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INFLUENCE OF NUMBER OF ECAP PASSES ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AZ31 MAGNESIUM ALLOY

WPŁYW ILOŚCI PRZEJŚĆ W PROCESIE ECAP NA MIKROSTRUKTURĘ I WŁAŚCIWOŚCI MECHANICZNE STOPU MAGNEZU AZ31

The aim of this work was to investigate the influence of the number of equal channel angular pressing (ECAP) passes on the microstructure and mechanical properties of AZ31 magnesium alloy. The microstructure after two and four passes of ECAP at 423 and 523 K was investigated by means of optical and transmission electron microscopy. The mechanical properties were carried out using Vickers microhardness measurements and compression test. The grain refinement in AZ31 alloy was obtained using ECAP routes down to 1,5 μm at 423 K. Processes of dynamic recrystallization during ECAP were observed. It was found that a gradual decrease of grain size occurs with the increasing of number of ECAP passes. The grain refinement increases mechanical properties at ambient temperature, such as Vickers microhardness and compression strength proportionally to $d^{-0.5}$.
Keywords: ECAP magnesium alloy, AZ31, microstructure, mechanical properties

Celem niniejszej pracy było określenie wpływu ilości przejść przez kanał kątowy na mikrostrukturę i właściwości mechaniczne stopu magnezu AZ31. Obserwacje mikrostruktury, po dwóch i czterech przejściach przez kanał kątowy w temperaturze 423 i 523 K, przeprowadzono za pomocą mikroskopii optycznej i transmisyjnej elektronowej. Właściwości mechaniczne wyznaczono w próbie mikrotwardości metodą Vickersa oraz w próbie ściskania. W wyniku procesu czterokrotnego przeciskania przez kanał kątowy stopu AZ31 w temperaturze 423 K uzyskano bardzo drobne ziarno o wielkości około 1,5 μm. W mikrostrukturze badanego stopu po procesie ECAP obserwowano zmiany świadczące o zachodzących procesach rekrystalizacji dynamicznej. Stopniowe zmniejszanie się wielkości ziarna następuje wraz ze wzrostem ilości przejść materiału przez kanał kątowy. Zmniejszenie wielkości ziarna w procesie ECAP powoduje zwiększenie właściwości mechanicznych stopu AZ31 w temperaturze pokojowej odwrotnie proporcjonalnie do $\sqrt{d}$.

1. Introduction

The issue of energy and environment has become a commanding priority. Therefore, there is a widespread emphasis on high strength and low density materials in many applications. Magnesium alloys are the lightest metallic structural materials progressively used in transportation, electronics and aerospace industry due to their many advantages compared with steels, cast irons or aluminium alloys [1,2]. However, poor formability and limited ductility of magnesium alloys at ambient temperature, attributed to hexagonal close-packed structure with two basic types of slip systems: $(0001)\langle 11\bar{2}0\rangle$ and $(10\bar{1}0)\langle 11\bar{2}0\rangle$, limits their application for industrial purposes.

The grain refinement is one of the method to improve the mechanical properties of magnesium alloys. It is known that Severe Plastic Deformation (SPD) techniques are successful in refining the grain structure of metallic materials and equal channel angular pressing (ECAP) is one of the most effective SPD techniques to achieve ultra-fine grains of relatively large volumes of metals and alloys [3,4]. A positive effect of ECAP on steels, aluminum, copper and various magnesium alloys with improved ductility, strength and superplasticity was reported [5,6,7,8,9,10]. The activation of non-basal slip system in magnesium alloys requires elevated temperatures due to their hexagonal close-packed structure, therefore the most of ECAP processes applied to magnesium alloy were performed at or above 473 K to

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avoid crack formation [11,12]. Severe plastic deformation by ECAP at such temperatures induces recrystallization processes and grain growth [13,14].

In order to avoid these unfavorable processes causing the grain growth, the pressing temperature during ECAP was carefully chosen as low as possible to avoid crack formation, but not to induce grain growth.

The present investigation was undertaken to examine the microstructure and mechanical properties of AZ31 magnesium alloy after two and four passes of ECAP at 423 K and 523 K.

