advantages, such as high specific strength, high specific stiffness, etc. However, the usages of the Mg-Li alloy are often limited by the insufficient properties, of which the poor strength is the most obvious aspect. According to the Mg-Li phase diagram, the alloys exhibit two phase structures of α (hexagonal close packed (hcp) Mg-rich and β (body-centered cubic (bcc)) Li-rich phases at room temperature when 5-11% Li is added to magnesium. This two-phase structure has excellent deformability and extremely low density, but exhibits moderate mechanical strength and poor corrosion resistance. Above 10.3 wt.%, the Li microstructure in all Mg-Li alloys is composed of β (Li) phase. An in creasein the Li content causes a reduction in the lattice constant ratio (c/a = 1.624) of magnesium, as shown by Li et al. with the analysis of an Mg-xLi-3 Al-Zn alloy, where the axial ratio (c/a) could bereduced from 1.624 to 1.608 when the Li fraction increased from 1 wt.% to 5 wt.% [7-8].

This situation causes a reduction in critical resolved shear stresses (CRSSs) of slip systems and more slip systems being activated at ambient temperature, thus enhancing the Mg-Li deformation capacity in comparison with other Mg alloys [9]. Further additions of Li (more than 11%) can transform the hcp α-Mg solid solution into highly workable, body-centered cubic alloys. They are of great importance also for medical purposes. Therefore, it is important to investigate mechanical properties at different temperatures and to estimate the deformation mechanisms responsible for the deformation behaviour of Mg-Li alloys at elevated temperature. At present, the alloy which shows the biggest potential is the LX41alloy (Mg-4%Li-1%Ca) which is

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a promising material for the use in biodegradable implants on a magnesium matrix [9-12]. Few scientific studies show that the use of complex thermoplastic treatment operations and cold forming under conditions of change of strain path will enable the improvement of mechanical properties, due to the grain size reduction to sub-microcrystalline or nanometric sizes [13]. The deformation method under conditions of cyclic change of the strain path is called the KoBo method. It offers the possibility of deforming a material by applying a cyclically varying action as an addition to the deformation process (drawing, forging, rolling, extrusion). The advantages of the KoBo extrusion process include an efficient and energy-saving production of products, the ability to produce products with complex geometries, even from materials that are difficult to deform, and reduced wear of working tools. In addition, the considerable size reduction of the structure to nano- and ultrafine grain results in favourable mechanical properties. All processes carried out using the KoBo method allow a significant reduction in energy consumption during the process by reducing the force required to deform the metal, sometimes by more than 50% compared to conventional deformation processes. In addition, they enable obtaining higher plastic deformation with lower deformation forces, as well as higher material throughput [13-14].

