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WROUGHT MAGNESIUM ALLOYS ZM21, ZW3 AND WE43 PROCESSED BY HYDROSTATIC EXTRUSION WITH BACK PRESSURE

PRZEROBIONE PLASTYCZNIE STOPY MAGNEZU ZM21, ZW3 I WE43 WYTWORZONE METODĄ WYCISKANIA HYDROSTATYCZNEGO Z PRZECIWCIŚNIENIEM

Cold hydrostatic extrusion with and without back pressure of commercial ZM21, ZW3 and WE43 magnesium alloys has been performed at originally designed hydrostatic extrusion press operating up to 2000 MPa with back pressure up to 700 MPa. Alloys were cold extruded in one pass into rods between 5 and 9 mm in the outer diameter with product velocities between 1 and 10 m/min and extrusion ratios above 2. Application of back pressure extended formability of all magnesium alloys. It was due to hydrostatic pressure superimposed on the extruded product what inhibits the cracks generation and propagation. Cold deformation restrained the grain growth and softening processes while severe deformation in one pass increased grain refinement and density of internal defects. Ultimate tensile strength ranging from 370 MPa (ZM21) through 400 MPa (ZW3) up to 410 MPa (WE43), with respective yield stresses from 270 MPa through 300 MPa up to 350 MPa and the respective elongation from 13% through 12% to 7% were obtained in extruded rods, which are the best reported data in literature up to this day. Wrought magnesium alloys after hydrostatic extrusion can serve as semi-products for structures that call for high strength, for example as biodegradable implants or fastening components in form of bolts, rivets, nuts, pins, joints, etc.
Keywords: hydrostatic extrusion, back pressure, magnesium alloys, processing, mechanical properties, grain refinement

Przeprowadzono wyciskanie hydrostatyczne na zimno z przeciwciśnieniem handlowych stopów magnezu ZM21, ZW3 i WE43. Eksperymenty prowadzono na oryginalnej, własnej konstrukcji prasie do wyciskania hydrostatycznego pracującej do 2000 MPa z przeciwciśnieniem do 700 MPa. Stopy odkształcano na zimno w jednej operacji wytwarzając pręty o średnicach pomiędzy 5 mm a 9 mm z liniową szybkością wyciskania pomiędzy 1 m/min i 10 m/min i stopniami redukcji powyżej 2. Zastosowanie przeciwciśnienia zwiększyło zdolność do odkształcenia plastycznego wszystkich badanych stopów magnezu. Stało się tak dzięki nałożenia na wyciskany produkt ciśnienia hydrostatycznego co powstrzymuje generowanie i propagację pęknięć. Odkształcanie na zimno powstrzymuje rozrost ziaren i procesy zmiękczania podczas gdy duże odkształcenie plastyczne w jednej operacji zwiększa rozdrobnienie ziaren oraz gęstość defektów mikrostruktury. Po wyciskaniu wytrzymałość na rozciąganie wynosiła od 370 MPa (dla ZM21) przez 400 MPa (ZW3) do 410 MPa (WE43) z granicą plastyczności, odpowiednio 270 MPa, 300 MPa i 350 MPa i wydłużeniem, odpowiednio 13%, 12% i 7%. Są to najwyższe własności podawane w literaturze światowej na dzień dzisiejszy. Stopy magnezu przerobione plastycznie metodą wyciskania hydrostatycznego mogą służyć jako półprodukty do wykonania elementów konstrukcyjnych wymagających wysokich wytrzymałości, jak np. biodegradowalne implanty kostne czy elementy złączne w postaci śrub, nitów, nakrętek, szpilek, łączników, itp.

1. Introduction

The last decade has shown that there is a large focus on magnesium and its alloys due to their strong potential in structural applications as lightweight and energy saving materials [1-3]. Almost 90% of used magnesium alloys are casting alloys. However, casting is often associated with difficulties related to fluidity of these alloys [4] or occurrence of internal defects [5]. Therefore, the wrought magnesium alloys gain their great importance as well. However, low strength and ductility significantly impede their broad application. According to the Hall-Petch relation grain refinement is one of the promising methods leading to strength improvement [6].

