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Forming of Magnesium Alloys at 100 °C by Hydrostatic Extrusion

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Magnesium wrought alloys are of special interest for use as structural parts due to the possibility of obtaining improved and more homogeneous microstructure and mechanical properties compared with cast components. The market for magnesium wrought alloys is still relatively small, and they are only used for special applications due to the high cost of the feedstock. Currently, with the decreasing prices for the primary magnesium extrusion, magnesium has become competitive with aluminum, and is important for upcoming research and development activities. In this study hydrostatic extrusion, as a quite rarely applied technique, was used for deformation of commercial magnesium alloys at 100 °C, which is significantly below the temperature necessary for activation of new gliding systems. All experiments were carried out using typical industrial extrusion parameters like extrusion rate and extrusion ratio but with the objective of obtaining extremely fine-grained materials as are received typically from equal channel angular extrusion processing. These experiments show that the processing of magnesium alloys is possible even at a temperature of 100 °C. The limitations of this processing and the influence of process parameters on the microstructure and mechanical properties of extruded profiles will be discussed.

Keywords extrusion, grain refinement, magnesium, mechanical properties, microstructure, processing

1. Introduction

Presently, it is possible to obtain fine microstructures of approximately 1 to 4 μm with equal channel angular extrusion (ECAE) pressing, multiple forging, or powder metallurgy (Ref 1-3). But these methods are associated with high costs and are mainly of academic interest. Recent experiments have shown that the hydrostatic extrusion technique is feasible for the production of profiles from magnesium alloys (Ref 4, 5). This forming method enables finer grains, higher extrusion ratios, and extrusion rates at lower extrusion temperatures than commercial direct and indirect extrusion (Ref 6, 7). Its well known that at temperatures below 200 to 225 °C the workability of magnesium alloys is limited or even not possible (Ref 8, 9). Therefore, typical processing temperatures for the extrusion of magnesium wrought alloys are in the range of 260 to 450 °C using direct or indirect extrusion (Ref 10). A similar temperature range is established for die forging and sheet forming too (Ref 11-13).

In the current study, the hydrostatic extrusion method will be used for extrusion trials to reduce the extrusion temperature to significantly below 200 °C. According to our own preliminary tests, this extrusion method should lead to grain refinement, which guarantees improved mechanical properties (Ref 14). This also includes the enhancement of the elongation to fracture, which is generally accepted to be extended even further by grain refinement (Ref 15).

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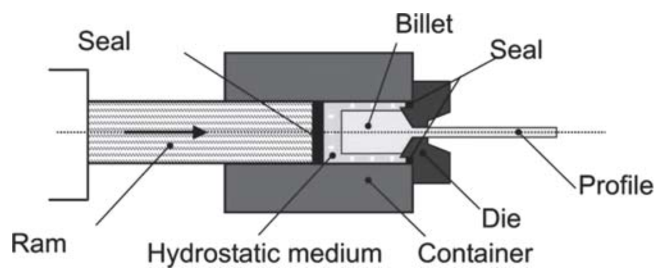

Fig. 1 Schematic drawing of hydrostatic extrusion process

Commercially established wrought magnesium alloys from different alloy series were taken to determine their processing behavior by hydrostatic extrusion trials. Process parameters like extrusion ratio and extrusion rate are similar to typical industrial parameters. It will be shown that the hydrostatic process has a strong influence on the workability, the microstructure evolution, and the mechanical properties of these magnesium alloys. First, the results of the influence of alloying elements and processing on the microstructure will be discussed.

2. Experimental

Commercial DC-cast feedstock with a diameter of 95 mm from the alloys AZ31, AZ61, AZ80, ZM21, ZK30, and ZE10 were machined to billets with a diameter of 80 mm that were used for extrusion trials. The materials were heat-treated for 12 h at 350 °C in the case of the alloys AZ31, AZ61, ZM21, ZK30, and ZE10, and for 12 h at 385 °C in the case of alloy AZ80. All billets were air-cooled afterward.

