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Abstract: Hydrostatic cyclic expansion extrusion (HCEE) process at elevated temperatures is proposed as a method for processing less deformable materials such as magnesium and for producing long ultrafine-grained rods. In the HCEE process at elevated temperatures, high-pressure molten linear low-density polyethylene (LLDPE) was used as a fluid to eliminate frictional forces. To study the capability of the process, AM60 magnesium rods were processed and the properties were investigated. The mechanical properties were found to improve significantly after the HCEE process. The yield and ultimate strengths increased from initial values of 138 and 221 MPa to 212 and 317 MPa, respectively. Moreover, the elongation was enhanced due to the refined grains and the existence of high hydrostatic pressure. Furthermore, the microhardness was increased from HV 55.0 to HV 72.5. The microstructural analysis revealed that ultrafine-grained structure could be produced by the HCEE process. Moreover, the size of the particles decreased, and these particles thoroughly scattered between the grains. Finite element analysis showed that the HCEE was independent of the length of the sample, which makes the process suitable for industrial applications.

Keywords: high-pressure fluid; elevated temperature; severe plastic deformation; hydrostatic cyclic expansion extrusion; mechanical properties; magnesium alloy

1. Introduction

Magnesium and its alloys are highly regarded due to their characteristics of low-density, excellent physical properties, and biocompatibility. Magnesium is not only used in the aerospace, electronics, and automotive industries but also in medical applications. However, as Akbaripanah *et al.* [1] have shown, as-cast magnesium has low ductility and poor strain hardening ability because of its hexagonal crystalline structure with a limited number of slip systems at room temperature. Improving its ductility and other mechanical properties is necessary for the development of various applications such as vascular stents, which were developed by Amani *et al.* [2]. Its mechanical properties can be improved by improving its microstructure, which is achieved by the severe plastic deformation (SPD) methods. Xia *et al.* [3] processed AZ31 alloy sheets via differential speed rolling and reached finer grains and higher elongation. Rahmatabadi *et al.* [4] demonstrated that the mechanical properties of Al/Cu/Mg metal sheets processed by SPD were very improved compared with those of coarse-grained sheets. Valiev *et al.* [5] claim that the strength-to-mass ratio of ultrafine-grained metals is much higher than that of the coarser-grain variants of the metals, and in some cases, by over 2–5 times. Ultrafine-grained metals also have more outstanding properties such as a homogeneous microstructure and high-angle grain boundaries, which has been shown by Islamgaliev *et al.* [6] in the processing of AM60 magnesium by equal-channel angular pressing (ECAP). Furthermore, after processing of titanium by hydrostatic extrusion, Pachla *et al.* [7] showed that the existence of a high hydrostatic pressure along with severe shear strain in the SPD techniques causes a high density of crystal defects, especially dislocations in the crystalline network. On the other hand, as Bridgman [8] has claimed, the presence of the hydrostatic pressure prevents crack formation during the SPD process, and this enables the application of severe deformation to brittle materials. However, there are some limitations in the length of the produced samples, which is a drawback in the implementation of the SPD methods on an industrial scale [9]. To overcome this drawback, Raab *et al.* [10] produced an unlimited length of ultrafine-grained rods by combining the conform process with ECAP. However, the drawback of the ECAP process as regards the non-uniform strain distribution along the radial diameter of

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the sample was not solved. Utsunomiya *et al.* [11] introduced another method called conshearing for the production of long ultrafine-grained metals, and they processed aluminum strips. However, producing samples with a homogenous structure remains a challenge for this method. Eskandarzadeh *et al.* [12] invented the process of continuous high-pressure torsion (HPT) as an industrially developed method for the production of long nanostructured rods. However, producing homogeneous samples is a limitation of the HPT method. Moreover, as already mentioned, the hydrostatic pressure, which is a positive factor in the processing of brittle metals and plays a crucial role in plastic deformation processes, is low in the aforementioned methods. Therefore, using a method that reduces all these problems is necessary. Among different SPD methods, cyclic extrusion compression (CEC) has several capabilities, including the ability to form the samples for several desired passes without removing them from dies and more hydrostatic pressure, which can make this method an industrial technique for producing ultrafine-grained metals. However, due to the nature of the CEC method, there is a need for a backpressure system, and the application of this pressure requires precision and expensive equipment. For this reason, Pardis *et al.* [13] invented cyclic expansion extrusion (CEE), which features a change in the design of the CEC process. In the CEE process, along with the benefits of the CEC process, the need for a backpressure system is eliminated. However, in the conventional CEE process, long specimens cannot be produced due to the high frictional forces between the die and the sample, which prevents the process from being performed. Samadpour *et al.* [14–15] invented the method of hydrostatic cyclic expansion extrusion (HCEE) in 2018. This process enables the production of long ultrafine-grained rods and also features the advantages of hydrostatic extrusion [16], which include high hydrostatic pressure, prevention of an increase in the temperature of the deformation zone, dynamic recrystallization, and high strain rates. The schematic of the HCEE process is shown in Fig. 1. As shown in this figure, the fluid covers the workpiece, prevents direct contact between the die and the sample, and eliminates friction between them. However, in the deformation region, the sample is directly in contact with the die; this area is similar to the case of the CEE method. The sample is severely deformed when it passes through this section, and its microstructure is changed.