Many technologies have been adopted for the preparation of fine grained magnesium alloys, including microalloying, severe plastic deformation, rapid solidification, etc. Severe plastic deformation (SPD) has attracted scientific attention in recent years for the preparation of high-performance materials by grain refinement. It in-

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cluded such processes as equal channel angular pressing (ECAP), high pressure torsion (HPT) or multiple forging (MPF), etc., which in recent years have been also introduced to the field of magnesium alloys.

The application of magnesium wrought components gives the strength as the main functional requirement. In comparison to the current designs in aluminium alloys and steel the weight reduction is the primary reason for considering of magnesium alloys application. Forming behaviour of magnesium is poor at room temperature, therefore most of conventional processes require that material is heated to temperatures between 230°C to 370°C [7,8]. The hexagonal structure of magnesium requires elevated forming temperatures to activate more slip systems [9,10]. Wrought alloys have higher strength and ductility in comparison with cast alloys, although their final strength is strongly suppressed by treating at elevated temperatures.

Lowering the deformation temperature and increasing the deformation rates became possible through application of the hydrostatic extrusion (HE) process at elevated temperatures [1,11]. During the hot hydrostatic extrusion the deformation temperature was lowered down to 100°C for such alloys as AZ31, AZ61, ZM21 ZK30 and ZE10 and to 110°C for AZ80. The major benefits of this method can be attributed to the hydrostatic stress factor present within the deformation zone. However, it was observed, that processing of magnesium alloys at elevated temperatures does not enable, even when severe plastic deformation in one pass is used, to eliminate recrystallisation and to achieve a submicrometer range of grains. Thus, the final tensile yield strength of processed material does not exceed, for example, 180 MPa for ZM21 alloy [1,11].

In this work, hydrostatic extrusion at room temperature of commercial ZM21, ZW3 and WE43 magnesium alloys has been performed. To enhance products integrity hydrostatic extrusion with back pressure has been utilized. Although, the back pressure applied for conventional extrusion has long history [12,13], for magnesium alloys it was applied seldom and with deformation at elevated temperature, as in hot forward extrusion [14] or hot equal channel angular pressing (ECAP) [15]. To run the process the unique press with precise control of back pressure has been designed and built. Its capability and performance parameters were described elsewhere [16]. Mechanical properties of three magnesium alloys with respect to process parameters are discussed and compared with up-to-date literature data.

2. Experimental procedure

The process of hydrostatic extrusion with back pressure differs from the classical hydrostatic extrusion [17] in high pressure acting on the product emerging from the extrusion die. While the extruded product penetrates the back pressure chamber (BP) the hydrostatic stress is superimposed on the material and inhibits its cracking. Extrusion experiments were carried out on unique, own designed Unipress press with extrusion pressure up to 2000 MPa and back pressure up to 700 MPa. The principle of hydrostatic extrusion with back pressure is presented in Fig. 1. The billet material located in high pressure chamber is extruded in form of product into the back pressure chamber. Extrusion starts up when certain pressure level, specific for given material in hydrostatic extrusion chamber is generated (higher pressure) and extruded product penetrates the back pressure chamber (lower pressure). Required pressure difference between hydrostatic and back pressure chambers is maintained throughout the full extrusion process.

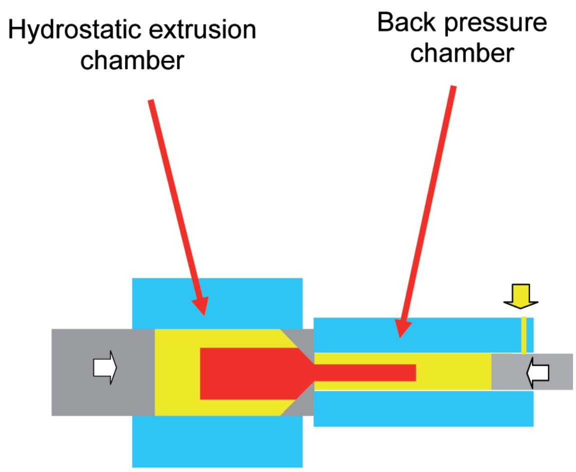

Fig. 1. The principle of hydrostatic extrusion with back pressure

Commercial wrought magnesium alloys: ZM21 (Mg-2.2%Zn-0.8%Mn), ZW3 (Mg-3.3%Zn-0.74%Zr) and WE43 (Mg-3.9%Y-2.2%Nd-0.56%Zr), where compositions are given in wt.%, in the form of extruded rods have been provided by Magnesium Elektron, England. The initial microhardness HV0.2 and the equivalent grain diameter d_2, defined to a diameter of a circle which has an area equal to the surface area of a given grain are shown in Table 1.