2. Experimental procedure

As cast ingots of magnesium alloy AZ31 (Mg-3%Al-1%Zn-0.3Mn, wt.%) was supplied by Jiangxi Royal, Ltd. The billets from as-received with initial thickness of 20 mm were rolled at 623 K down to the thickness of 10 mm. The samples were cut into the rod with dimensions 10×10×50 mm and then subjected to ECAP passes according B_C route – the rotation of sample by 90° in the same direction between passes. As prepared samples were pressed using ECAP die at 423 K and 523 K. The ECAP die is schematically shown in Fig. 1a.

The ECAP die was constructed of H13 tool steel with a square-shaped channel of 10.2×10.2 mm and designed as two parts. Die was surrounded by electric resistance heater. The temperature was controlled using thermocouple placed near the channel wall with accuracy to ±5°C. An internal angle ϕ between two parts of channel equals 90° and the corner angle (ψ) of 0° with a small radius of approximately 2 mm. The equivalent strain ε_N, after N passes, can be expressed by following relationship [4]:

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\cos ec\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right] \quad (1)$$

The specimens were pressed using Instron modified universal testing machine with maximum applicable load of 100 kN at the pressing speed 0.1 mm/s. For each pass, ECAP die was heated to proper temperature and a sample was held in die channel for 10 minutes to reach the temperature stabilization. Before inserting in ECAP die channel, a sample was coated with graphite paste (Molydal Multigraph) to assure lubrication during ECAP process. The specimens for microstructural examinations and mechanical testing were cut from the middle of sample along the plane Y – parallel to the extrusion direction (Fig. 1b).

The microstructures after ECAP were observed by means of optical microscopy (OM) and transmission electron microscopy (TEM). The specimens for OM examination were prepared by mechanical grinding and polishing. The polished surface was etched in solution of 1ml acetic acid, 4.2 g picric acid, 10 ml H_2O and 75 ml ethanol. The grain size was measured by the method of secants on optical microscopy images. For TEM study, thin foils were electropolished in solution of 80% perchloric acid and 20% ethanol at 223 K. The prepared TEM specimens were observed using Tecnai G2 F20 (200 kV).

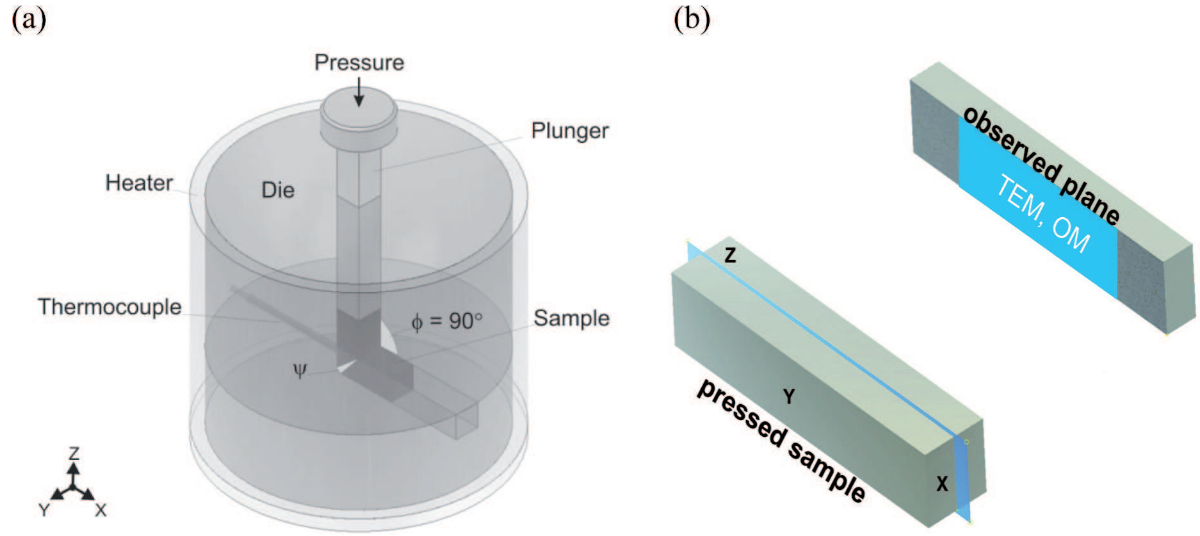

Fig. 1. Schematic illustration of the facility for ECAP (a) and the pressed sample (b) with Y-plane parallel to ECAP direction chosen for the micorstructural observation

The microhardness measurements and compressive tests were performed to estimate the effect of ECAP on mechanical properties of AZ31 magnesium alloy. Vickers microhardness measurements (HV0.1) of investigated samples were determined at the load of 100 g for 10 s. The compressive tests of specimens after ECAP were carried out at the strain rate $1.8\times10^{-3}s^{-1}$. Compression specimens were machined of cylindrical shape with diameter of 4 mm and height of 6 mm from the billets before and after ECAP.