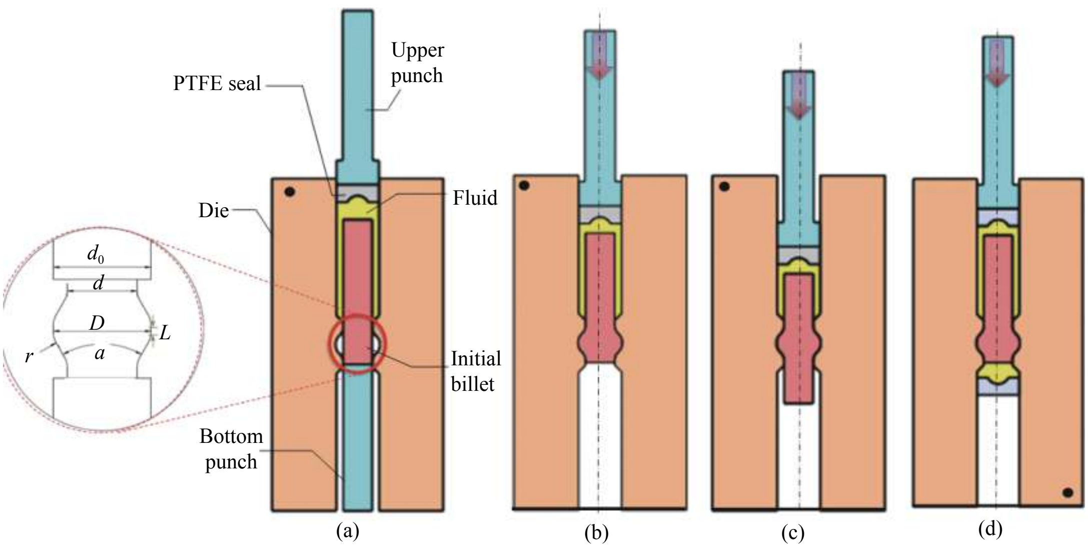


Fig. 1. Schematic of the hydrostatic cyclic expansion extrusion (HCEE) process: (a) the initial state; (b) the expansion state; (c) the extruding state (performing the first pass); (d) the second pass. D = 14 mm, d = 10 mm, d_0 = 12 mm, L = 1 mm, r = 3 mm, and α = 90°.

The equivalent plastic strain ($\bar{\varepsilon}$) is calculated for each cycle of the HCEE [15]:

$$\bar{\varepsilon} = 4\ln D/d \quad (1)$$

where D is the diameter of the expanding region, and d is the diameter of the extrusion region. By considering the die parameters used in this study, the total accumulated strain for each pass of the HCEE process is calculated as 1.34.

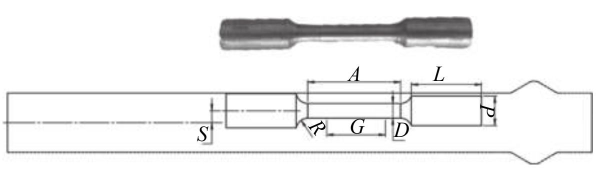
In the previous study [15], long ductile aluminum 1050 rods became ultrafine-grained by the HCEE process at room temperature, and the detailed principle of the process is presented. However, the capability of HCEE to process brittle metals needs further experimental evidence. In the present study, the capability of the HCEE to produce ultrafine-grained materials from initially brittle metals, magnesium alloy, was investigated. The HCEE at elevated temperatures was demonstrated to produce long ultrafine-grained AM60 magnesium rods for the first time, and the microstructural evolution and mechanical properties were investigated. In addition, finite element (FE) analysis was employed to study the deformation behavior and the required load and strain distribution.