TABLE 1
Microhardness HV0.2 and average grain size d_2 of tested materials in initial state before hydrostatic extrusion

material	microhardness HV0.2	standard deviation $SD_{HV0.2}$	average grain diameter d_2	standard deviation SD_{d2}
			[μm]	[μm]
ZM21	56	7	21	9
ZW3	63	4	5	2
WE43	78	2	14	7

The as-received rods have been hydrostatically extruded without and with BP up to extrusion ratio R~4. Extrusion ratio is defined by $R = (d_0/d_f)^2$, where d_0 denotes the billet diameter and d_f the product diameter, while the true strain is defined as ε = lnR. Extrusion ratio has been increased until the maximum possible R for sound (uncracked) product was evaluated. All extrusion tests were carried out at room temperature with back pressure of 400 MPa either 700 MPa and the die angle of 45° (rarely 30° and 18°) leading to final product diameters between 5 mm and 9 mm in outer diameter. Linear extrusion velocities were tried from a range between 1 m/min and 10 m/min. Mixture of ~60% MoS_2 and refined oil was used as the lubricant.

Microhardness was measured on the transverse and longitudinal sections using load of 200g and a 15s test period. Tensile specimens with a gauge section of 4mm in OD and 5 times in length were machined from extruded rods and investigated in room temperature and at a constant strain rate of $0.008s^{-1}$. The optical profilometer was used to investigate the surface quality of selected initial billets and extruded rods. The structures were examined using an optical and the transmission electron microscopy (TEM) on longitudinal and transverse cross-sections.

3. Results and discussion

The pressure characteristics of hydrostatic extrusion without and with back pressure are shown in Fig. 2. Extrusion pressures increase with an increase of true strain and are higher for harder initial material, see Table 1. For increasing BP the plots inclination become steeper. The extrusion pressure with BP was not a pure arithmetic sum of extrusion pressure without back pressure plus back pressure value itself. Differences between these values reached up to 400 MPa and are attributed to differences in deformation geometry, strain hardening properties, strain rates and lubrication conditions between individual experiments.

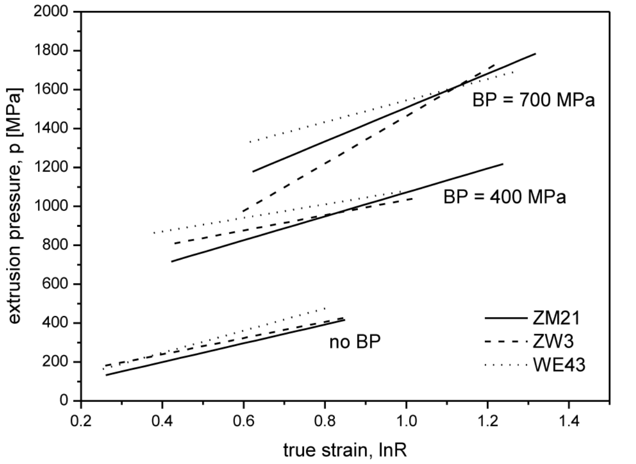

Fig. 2. The pressure characteristic of hydrostatic extrusion without and with back pressure of 400 MPa and 700 MPa for ZM21, ZW3 and WE43 alloys