3. Experimental results and discussion

3.1. Microstructure before and after ECAP

The microstructure of as-cast AZ31 alloy (in the as received condition) is characterized by inhomogeneous grain size distribution with a very large grain sizes as shown in Fig. 2a. The initial average grain size for the as-cast alloy was about 250 μm. After hot rolling at 623K, the microstructure is heterogeneous and consists of elongated grains in the direction of rolling with average grain size estimated at about 15 μm (Fig. 2b).

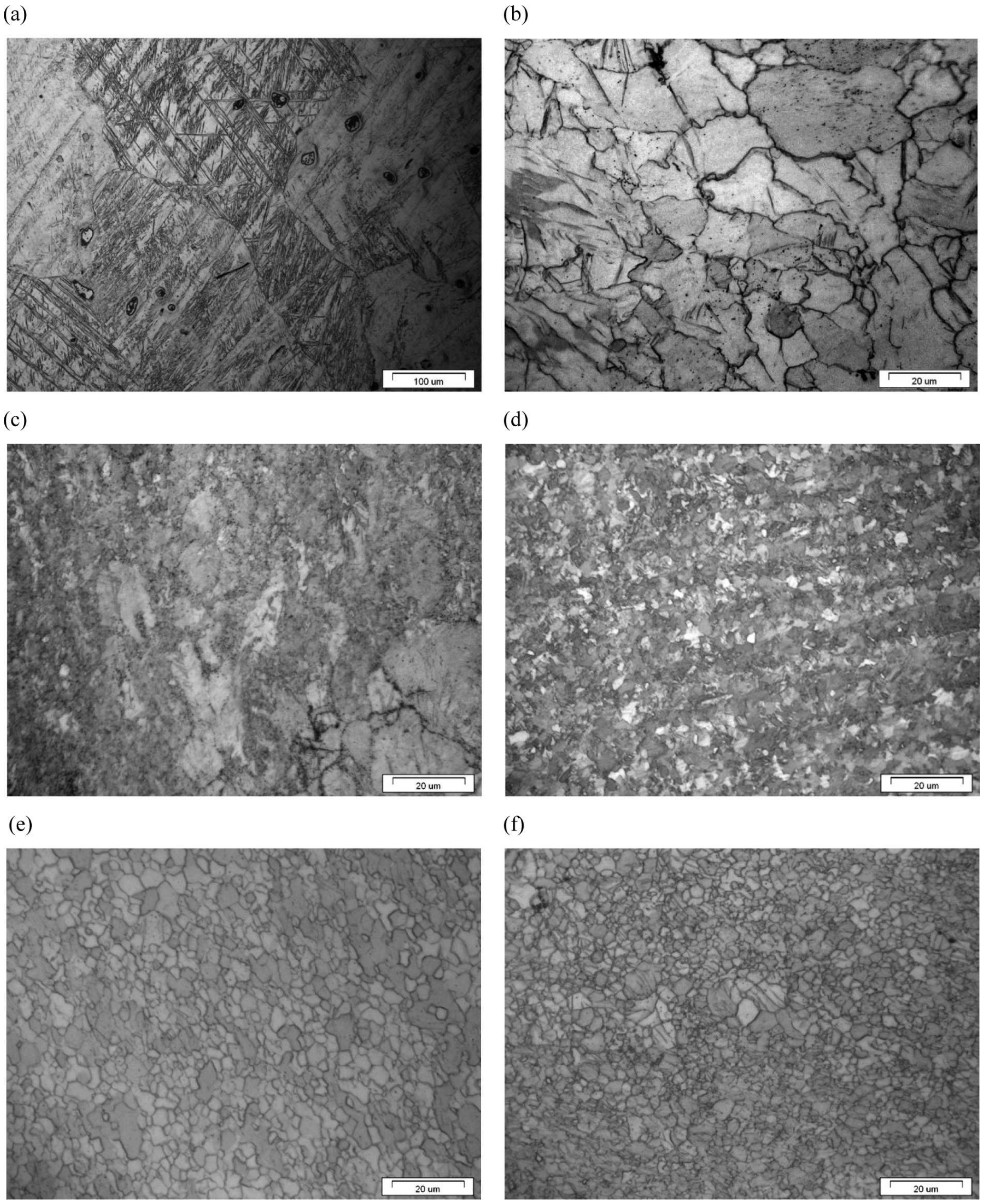

Fig. 2. Grain microstructure of the AZ31 alloy processed by ECAP: (a) as cast, (b) hot rolled at 523 K, (c) after 2 passes and (d) after 4 passes at 423 K, (e) after 2 passes and (f) after 4 passes at 523 K

The grain structure changes of AZ31 magnesium alloy after two and four passes of ECAP at 423 K and 523 K are presented in Fig. 2c-f.

The plastic shear deformation by the ECAP causes accumulation of a large plastic strain and an increase of structural defects. After two passes at 423 K, the grains characterize as-called "bimodal" distribution (Fig. 2c) and the microstructure consists of very fine grains (< 1μm), as well as coarse grains (> 5μm), which was observed also in earlier studies [4,15]. Bimodal distribution of grains characterizes deformed microstructure and is possibly caused by limited slip systems in magnesium, therefore only favourable oriented grains were deformed and refined as first during ECAP process and areas of larger less deformed grains were left in microstructure. However, after 4 passes at 423 K brighter grains appear, which possibly result from dynamic recrystallization processes (Fig. 2d). The grains structure becomes homogenized with an average grain size of 1,5 μm.

The grain size changes after two and four passes of ECAP at 423 and 523 K of the AZ31 alloy are summarized in Table 1.

TABLE 1