2. Experimental and FE analysis procedure

In this study, AM60 magnesium alloy was processed by the HCEE method at elevated temperatures, and its characteristics were investigated. The outer diameter of the rod was 10 mm, and its length was 100 mm. This length-to-diameter ratio can never be processed by regular CEC or CEE method since the pressing punch may buckle and yield. A die was fabricated from H13 steel, which was hardened by heat treatment up to HRC 55. According to the analysis, this die can withstand a pressure of ~1500 MPa and is suitable for processing of different metals. Due to the limitation in the usage of oils at a high temperature of 300°C, molten linear low-density polyethylene (LLDPE) was used as a high temperature fluid in the die [17]. The LLDPE must be poured into the die cavity immediately after heating the die to melt rapidly and fill up the gaps. To seal the contact areas of the die components, pure copper was used as the sealant. A hydraulic pressing machine with a ram speed of 5 mm/min was used to carry out the process. The process was conducted at a temperature of 300°C. To increase the temperature of the workpiece and the die to the desired extents, an electric belt heater was used around the die.

In this research, a tensile test was conducted to examine the mechanical properties of the sample before and after the process at room temperature. Dimensions of the test specimen were determined according to ASTM E8, where the gage length and diameter were 10 mm and 2.5 mm, respectively. The exact position and dimension of the tensile sample are shown in Fig. 2. The tensile test specimen was cut along the longitudinal direction near the outer surfaces of HCEE-processed (HCEEed) rods. The test was conducted by an Instron tensile machine at a strain rate of 0.5 mm/min.

Furthermore, for hardness measurement, the samples before and after the HCEE process were prepared; the hardness test after mounting and polishing was carried out using a Vickers microhardness machine under a force of 0.5 N, which was applied for a period of 20 s.



S	*d*	*L*	*A*	*R*	*G*	*D*
2	5	12	16	2	10	2.5

Fig. 2. Exact position and specification of the tensile test specimen in HCEEed sample according to ASTM E8 (mm).

To study the microstructural variation after the HCEE, the cross sections of the samples were cut and then mounted. To investigate the microstructure, the surfaces of the specimens were mirrored using papers of silica and alumina powder. Then, using a solution of 10 mL of deionized water, 70 mL of acetone, 10 mL of acetic acid, and 4.2 g of picric acid powder, the surface of the samples was chemically etched [18]. Finally, microstructural variations were studied using optical microscopy.

FE investigations were also performed by the commercial software Abaqus/Explicit for the evaluation of the deformation behavior, strain distribution, and the required force for processing during HCEE and conventional CEE processes. An axisymmetric 2D analysis was used with automatic remeshing for the accommodation of the applied larger strains. The die parts were defined as rigid bodies, while the sample was considered to be deformable. The Poisson's ratio and Young's modulus of the experimental alloy were considered as 0.35 and 45 GPa, respectively. The stress–strain behavior of AM60 alloy was determined by a compression test at 300°C at a strain rate of 10^{-2} s^{-1} and was used in the FE simulation. Frictionless contact was considered between the sample/die and fluid. At the deformation zone, the friction coefficient at the die/sample interface was considered to be 0.07. Moreover, for the conventional CEE simulations, the friction coefficient was selected as 0.07 in all contacting surfaces [19–20].

3. Results and discussion

3.1. HCEE processing of AM60 magnesium alloy

In the HCEE process, pressurized fluid was used, which greatly affected the process and type of sealing. In this study, the surfaces of the die components were well sealed with copper multi-layer sealants, and the AM60 magnesium rod was severely deformed using the HCEE process at the elevated temperature of 300°C. Fig. 3 shows the sample of the AM60 magnesium alloy after the process.

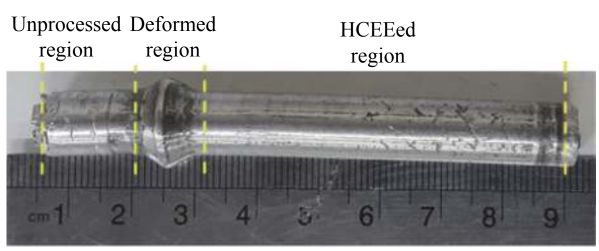


Fig. 3. HCEEed AM60 magnesium sample.