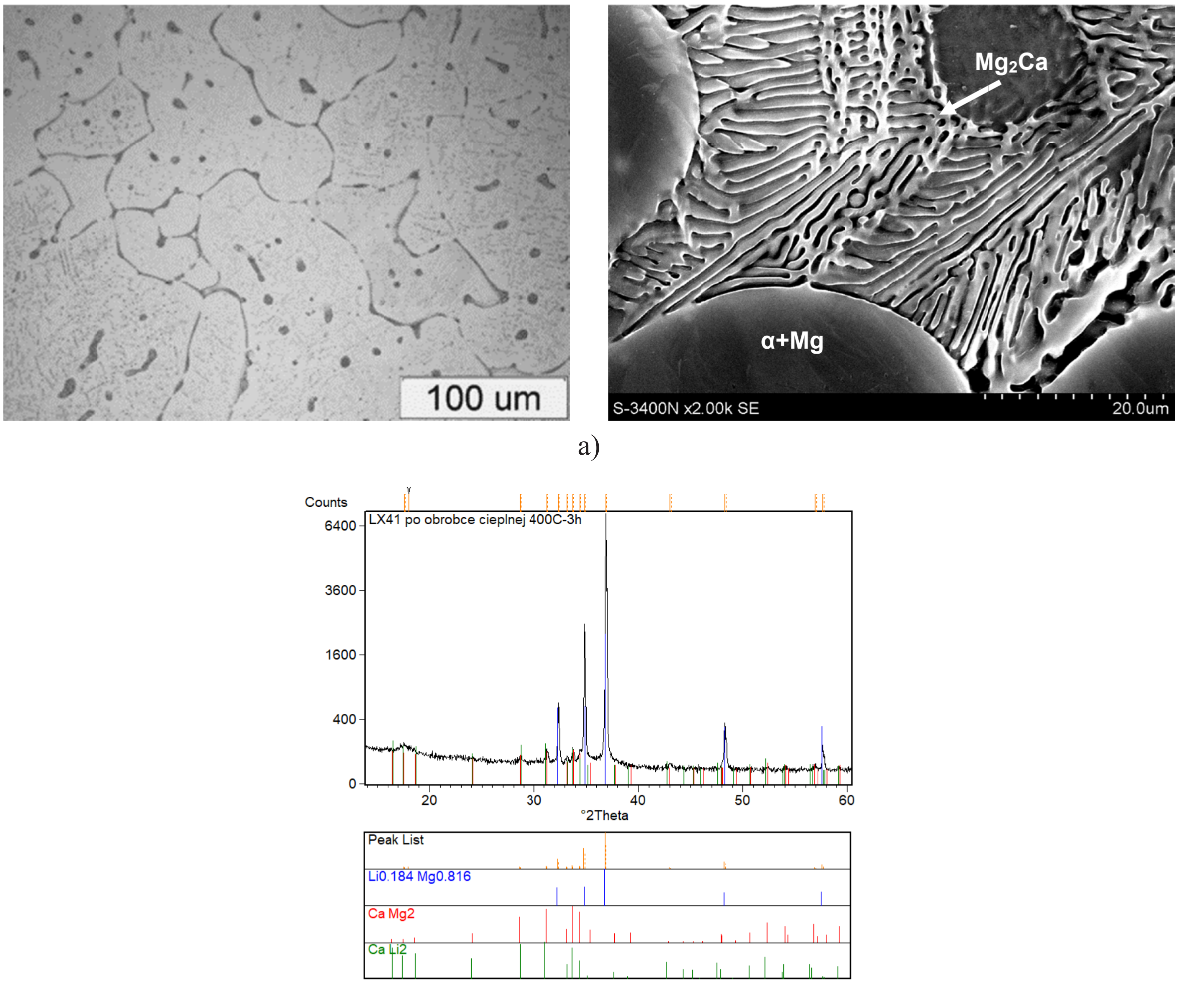
Therefore, this paper presents the results of a study which aimed to determine the influence of parameters of plastic deformation processes carried out using the KoBo method on changes in the microstructure and properties of the Mg-4%Li-1%Ca alloy. The results of mechanical properties determined in a static tensile test at room temperature are presented. Based on the results obtained, the plastic formability of the Mg-4%Li-1%Ca alloy was determined. Conventional extrusion and KoBo extrusion tests were carried out.