The average grain size in μm after two and four passes of ECAP of the AZ31 alloy

ECAP temperature	Number of passes			
(K)	0 (as cast)	0 (hot rolled)	2	4
423	250	16	2,2	1,5
523	250	16	6,2	3,2

After two passes at 523 K (Fig. 2e), reasonably uniform distribution of grains with an average grain size of 6,2 μm, and after 4 passes (Fig. 2f) fully homogenized grains structure with an average grain size of 3,2 μm was obtained. It was found that fully homogenized grains structure can be achieve after 4 to 8 of passes in ECAP experiments at 473 K [4,16].

Homogenous microstructure after two passes at 523 K suggest that dynamic recrystallization occurs during ECAP, which is manifested by a nucleation and growth of a new grains, because there is no evidence of large grains and the fraction of fine grains successively increases with the subsequent passes. An earlier examinations demonstrated that fine grains were nucleated in the shear zone during ECAP and these grain grow at the pressing temperature 473 K to introduce homogenous microstructure [18]. On the other hand, in another study this grain growth facilitates the homogenization of the structure in AZ31 alloy and additionally reduces the effectiveness of the ECAP processing in grain refinement [8].

TEM micrographs of two and four passes of ECAP at 423 K of AZ31 alloy are shown in Fig. 3. The presence of a heavy deformed microstructure with high dislocation density was observed in most of grains.

After two passes, coarse and fine grains of ECAP were observed during TEM study (Fig. 3a). The dislocations are visible as heterogeneously distributed in a coarse and fine grains. The formation of subgrain structure especially in coarse grains and formation of subgrain boundaries (visible as diffused, composed of dislocations) was observed. After four passes, the microstructure consisted of fine-grains and better developed subgrain boundaries (Fig. 3b).