3.2. Microstructure

Fig. 4 shows the images of the cross section of the sample in both unprocessed and HCEEed regions in different paths along the outer surface of the sample (longitudinal path A–B–C), along with the central part of the sample (longitudinal path D–E–F), and along the radius of the sample (transverse path G–H). As it is shown, in the unprocessed region (points A and D) with a coarse microstructure (grain size of

107 μm), $Mg_{17}Al_{12}$ interphase was located between grains. Fast cooling during the casting of AM60 caused non-equilibrium solidification, which contributes to the formation of β-Mg ($Mg_{17}Al_{12}$) rather than the α-Mg and its distribution along the grain boundaries. These hard and brittle $Mg_{17}Al_{12}$ interphase particles reduce the percentage of elongation in magnesium cast alloys [21]. Furthermore, due to the random grain orientation and the lack of mechanical twins and subgrains, dynamic recrystallization (DRX) was not affected by the initial texture of the as-cast Mg [22]. Based on the die geometry, the material in point B (expansion region) was deformed due to the shear strain, which caused variation in the microstructure. Therefore, initial grains were elongated from the center to the outer regions. The nucleation of fine and ultrafine grains along the preexisting boundaries was due to stress concentration at the grain boundaries, which is related to DRX and the pinning effect of β-phase during the process [23]. By the further movement of the punch, the material enters the extrusion region. In this region, the material that has been expanded in the former region is extruded. Elongated grains are also observed in this region (point C), and the ultrafine-grained microstructure is formed. Under the effect of SPD, the HCEE process not only refines the α-Mg grains but also ruptures the coarse intermetallic phases and makes them distribute evenly, which increases the percentage of the elongation. Furthermore, as shown in Fig. 4 (point C), fine grains surrounded the coarse grains and the microstructure of the alloy was bimodal, which reflects the emergence of DRX