2. Materials and methods

The test material consisted of 40 mm diameter magnesium alloy ingots with the addition of lithium (4 wt-%) and calcium (1 wt-%). The Mg-4%Li-1%Ca alloy was melted in a Balzers' VSG 02 single chamber vacuum induction furnace. The casting process of the Mg-Li-Ca alloy was carried out in graphite moulds due to the absence of reaction of the liquid alloy with the mould material. The ingots underwent extrusion to the diameter of 40 mm [16]. Next, they were subject to annealing for 3h at a temperature of 400°C and cooling in air. The Mg-4%Li-1%Ca alloy was subjected to coextrusion 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. 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 following parameters were used for the KoBo deformation process: reverse die torsion at a frequency of 5 Hz, die oscillation angle of ±8°, punch travel speed of 0.5 mm/s, and diameter reduction from 40 mm to 4 mm. The compact was cooled freely on the press run-out table. The plastic deformation processes resulted in rods with a diameter of 4 mm. The examination of the microstructure of the Mg-4%Li-1%Ca alloy was performed on the cross-section parallel to the axis of the sample. The samples were included in a conducting material and etched in a solution intended for etching magnesium alloys, containing: 5% nital (nitric acid, ethyl alcohol). The microstructure of the Mg-4%Li-1% alloy was analysed in the initial state after deformation using an Olympus GX71 light microscope in bright field mode. Additionally to the analysis of the microstructure, quantitative and qualitative analyses were conducted with the use of Metilo program [15]. The grain size was measured using a surface method based on images recorded on a light microscope. The analyses of the microstructures after casting and extrusion processes were conducted with the use of light and scanning microscopy techniques. 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 20mA and voltage of 40 kV, and a graphite monochromator. The registration was conducted using the stepwise method with steps of 0.05° and a counting rate of 5 seconds in range from 10 to 100° 2θ. Tests were conducted on solid samples after casting and heat treatment processes. The studies of the microstructure were supplemented by observation using a scanning transmission electron microscope (STEM), Hitachi HD-2300 A (Hitachi Science & Technology, Tokyo, Japan), equipped with a Field Emission Gun (FEG) operated at 200 kV, which was used for microstructure characterization on longitudinal sections of the extruded billet. For microstructure examination, we used transmitted electron (TE) imaging. For STEM investigations, foils with a diameter of 3.0 mm after electrolytic thinning were used. Tests of mechanical properties of the obtained rods were conducted using a tension machine, Zwick/100 (Zwick Roell AG, Ulm, Germany), and the rods were stretched in room temperature. Measurements of HV0.2 microhardness were performed using a Zwick hardness tester in the initial state and after plastic deformation processes.

3. Results

Fig. 1 presents an example microstructure of alloy Mg-4%Li-1%Ca after the process of casting and heat treatment. In order to improve the ductility of the Mg-4%Li-1%Ca alloy, a heat treatment was carried out at 400ºC for 3h, followed by cooling with the furnace. Analysis of the microstructure after casting and heat treatment for the Mg-4%Li-1%Ca alloy revealed the occurrence of a coarse-grained structure and the presence of eutectics (α + Mg, Mg_2Ca) located at grain boundaries with an elongated or spherical shape. The identification of phase composition after casting and homogenisation process was conducted with the use of an X-ray phase analysis. An example view of the X-ray diffraction pattern is shown in Fig. 1b. Phase α-Mg (solid solution of lithium in magnesium) and phase Li_2Ca were identified.



a)

b)

Fig. 1. a) Microstructure of alloy Mg-4%Li-1%Ca after casting and heat treatment, b) X-ray diffraction pattern after casting and heat treatment processes

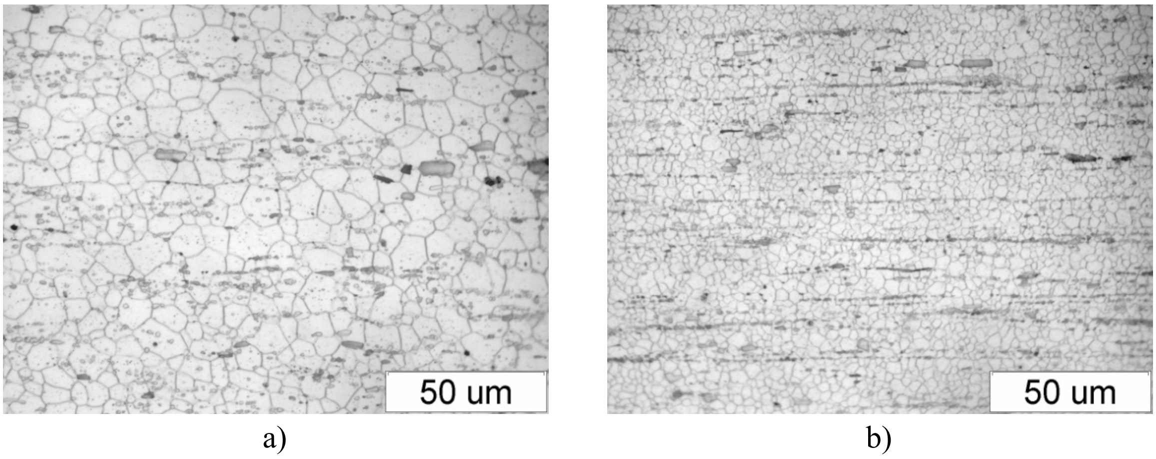
The microstructure of the Mg-4%Li-1%Ca alloy after extrusion is shown in Fig. 2. Analysis of the microstructure after extrusion with the KoBo method has shown the presence of a recrystallised structure. On grain boundaries, eutectics forming a banded structure are present. After comparison of the microstructure obtained after the KoBo process with the microstructure after classic extrusion of alloy Mg-4%Li-1%Ca, it can be concluded that there is a significant fineness of structure present after applying the KoBo method.The results of quantitative analysis of the microstructure of the Mg-4%Li-1%Ca alloy after conventional extrusion and KoBo extrusion are shown in TABLE 1. Stereological parameters were determined: average grain diameter, shape factor and surface area. Grain with the average equivalent diameter of about 3.40 µm was obtained, which was smaller in comparison with the grain obtained after classic extrusion, where the average grain equivalent diameter was 4.70 µm.