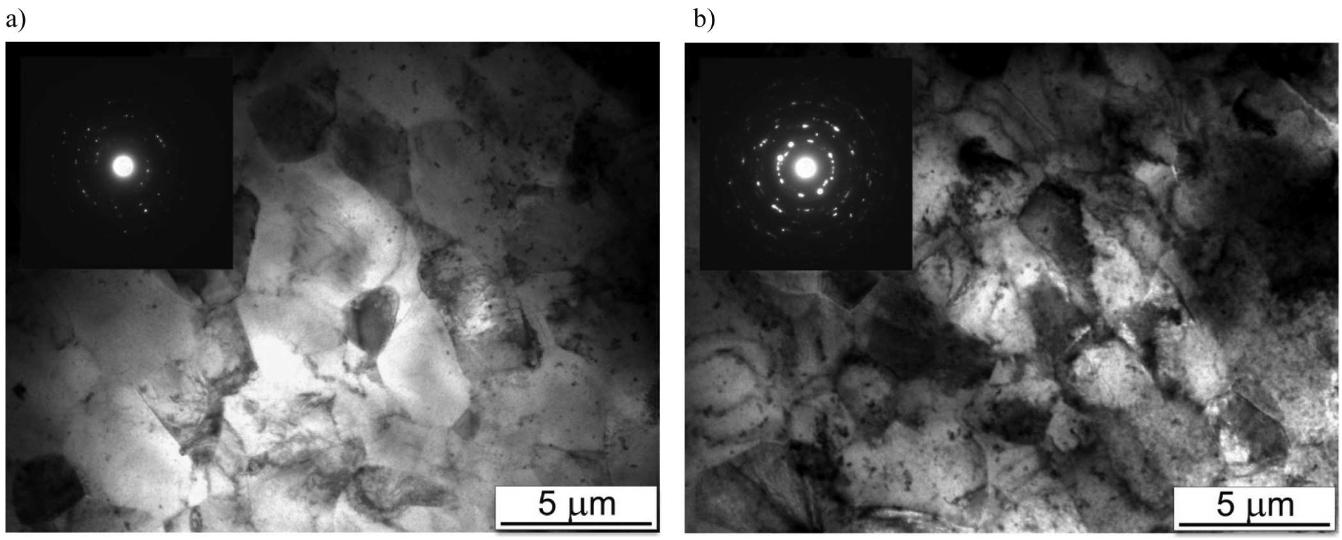

Fig. 3. TEM micrographs of microstructure of the AZ31 alloy after ECAP at 423 K: (a) after 2 passes and (b) 4 passes

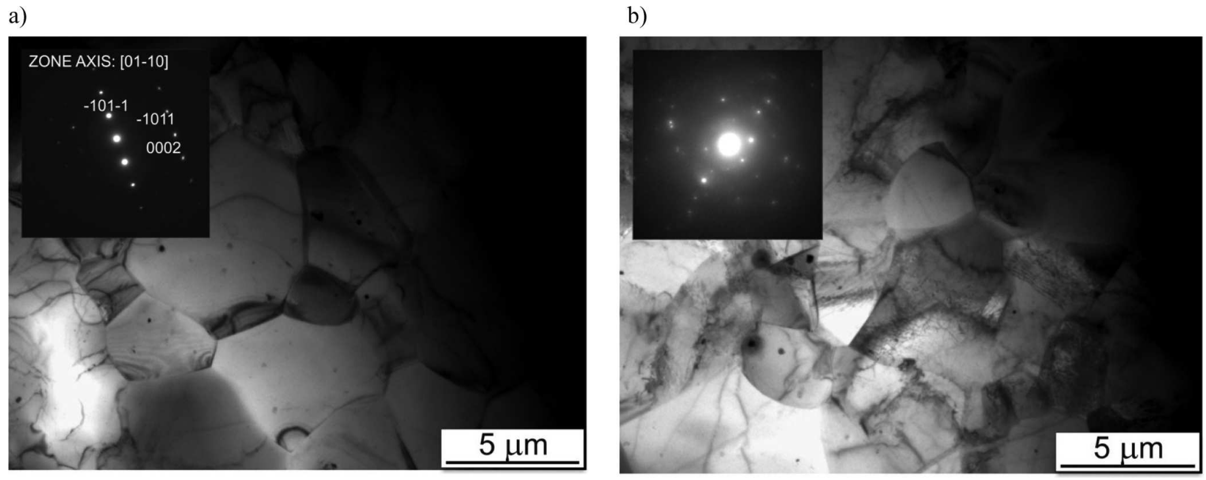

Fig. 4. TEM micrographs of microstructure of the AZ31 alloy after ECAP at 523 K: (a) after 2 passes and (b) 4 passes