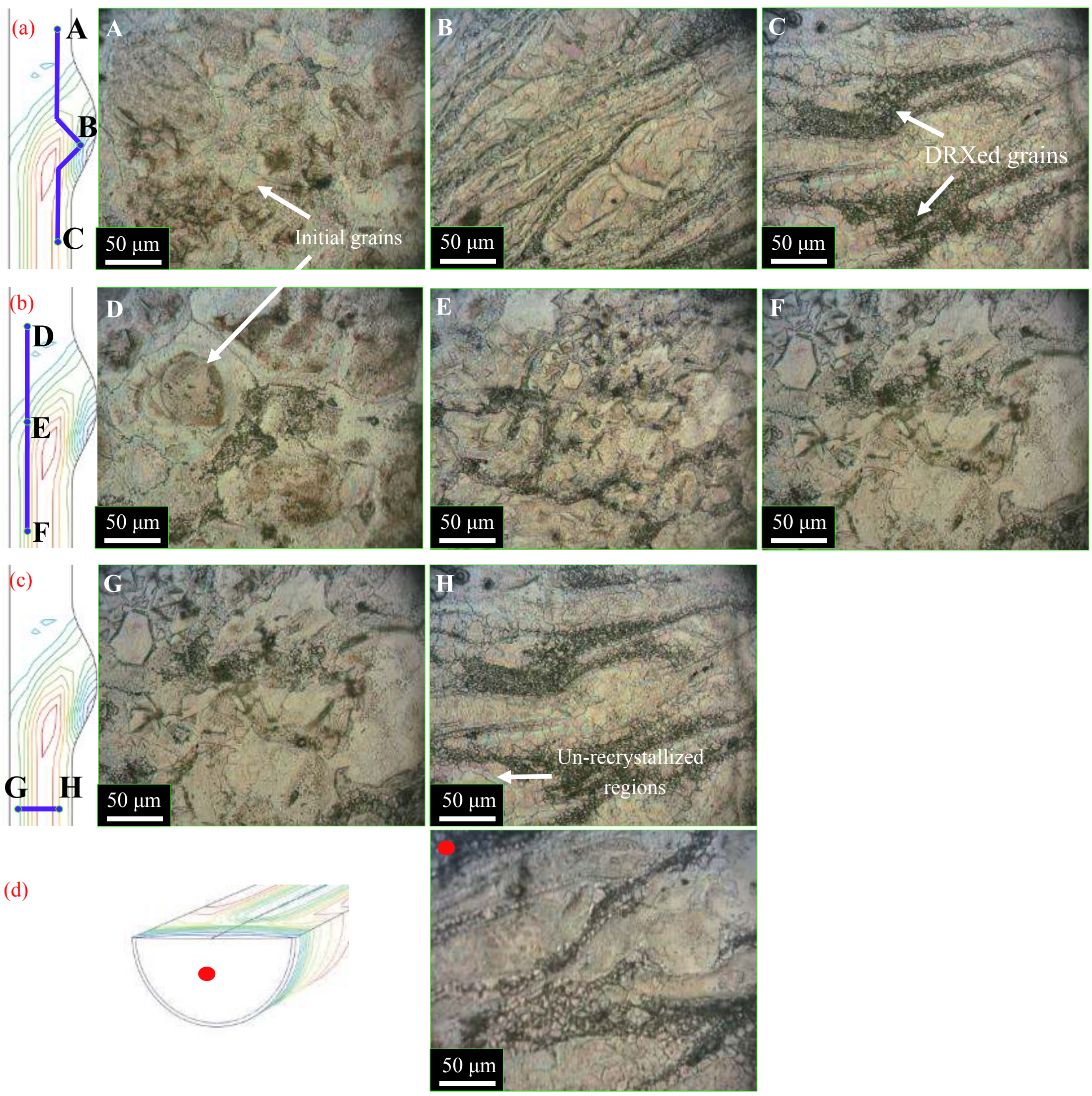


Fig. 4. Variation of the microstructure of AM60 magnesium alloy (a) in longitudinal path A−B−C from unprocessed zone A to processed zone C, (b) in longitudinal path D−E−F from unprocessed zone D to processed zone F, (c) in transverse path G−H in the processed zone, and (d) in the transverse cross section during HCEE.

[23]. Path D−E−F is another investigated path along the centerline of the sample. In this path, the material was mostly affected by the normal strain rather than shear strain; the grain refinement, which is influenced by the shear strain, was not significant in the inner region compared with the regions far from the centerline. Dynamic recrystallization occurred in point E; however, the amount of the nucleation was less than in the region near the outer surface. The shape of the grains in these regions seemed like that of the as-cast one, while elongated grains were observed in regions near the surface. The microstructures in path G−H were almost similar. However, more nucleation and finer structures appeared in point H. The different contributions of shear and normal strains led to a heterogeneous microstructure. Microstructure evolution was also investigated in the transverse cross section. The obtained microstructures are shown in Fig. 4(d). As it is shown, different microstructures at various zones were obtained. The initial large β-phases were crushed into small pieces and distributed at grain boundaries.

Furthermore, the material flow of AM60 during the process of HCEE is shown in Fig. 5. The material flow was in such a way that finer and elongated grains were formed at the outer surface of the die after the process. The material in the deformation region and near the die surface underwent severe shape changes, and therefore experienced a different shear and normal strain and stress contribution. A higher shear strain was more tangible in this region of the sample rather than the inner region. In central regions, the shear strain was lower, which resulted in lower shape changes of grains [22]. A higher shear strain in the outer parts of the sample caused the grains to elongate and become finer (Figs. 5(b)–5(d)).

3.3. Mechanical properties

Fig. 6(a) shows graphs obtained from the tensile test of the initial sample and the ultrafine-grained specimen processed by the HCEE at 300°C. The strength of the AM60 samples increased after the HCEE process. This indicates the importance of the HCEE process, which not only led to the production of the long ultrafine-grained rods but also a sample that reached optimal mechanical properties. The sharp reduction in the grain size and distribution after the SPD process increased the dislocation density; thus, the strength increased according to the Hall-Petch equation established by Hall [24]. Dynamic recrystallization is the main cause for grain size reduction in magnesium alloys. As a result, new grains nucleate, and the fracture volume of the grain boundaries goes up. Therefore, the grain size reduced after one pass of the HCEE process and the strength incredibly increased. The yield (YS) and ultimate tensile strength (UTS) of the AM60 magnesium rod after a pass of the HCEE process increased from 138 and 221 MPa to 212 and 317 MPa, respectively (Fig. 6(b)). Moreover, in this study, the elongation of AM60 increased. The reason for these increases is the uniform distribution of particles ($Mg_{17}AL_{12}$) in the grain boundaries [25], grain refinement, and higher hydrostatic pressure, which demonstrates the importance of the HCEE. Grain refinement leads to a decrease in the ratio of $CRSS_{prismatic}/CRSS_{basal}$ ($CRSS_{prismatic}$: the critical resolved shear stress of prismatic slip rate; $CRSS_{basal}$: the critical resolved