Fig. 3 shows histograms of the grain size distribution for the Mg-4%Li-1%Ca alloy after conventional and KoBo extrusion. After conventional extrusion, a predominance of grain with a diameter of 47-90 µm was found, representing about 51% of the total analysed area. Approximately 21% are grains with a diameter of more than 140 µm (Fig. 3a). The histogram of the grain size distribution after KoBo extrusion is dominated by grains with a diameter of 24-47 µm, representing about 66%

TABLE 1

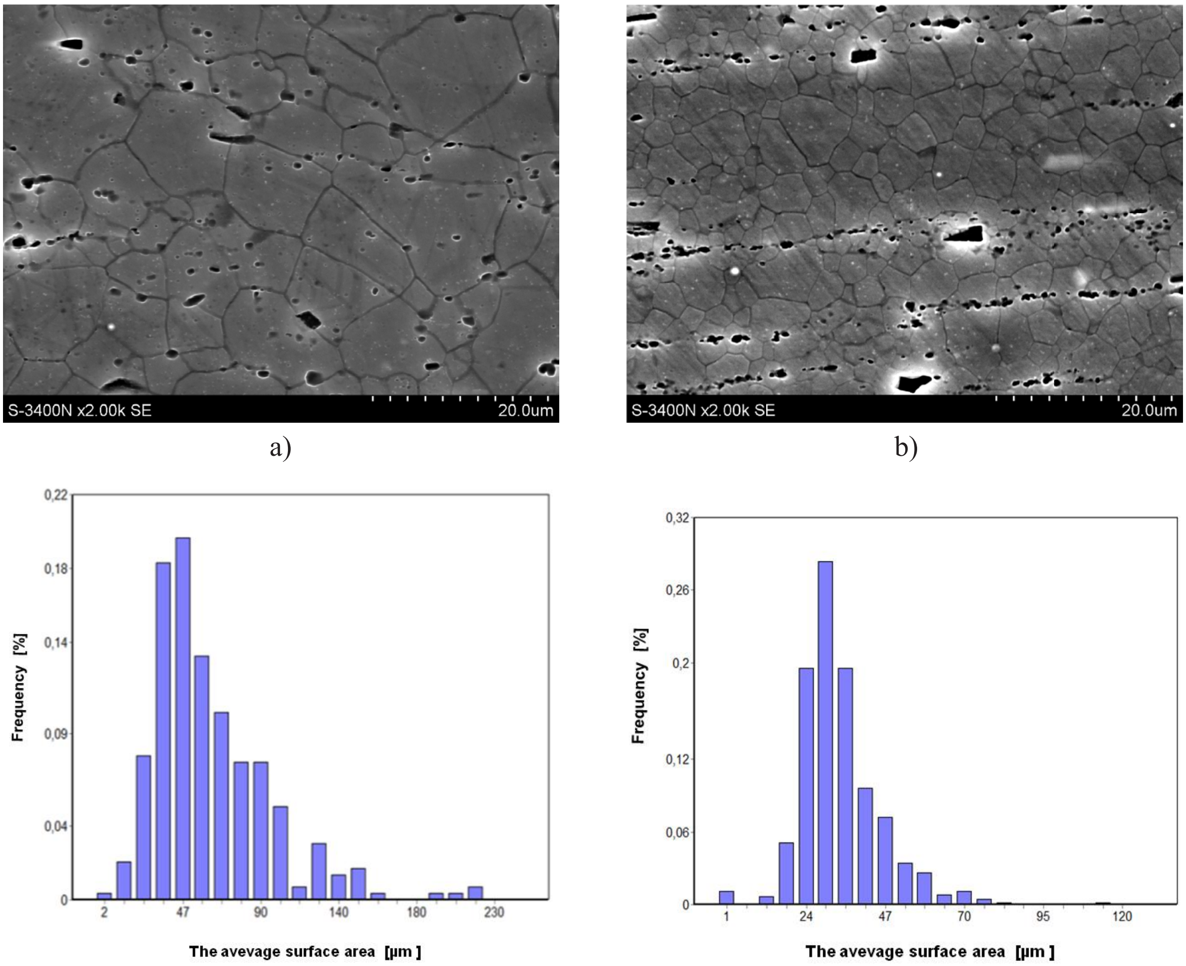
Results of quantitative characterization after deformation of the Mg-4%Li-1%Ca alloy