The application of back pressure has extended available true strains for which sound products were obtained, Table 2. For different materials that strain increase was different: highest, over threefold for ZW3, moderate by 50% for ZM21 and the smallest by 10% for the most hard WE43 alloy. It correspond to maximum extrusion ratios R~2.7 for two first alloys and over 2.3 for the last one. Bulk, sound products obtained at the maximum true strains without and with back pressure are shown in Fig. 3. Some products have revealed the surface defect call 'hot shortness', i.e.: tendency of alloy to inter-crystalline fracture with presence of liquid face along grain boundaries. This defect is usually associated with hot working and higher deformation velocities [18]. In current work, which has involved cold deformation the 'hot shortness' effect can be attributed to adiabatic heating acting on material within the working zone in extrusion die. Estimation of this effect based on the mechanical work of plastic deformation done during extrusion gives the range between 0.38 and 0.63 of the absolute melting temperature of investigated alloys. The other factor enhancing the temperature effect is the extrusion velocity, which is difficult to maintain low during hydrostatic extrusion process. When the extrusion velocity increases the temperature rise due to more intense plastic deformation. This effect occurs mainly at the surface where friction adds to the phenomenon. The lowest velocity during present experiments was ~1 m/min and in most cases a few meters per minute, i.e.: equal or higher than the values commonly reported as the maximum possible extrusion velocity for this kind of materials [11,19].

TABLE 2
Maximum extrusion ratio and true strain for which sound, uncracked products were obtained during cold hydrostatic extrusion without and with back pressure

Material	Without back pressure		With back pressure	
	Extrusion ratio, R	True strain, ε	Extrusion ratio, R	True strain, ε
ZM21	1.93	0.66	2.67	0.98
ZW3	1.37	0.31	2.66	0.98
WE43	2.13	0.76	2.33	0.85

Figure 4 shows the dependence of surface roughness on true strain during hydrostatic extrusion without and with back pressure in case of WE43 alloy. Character of graphs for two other alloys is identical. It is seen in Fig. 4, that: surface roughness for extrusion with BP is much higher than that for extrusion without BP, roughness values steeply increase with strain and for extrusion without BP roughness slightly decreases with strain. These differences result from higher velocities associated with higher extrusion strains, i.e.: stronger adiabatic heating during extrusion with BP in comparison to extrusion without BP. Therefore, the surface roughness during extrusion with BP was deteriorated in comparison to machined, starting surface of the billet, in contrary to extrusion without BP for which slight improvement in surface quality was detected. The longitudinal roughness values in case of the first did not exceeded Ra<3.5μm while for the last Ra<0.5μm was measured. More precise determination of the onset of surface cracking by 'hot shortness' effect requires detailed study which is beyond the scope of present work.