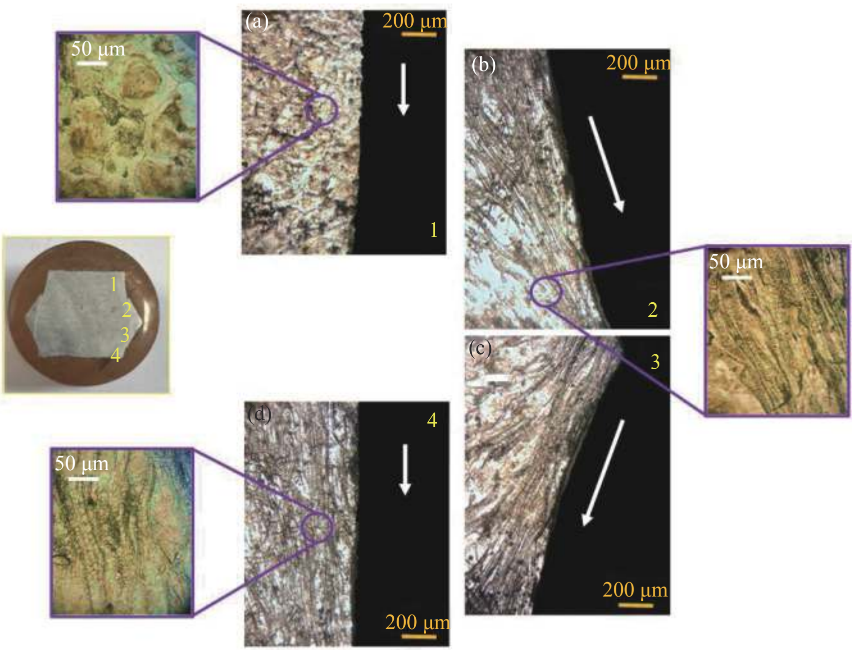


Fig. 5. (a–d) Material flow structure of HCEEed magnesium AM60 sample.

shear stress of basal slip rate). As a result, the reduction in grain size activates the prismatic slip and increases the elongation of the Mg alloy. Table 1 shows the YS, UTS, and elongation of HCEEed AM60 magnesium rod in comparison with other SPD methods. Unlike other SPD methods, in this study, better mechanical results were achieved. For instance, Kulyasova *et al.* [26] realized an increase in the ultimate strength of AM60 Mg alloy by up to 290 MPa after one pass of ECAP at 350°C. In another research of AM60 Mg alloy, which is being ultrafine-grained by the ECAP process, the ultimate strength was 319 MPa after two passes of ECAP process at 220°C [1], and this is close to the result of this study after one pass of the HCEE process. The ultimate strenghs in CEE and HCEE were almost the same. However, other mechanical properties in HCEE were better. As earlier mentioned, one of the important reasons for these superior properties in HCEE compared with the conventional CEE is the presence of hydrostatic pressure in the process, which prevents the formation of microcracks and improves the percentage of elongation.

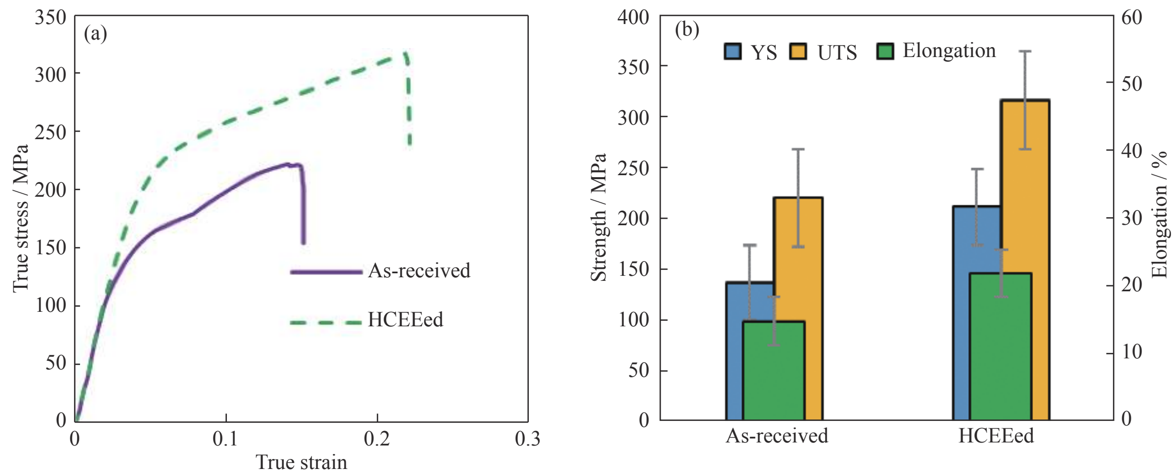


Fig. 6. (a) Stress–strain curves of the initial and the fine-grained AM60 magnesium sample processed by the HCEE; (b) YS, UTS, and elongation of AM60 magnesium before and after HCEE.

Table 1. YS, UTS and elongation of HCEEed AM60 rod in comparison with other SPD methods

Method	Process conditions			Mechanical properties			Ref.
	Pass number	Effective strain	Temperature / °C	YS / MPa	UTS / MPa	El / %	
ECAP	2	2.00	220	240	319	26	[1]
ECAP	1	1.05	350	130	290	23	[26]
CEE	1	1.34	300	150	320	14	[23]
HCEE	1	1.34	300	212	317	22	This study

To determine the hardness homogeneity along the radius of the HCEEed sample, Vickers microhardness measurement was conducted. Fig. 7(a) shows the microhardness of the HCEEed sample compared with the initial value. The average microhardness increased from an initial value of HV 55.0 to HV 72.5 after the process. Furthermore, Fig. 7(b) represents the distribution of the microhardness through the thickness of the unprocessed and HCEEed samples. Due to the Hall-Petch relationship and its coefficients dependency on texture, it may be demonstrated for the HCEEed AM60 alloy that the microhardness values can be controlled by reduction in grain size and the texture variation [27]. According to Fig. 7, microhardness after the HCEE process increased by over 20% of the initial unprocessed state. Distribution of microhardness after the HCEE process was rather non-uniform, and the main reason for this is partially the non-uniform distribution of the equivalent strain.