Alloy Mg-4%Li-1%Ca	**Average equivalent grain diameter [µm]**	**Average surface area [µm^2]**	**Shape factor**	**Coefficient of variation [%]**
after hot extrusion rods with diameter of ϕ 4 mm	4.70	3811	0.18	137
after cold extrusion with KoBo method rods with diameter of ϕ 4 mm	3.40	8408	0.09	170



a) b)

Fig. 2. Microstructure of alloy Mg-4%Li-1%Ca after deformation: a) classic extrusion, b) KoBo method



a) b)

c) d)

Fig. 3. Microstructure of alloy Mg-4%Li-1%Ca after deformation: a) classic extrusion, b) KoBo method, c-d) grain size distributions

of the total analysed area. Grains ranging from 47 µm to 70 µm account for an approx. 9% share (Fig. 3b). The obtained values of the shape factor and the coefficient of variation indicate that the microstructure after extrusion with the KoBo method shows features of a non-uniform microstructure, and the shape of the analysed grains is close to equiaxial.

Analysis of the substructure was also conducted with the use of scanning-transmission microscopy (Fig. 4) and it revealed the presence of areas of free dislocation (Fig. 4). Recrystallised areas with the presence of new grains and the grain/subgrain of recrystallisation were observed.

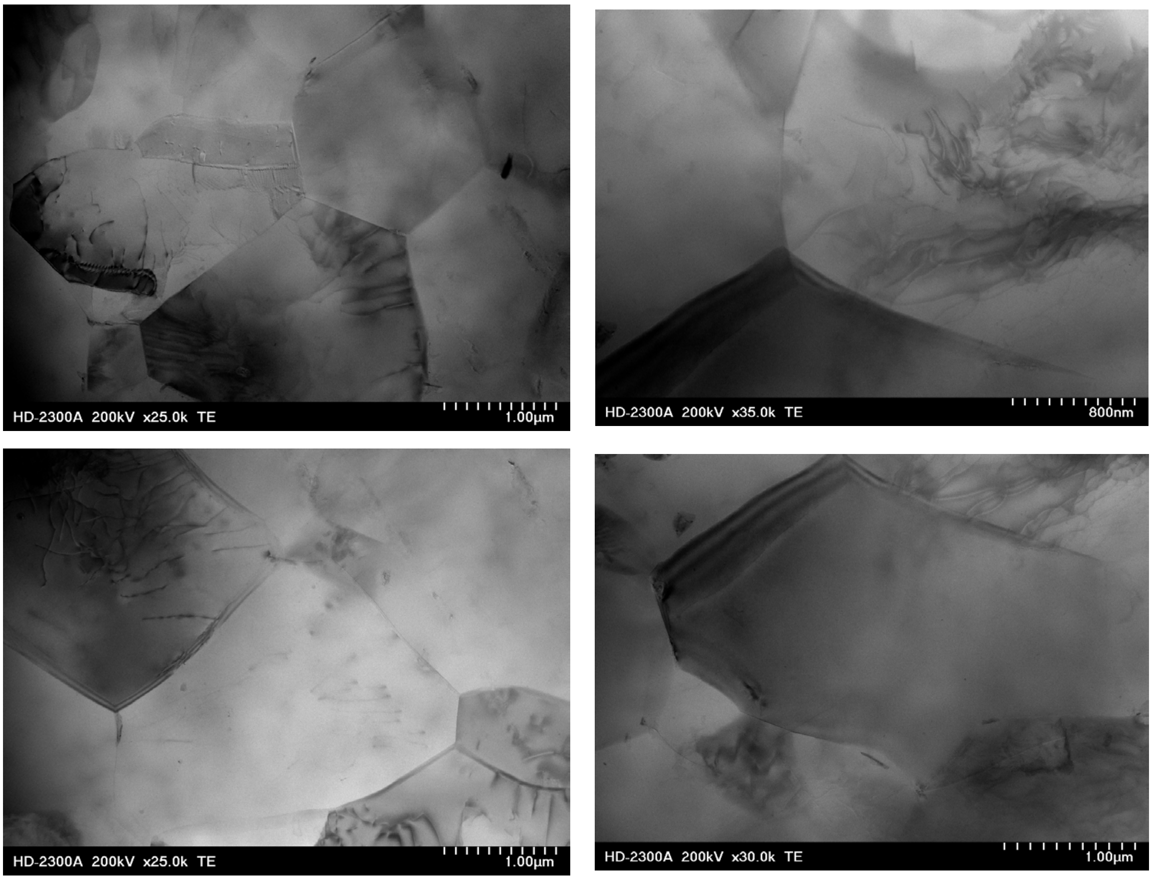


Fig. 4. Microstructure of Mg-4%Li-1%Ca alloy after the extrusion process with the use of KoBo method (STEM)