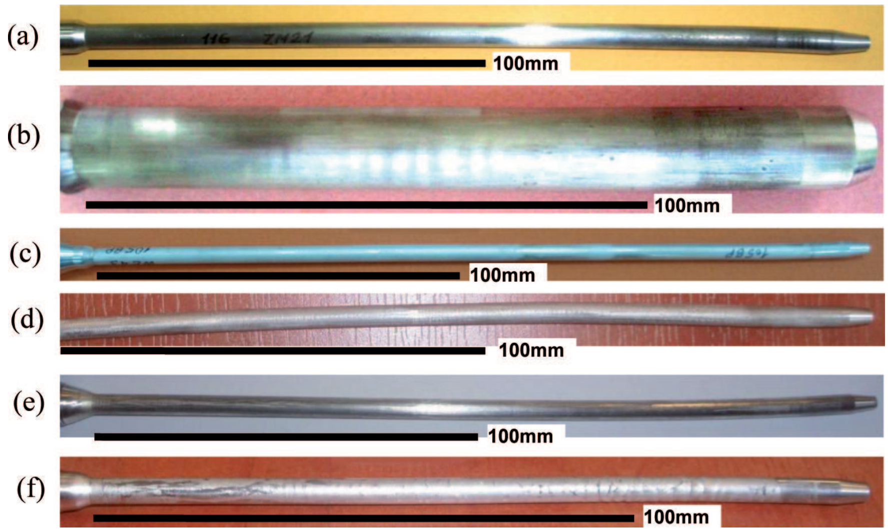

Fig. 3. Magnesium wrought alloys (a), (d) ZM21, (b), (e) ZW3 and (c), (f) WE43 after cold hydrostatic extrusion, (a)-(c) without back pressure and (d)-(f) with back pressure; (a) ZM21, true strain ε =0.66, extrusion ratio R=1.93, diameter 6.5 mm, and respectively, (b) ZW3, ε =0.31, R=1.37, 15 mm, (c) WE43, ε =0.76, R=2.13, 5.5 mm, (d) ZM21, ε =0.98, R=2.67, 5.5 mm, back pressure BP=400 MPa, (e) ZW3, ε =0.98, R=2.66, 5.5 mm, back pressure BP=700 MPa and (f) WE43, ε =0.85, R=2.33, 5 mm, back pressure BP=400 MPa

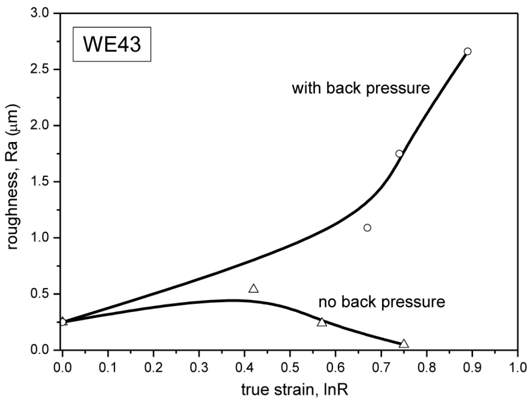

Fig. 4. The dependence of surface roughness on true strain for hydrostatic extrusion of WE43 magnesium alloy without and with back pressure

The application of back pressure has enabled to apply over 60% reduction per one pass leading to high elongation of grains, Fig. 5. Their alignment has reflected the starting grain diameter and the applied extrusion ratio. Grains elongation is accompanied by the high refinement of grains in extruded rods, Fig. 6. Observed microstructures are highly inhomogeneous with a high density of internal defects. Presence of mixed fractions of coarse grains above 1μm, Fig. 6(d), together with nanometric scale sub-grains and grains, Fig. 6(a-c), is observed in all alloys. Commonly observed are slip bands, Fig. 6(b), and disorder zones proving occurrence of severe deformation, Fig. 6(c). This high diversity of microstructure's forms is the consequence of competitive processes: strain hardening due to severe plastic deformation and strain softening due to dynamic recovery caused by the adiabatic heating during deformation. The deformation followed by recovery processes leads to the segmentation of grains and the appearance of sub- and nano-grains.