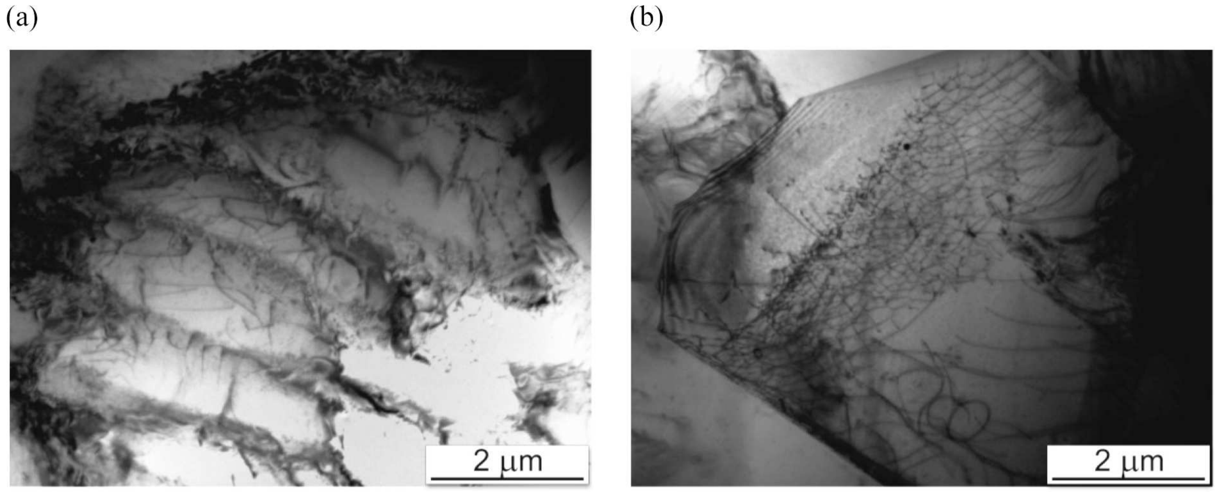

Fig. 5. TEM micrographs of microstructure of the AZ31 alloy after 4 passes of ECAP: (a) at 423 K, (b) at 523 K

More homogeneous structure was formed after two and four passes of ECAP of AZ31 alloy at 523 K (Fig. 4). The higher temperature promotes dynamic recrystallization processes during strain accumulation, therefore well-defined grain boundaries and a low dislocation density within grains were observed. The occurrence of dislocation substructure only in a few grains implies occurrence dynamic recovery.

The TEM examination showed that dynamic recovery and recrystallization take place in the AZ31 during ECAP in each pass. TEM micrograph in Fig. 5a shows the well developed subgrain boundaries, built from dislocations arranged in nets at subgrain boundaries after four passes at 523 K. The subgrain boundaries can change into high angle grain boundaries what lead to the grain refinement with the strain increase. The dislocation networks and rearrangements of dislocation formation of subgrain boundaries after ECAP at 523 K was shown in Fig. 5b.

The grain refinement mechanism of magnesium alloys during ECAP, proposed by Su at al. [16], was explained as a combination of mechanical shearing and subsequent continuous recovery, recrystallization and growth of grains and subgrain cells. In the present work, a heterogeneous structure of dislocations was observed. After two ECAP routes, the dislocations organize into walls and cells. In the next ECAP passes, the dislocation arrays and sub-boundaries are formed. The energy stored during plastic deformation by ECAP induces dynamic recrystallization particularly at sites with a high dislocation density.

Possibly with further ECAP passes, the dislocations are accumulated in coarse grains and consequently induce dynamic recrystallization in these regions. The fraction of fine grains increases with increasing ECAP pass number and at some stage the microstructure becomes homogenous and new recrystallized grains fill whole volume of alloy. An almost fully recrystallized and homogeneous microstructure in AZ31 alloy was observed after four passes of ECAP at 523 K.

3.2. Mechanical properties of the AZ31 alloy processed by ECAP

The values obtained by Vickers microhardness tests of AZ31 alloy after ECAP at 423 and 523 K are plotted against $d^{-0.5}$ (Fig. 6). It shows consistency with the Hall-Petch relationship and confirms that strengthening effect initiated by ECAP of AZ31 is a direct result of grain refinement. This trend for magnesium alloys (as well as for aluminum alloys) deformed by ECAP was also confirmed in other investigations [15,16,17]. The grain refinement of AZ31 alloy proceeded by ECAP is more effective at lower deformation temperature. The Vickers microhardness value after 2 passes of ECAP at 423 K is higher than after 4 passes at 523 K.