Table 2 shows the results of static tensile tests for the Mg-4%Li-1%Ca magnesium alloy after conventional extrusion and KoBo extrusion at room temperature.

More favourable mechanical properties were obtained after KoBo extrusion, where lower values of Young's modulus were obtained compared to conventional extrusion. Also, higher elongation values were obtained for the analysed strain rate of 0.5 mm/s. The elongation value was 8%. A decrease in tensile strength was observed compared to conventional extrusion.

Fig. 5 shows the collective results of the measurement of microhardness HV0.2 in the initial state and after the applied plastic deformation processes.

In the initial state, a microhardness of 46 HV0.2 was obtained. After the extrusion process using two deformation methods: conventional and KoBo extrusion, different microhardness values were obtained. After conventional extrusion, the microhardness was 56 HV0.2, while after extrusion with the KoBo method, it was 76 HV0.2.

TABEL 2

Test results of mechanical properties after conventional and KoBo method for the Mg-4%Li-1%Ca alloy at room temperature

Mg-4%Li-1%Ca alloy	R_m [MPa]	$R_{p0,2}$ [MPa]	E [GPa]	A[%]
after extrusion process	214	155	44	4
after cold extrusion with KoBo method	285	206	48	8

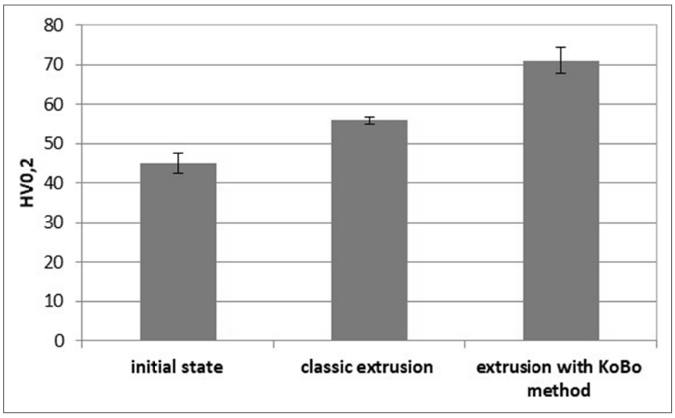


Fig. 5. Summary of results of microhardness measurements of the Mg-4%Li-1%Ca alloy in its initial state and after extrusion processes