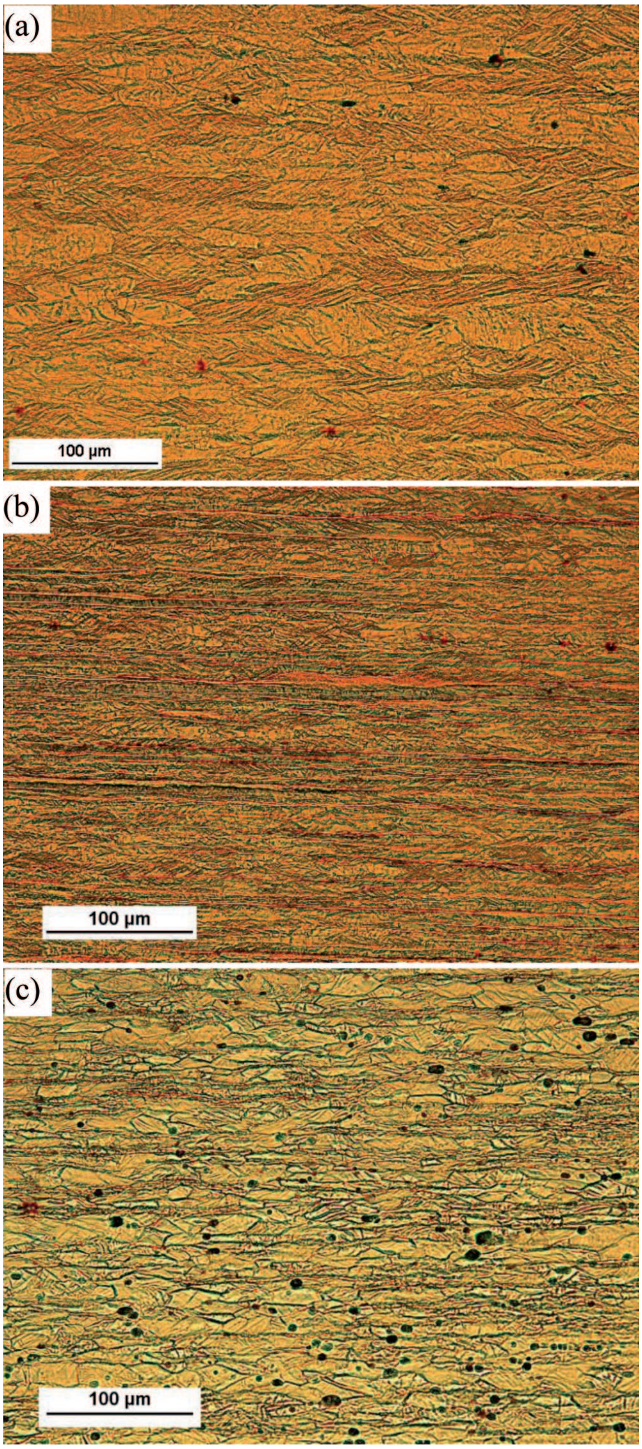

Fig. 5. Microstructures at longitudinal cross sections after hydrostatic extrusion with back pressure of (a) ZM21 alloy, back pressure BP=700 MPa, extrusion ratio R=2.56 (true strain ε =0.94), (b) ZW3 alloy, BP=700 MPa, R=2.66 (ε =0.98), and (c) WE43 alloy, BP=400 MPa, R=2.10 (ε =0.85)

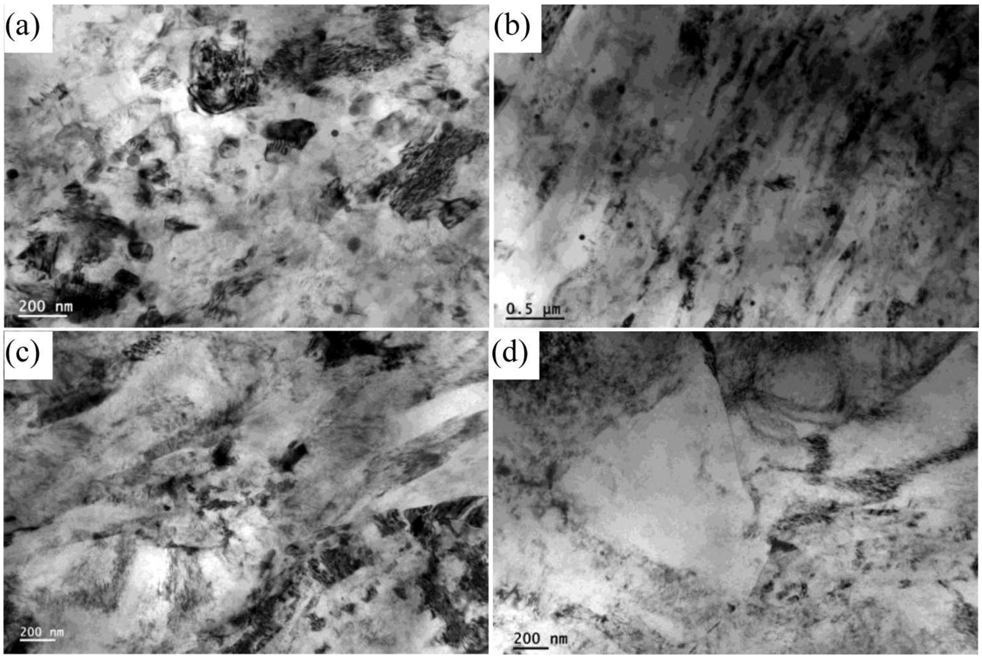

Fig. 6. Various forms of micro- and nanostructures observed in magnesium alloys after cold hydrostatic extrusion with back pressure: in ZM21 alloy, back pressure BP=400 MPa, extrusion ratio R=2.67, true strain ε =0.98 (a) nano-grains and subgrains, (b) slip bands, and in WE43 alloy, BP=400 MPa. R=2.1, ε =0.85 (c) localized high strains, and (d) contact point of bigger adjacent grains