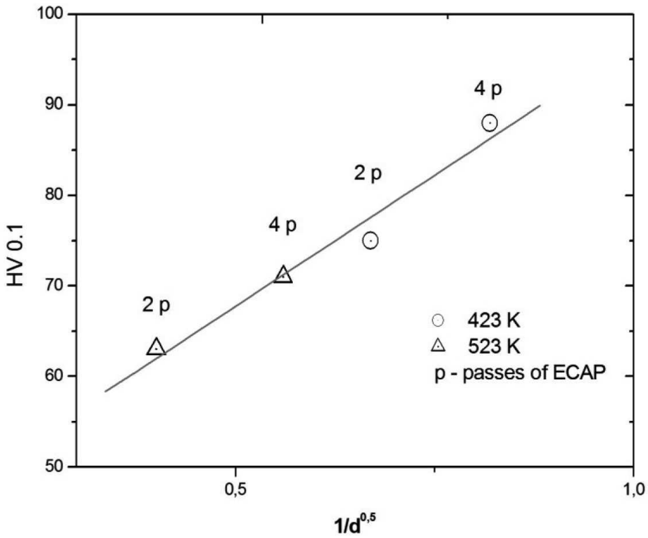

Fig. 6. Hall-Petch relationship for the two and four passes of ECAP at 423 K and 523 K of the AZ31 alloy

Mechanical properties of AZ31 alloy after ECAP were also investigated in the compression tests at ambient temperature. The deformation curves after 2 and 4 passes (2p and 4p) of ECAP at 423 and 523 K were plotted as the true stress versus the true strain (Fig. 7).

It is evident that there is a considerable increase in the strength after 2-4 passes of ECAP at both process temperature. The ultimate compressive strength and compressive yield strength of AZ31 alloy increase with increasing numbers of passes through the ECAP die. For the as cast state of examined samples, the compressive yield strength was about 75 MPa, which increased to about 130 and 150 MPa after 2 and 4 passes at 523 K, and increased to about 175 and 190 MPa after 2 and 4 passes at 423 K, respectively. It can be concluded that equal channel angular pressing provides an effective procedure for improving the mechanical properties of AZ31 alloy at ambient temperature.

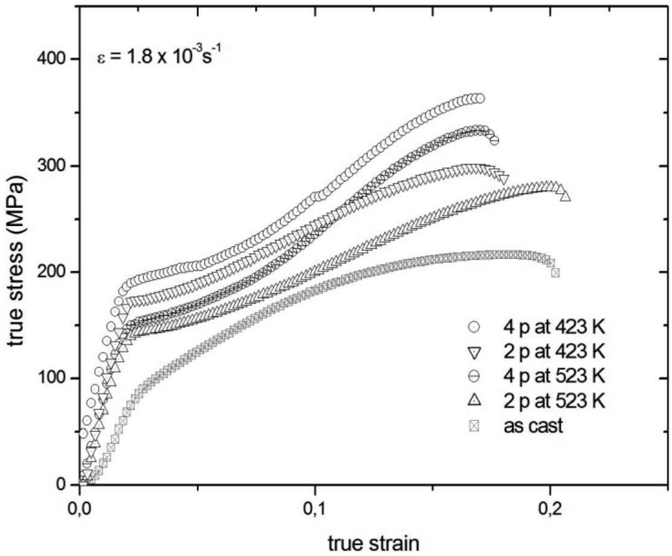

Fig. 7. Compression true stress versus true strain at room temperature of the AZ31 alloy after ECAP

4. Conclusions

The grain refinement in AZ31 magnesium alloy was carried out using the ECAP processing at 423 K and 523 K. The grain size was successfully reduced after 4 passes down to 1.5 μm at 423 K, while at 523 K down to 3.2 μm.

The microstructure of AZ31 after 2 and 4 passes of ECAP at 523 K consist of homogenous grains, whereas after 2 passes at 423 K the formation of deformation induced bimodal grain structure was present. The larger grain size at 523 K is due to higher activity of dynamic recrystallization processes which are manifested by the dislocation rearrangements in cellular structures and formation of subgrain boundaries.

The Hall-Petch relationship based on the Vickers microhardness was confirmed for the ECAP deformed samples and the highest obtained compressive strength was about 360 MPa ant the yield strength about 190 MPa.

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