3.4. FE results

For a better investigation of the fluid's role in the HCEE process, FE simulations were conducted for two different AM60 sample lengths of 30 cm and 10 cm with and without fluid pressure in ABAQUS. The required force for processing is depicted in Fig. 8. As shown, the maximum required force for CEE processing of 30 cm sample was 1150 kN. However, this amount was reduced to the maximum value of 90 kN for the hydrostatic CEE. For 10 cm sample, the maximum required force for CEE processing was 350 kN; however, in HCEE, the force was reduced to the maximum value of 90 kN. Furthermore, the force contour for HCEE processing of 10 and 30 cm samples are overlapped, which indicates that the HCEE process is independent of the

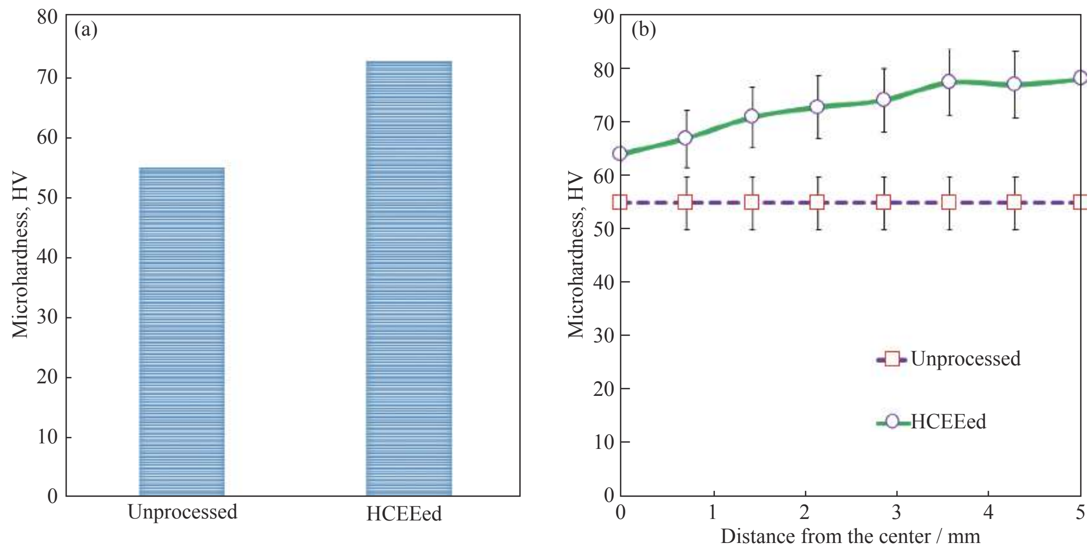


Fig. 7. (a) Average microhardness and (b) microhardness vs. distance along the radial direction of the unprocessed and HCEE-processed AM60 magnesium alloy.

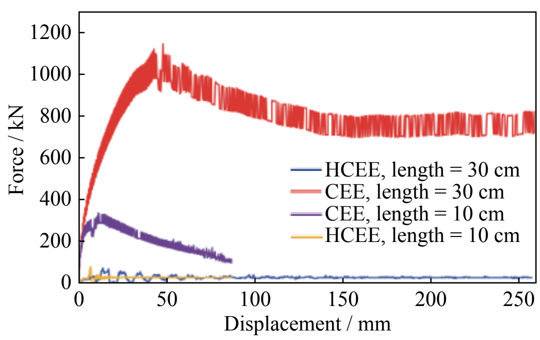


Fig. 8. Force–displacement diagrams of CEE and HCEE for different lengths of the AM60 sample.

length of the sample.