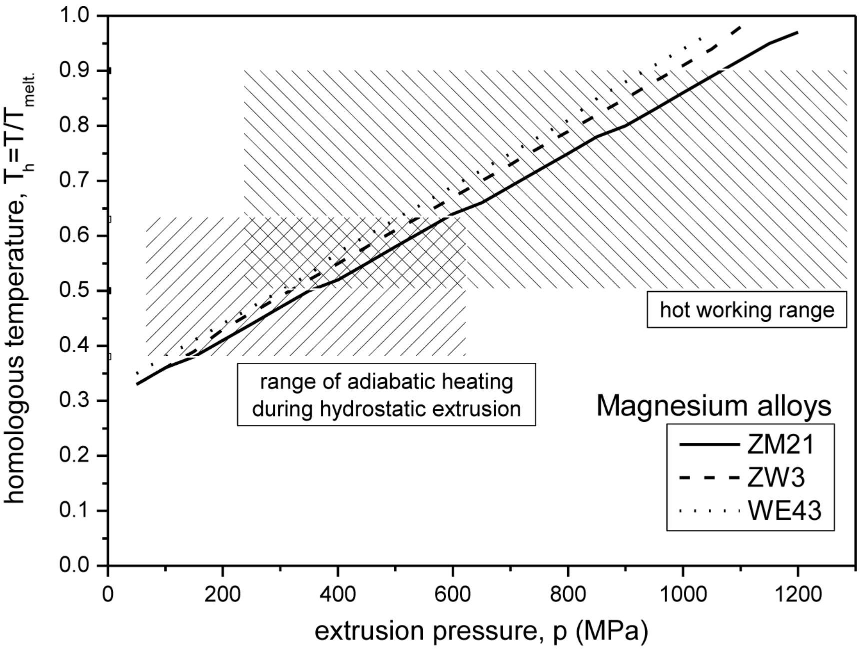

Fig. 7. The hot working range and the range of adiabatic heating during cold hydrostatic extrusion for ZM21, ZW3 and WE43 magnesium alloys. Note: both ranges overlap

For investigated magnesium alloys the magnitude of adiabatic heating during cold hydrostatic extrusion can situate the temperature of metal workpiece within the range attributed for their hot working range, see Fig. 7. Temperature increases with extrusion pressure since pressure reflects the work of deformation per unit volume [20] and has a direct influence on the final microstructure causing its high diversity in observed forms and sizes.

Substantial improvement of mechanical properties was observed for all investigated magnesium alloys after hydrostatic extrusion as the consequence of accumulated plastic deformation, large number of internal defects and grain refinement. Magnitude of this effect was much above the level observed for these materials processed by conventional methods, as drawing, rolling, forging, extrusion or by special method as ECAP. All of these processes must be performed at elevated temperatures when integrity of materials has to be maintained. In Table 3 the comparison between the best results obtained in current work and the best literature data is presented. The strength of all materials processed by cold hydrostatic extrusion with back pressure is evidently higher in comparison to materials conventionally extruded [2, 21], ECAP-ed [22], forged [21] and hydrostatically extruded [1]. Application of back pressure allows to apply higher strains and strain rates at room temperature in comparison to conventional processes what is responsible for such distinct increase of the strength. This increase exceeds 40% in case of UTS and 70% in case of YS for ZM21 and WE43 alloys and it attain over 30% in both properties for ZW3 alloy. Obviously, elongation to fracture is reduced inversely to measured strength by 40-55% in case of two first alloys and maintaining at similar lever for the third alloy. Enormous strength enhancement results from one main reason: room temperature of deformation enabling substantial grain refinement due to increase of available extrusion ratio in one pass by applying the back pressure. It has allowed to increase attainable extrusion ratios by 1.5-2 times for softer ZM21 and ZW3 alloys and by10% for the most hard WE43 alloy, see Table 2. Cold deformation restrains the grain growth and softening processes occurring within microstructure while severe deformation in one pass increases density of internal defects and decreases dislocation mobility.