5. Conclusions

The paper presents the results of tests for the Mg-4%Li-1%Ca alloy. Based on the literature analysis, it can be concluded that the disadvantages of magnesium alloys are their low ductility and difficulties in plastic forming, therefore, solutions are being sought to improve the ductility of this group of materials. The research undertaken is aimed at determining the influence of the deformation method on changes in the microstructure and properties of the Mg-4%Li-1%Ca alloy. The first stage of the study was to analyse the microstructure of the Mg-4%Li-1%Ca alloy after the casting process. The next stage was a heat treatment process, whose main objective was to homogenise the microstructure of the alloy and increase its plastic formability. Analysis of the microstructure of the Mg-4%Li-1%Ca alloy after casting and homogenisation showed the presence of a residual dendritic structure and the occurrence of eutectics with a lamellar structure located mainly at grain boundaries. The observed eutectics are a mixture of phases: a-Mg, Mg_2Ca (Fig. 1b). X-ray phase analysis showed the presence of a Li_2Ca phase in the microstructure. After casting and homogenisation, the Mg-4%Li-1%Ca alloy was subjected to conventional extrusion and extrusion using the KoBo method. Rods of 4 mm in diameter were obtained. The analysis of the microstructure after conventional and KoBo extrusion revealed a recrystallized structure with the presence of eutectics forming a banded structure (Fig. 2). A significant effect of KoBo extrusion parameters on grain fineness was demonstrated. An average equivalent grain diameter of 3.40 μm was obtained, which is lower compared to conventional extrusion (4.70 μm). The final stage of the study was the assessment of the mechanical properties. Measurements of microhardness HV0.2 and a static tensile test at room temperature were performed. HV0.2 microhardness measurements were carried out for the Mg-4%Li-1%Ca alloy in the initial state (after casting and homogenisation) and microhardness of 46 HV0.2 was obtained. After applying deformation processes, the microhardness values varied. After conventional extrusion, a HV0.2 value of 56 was obtained. For KoBo extrusion, the microhardness value was above 70 HV0.2. The increase in microhardness after KoBo extrusion may be due to the refinement of the structure and more grain boundaries. Additionally, the tested alloy may strengthen as a result of the given plastic deformation and the impact of defects, as well as the presence of precipitates/phases occurring in the microstructure of the tested alloy (Fig. 5).

From the results obtained in the static tensile test it was found that after conventional extrusion, the value of Young's modulus was equal to 44 GPa and the value of elongation was about 4%, while more favourable properties were obtained after extrusion with the KoBo method, with the 8% elongation and Young's modulus of 48 GPa (TABLE 2).

Based on the research and analysis of the results, the following conclusions have been formulated:

1. In the microstructure of the Mg-4%Li-1%Ca alloy after casting and heat treatment, the presence of a residual dendritic structure was found and eutectics with a lamellar structure in the interdendritic spaces. The X-ray phase analysis showed the occurrence of the following phases in the microstructure of the Mg-4%Li-1%Ca alloy: $Mg_{0,818}Li_{0,184,}$ Li_2Ca, Mg_2Ca.
2. After conventional extrusion and KoBo extrusion of the Mg-4%Li-1%Ca alloy, a fine-grained structure was obtained as a result of the recrystallization process.
3. When KoBo extrusion was used, the highest refinement of the microstructure was obtained for a punch travel speed of 0.5 mm/s. The average equivalent diameter of the grains was 3.40 μm and was smaller compared to conventional extrusion, where grain with an average equivalent diameter of 4.70 μm was obtained.
4. After extrusion of the Mg-4%Li-1%Ca alloy with the KoBo method, more favourable mechanical properties were obtained compared to conventional extrusion.
5. The presented results prove that the applied plastic deformation methods (conventional extrusion, KoBo method) can also be used to prepare a Mg-4%Li-1%Ca alloy charge for further plastic working operations.

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