Equivalent strain distribution contours for HCEE and CEE with a frictional coefficient of 0.07 during the process are indicated in Fig. 9(a). To better understand the inhomogeneity in the radial direction, the strain variations are plotted as a function of distance along path A−B at the end of the process in Fig. 9(b). Based on its slope, the curve is divided into three various regions. The slope of the curve changes at the end of each region, which indicates the entrance of another zone. By moving forward to the next zone in both cases, the value of strain increases. Moreover, the microstructure of the three different zones is indicated in Fig. 9(c) to further investigate the relationship of the effective strain and the mi-

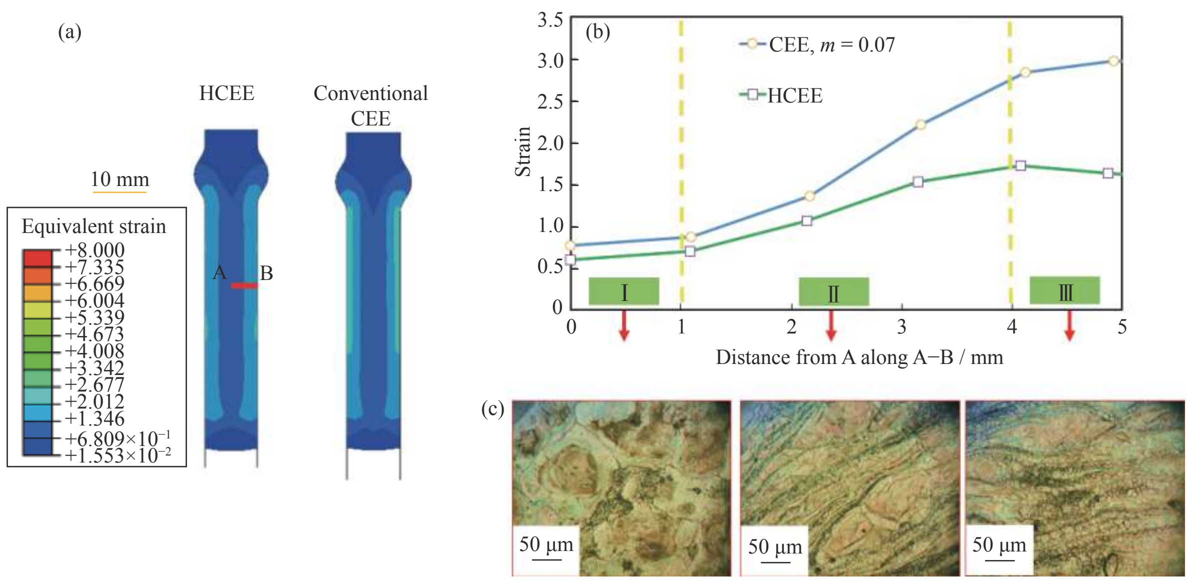


Fig. 9. (a) Strain distribution contour, (b) strain along the path A−B during HCEE and CEE, and (c) AM60 HCEEed microstructure at 300°C in three mentioned regions.

crostructure variation. More strain is shown in zone III; subsequently, more DRXed grains were nucleated and grown in this zone. The initial large grains are shown in zone I, which were decreased by moving to zone III. The inhomogeneity in microstructure and also the distortion of grains is in good agreement with the inhomogeneous distribution of effective strain obtained from FE analysis. Moreover, the inhomogeneity in strain distribution and microstructure resulted in the inhomogeneous microhardness, which is shown in Fig. 7. At the end of the process, the strain value at the outer surface of the sample was higher in CEE than in HCEE. Although higher frictional coefficient caused higher strain in the outer surface of the sample, a better strain distribution along the radius of the sample is more important than the value of strain and helps in achieving a more homogenous structure and better properties. In HCEE, along with lower required processing force, strain distribution was better, and this is the reason HCEE yields superior mechanical properties than conventional CEE.

4. Conclusion

In this study, a HCEE method was used to produce long ultrafine-grained rods from less deformable metals at an elevated temperature. To eliminate frictional forces and produce longer samples, high-pressure molten linear low-density polyethylene (LLDPE), as a pressurized fluid, was used in this process. This method was successfully carried out on magnesium AM60 at 300°C. Optical microscopy images showed that the HCEE process is a suitable method for fabrication of fine-grained microstructures. Equiaxed and elongated grains were shown in the central and outer regions of the processed rods, respectively, and this can be attributed to the different contributions of shear and normal strain in this process and the recrystallization mechanism. Furthermore, the following results were obtained.

(1) The yield and ultimate tensile strength of the AM60 Mg rod increased significantly after the HCEE process. Their values increased from the initial values of 138 and 221 MPa to 212 and 317 MPa, respectively.

(2) The elongation of the HCEE-processed magnesium increased due to the refined grains and secondary phases.

(3) The microhardness increased as the strength did; its value was 20% better than the initial state. The microhardness distribution was almost non-uniform across the sample's radius.

(4) FE analysis showed that the HCEE process was independent of the length of the sample.

Acknowledgement

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