4. Possible applications

Wrought magnesium alloys after hydrostatic extrusion can be applied for structures that call for high strength as such and accept these alloys in the as extruded condition. It is possible when given alloy can be extruded into final shape product or the final geometry can be obtained by machining, welding, forging and rolling of the extruded semi-products. However, welding or forging can be limited by necessity to avoid temperature effect and by the high strength and lower ductility of the as-extruded material.

The high strength magnesium alloys can be utilized as semi-products for miniaturized devices in application areas such as medical equipment, semiconductor manufacture, robotics and biotechnology. The low Young modulus is of benefit in reducing the stress shielding at the bone–implant interface, which makes magnesium alloys very interesting candidates for biodegradable bone implants [23]. WE43 alloy has been recently considered as a candidate for coronary stents, what became as a novel technological approach [24].

TABLE 3

Comparison of mechanical properties of ZM21, ZW3 and WE43 magnesium alloys after cold hydrostatic extrusion with back pressure and the best literature data for this same alloys fabricated by traditional processing methods

Material	Processing	Ultimate tensile strength, UTS [MPa]	Tensile yield stress, YS [MPa]	Elongation to fracture, ε_f [%]	Reference
ZM21	HE + BP[(a)]	371	268	13.3	current
	HE 220-260^{o}C	258	175	23	[1]
	extruded rod	245	160	10	[21]
ZW3	HE + BP	398	296	11.7	current
	extruded rod	305	225	8	[21]
WE43	HE + BP	411	344	6.8	current
	ECAP 320-370°C +EX+DR+ANNEAL[(b)]	300	250	23	[22]
	forgings precipitation treated	285	155	6	[21]
	extruded, artificially aged	270	195	15	[2]

[(a)] HE – hydrostatic extrusion
BP – back pressure
[(b)] ECAP – equal channel angular pressing
EX – extrusion
DR – drawing
ANEAL – annealing

Development of special forming processes, such as cold hydrostatic extrusion with back pressure allows to extend the use of magnesium alloys in different industrial applications, which require extremely high mechanical properties. Most of up to date innovative processes as for example hydroforming in ZM21 [1,25] are associated with an increase of forming temperature to overcome formability limitations in magnesium alloys what lowers the strength of fabricated elements.

Direct application of investigated magnesium alloys can also appear in the area of fastening components such as bolts, nuts, pins, joints or different combinations of welded parts, etc., which can be machined from the as-extruded semi-products. If further plastic deformation operations are required such semi-products can be hydrostatically extruded with moderate strength, which are chosen individually, according to next planned forming processes.

Wrought magnesium alloys after hydrostatic extrusion can serve as semi-products for structures that call for high strength, for example as biodegradable implants or fastening components in form of bolts, rivets, nuts, pins, joints, etc.

5. Summary

The study clearly shows applicability of special (unique) forming process as cold hydrostatic extrusion with back pressure for fabrication of ZM21, ZW3 and WE43 commercial wrought magnesium alloys with extremely high mechanical properties. It was due to cold deformation which restrains the grain growth and softening processes occurring within microstructure while severe deformation in one pass increases grain refinement, density of internal defects and decreases dislocation mobility. Ultimate tensile strength ranging from 370 MPa (ZM21) through 400 MPa (ZW3) up to 410 MPa (WE43), with respective yield stresses from 270 MPa through 300 MPa up to 350 MPa and the respective elongation from 13% through 12% to 7% were measured for extruded rods. Obtained results are placed among the best reported in literature up to this day.

Application of back pressure allows to apply higher strains and strain rates at room temperature in comparison to conventional processes, which usually require elevated temperatures, as drawing, rolling, forging or extrusion. An increase of available true strains was also observed when comparison with hydrostatic extrusion without back pressure is made: over threefold for ZW3, moderate by 50% for ZM21 and the smallest by 10% for the most hard WE43 alloy. Increase in available strains is due to the hydrostatic pressure superimposed on the extruded product. Compressive stresses inhibit cracks generation and propagation and increases the material ductility.

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