The principle of the hydrostatic extrusion process is shown in Fig. 1. During hydrostatic extrusion, a hydrostatic medium presses onto the billet. The pressure is developed from a ram and is passed onto the medium, leading to hydro-

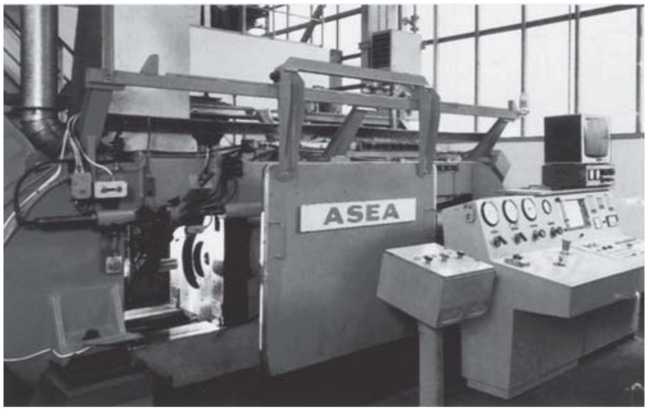

Fig. 2 Used ASEA-12MN hydrostatic press (Freiberg, Germany)

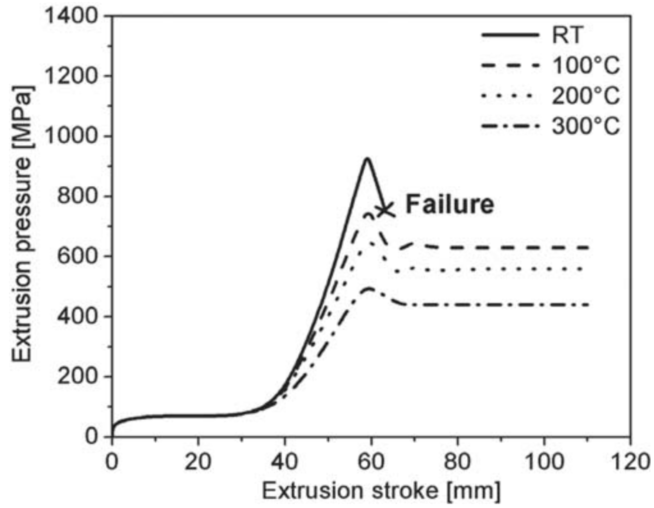

Fig. 3 Developing of the extrusion pressure at increasing extrusion stroke for extrusion trials of alloy AZ31 at different temperatures. The maximum pressure (peak pressure) increases with decreasing extrusion temperature.

static pressure from all sides onto the billet to make it flow through the die. Forming experiments were carried out by hydrostatic extrusion using a 12MN-ASEA (ABB, Sweden) press with a maximum extrusion pressure 1400 MPa (Fig. 2). A detailed description of hydrostatic extrusion can be found elsewhere (Ref 16).

Basic trials were carried out on the wrought magnesium alloy AZ31 with temperatures varying from 300 °C down to room temperature in steps of 100 °C. These experiments were performed to determine the lowest extrusion temperature for the following extrusion trials presented in this article. The rod profiles from the AZ31 alloy were extruded using an extrusion ratio of 1:28 at all temperatures, and the lubricant MoS_2 was used. Despite a high extrusion rate and low forming temperatures, the limit of press capacity (maximum pressure 1400 MPa) was not exceeded, as is seen in Fig. 3 where the extrusion pressure is plotted as a function of the extrusion stroke. To avoid materials failure during the start of the extrusion process, the extrusion die was preheated at 300 °C, but a reduction of the die temperature to 100 °C was also successful. The results shown in this article refer to the die preheated at 300 °C.

All experiments with alloys AZ31, AZ61, ZM21, ZK30, and ZE10 were performed at a process temperature of 100 °C. In preliminary extrusion trials of alloy AZ80 carried out at 100 °C, cold cracks could be observed on the surface of the rods. Therefore, for this magnesium alloy the trial temperature was increased up to 110 °C. Table 1 summarizes the parameters of the extrusion experiments. As is known from conventional extrusion trials of alloys AZ61 and AZ80, extrusion rates of about 2 and 4 m/min, respectively, are regarded as the maximum possible velocity (Ref 17). The experiments shown in this article were performed at an extrusion rate of 8 m/min, representing the lowest possible velocity for hydrostatic extrusion with the 12MN-ASEA press used (Fig. 2).

The trials were accompanied by microstructural analyses, which were carried out on polished and etched longitudinal sections using optical microscopy. For sample preparation, an etching solution based on picrid acid was applied (Ref 18). Tensile and compression tests of samples were performed at room temperature following DIN 50,125 instructions. These experiments were performed using a commercial testing machine (Zwick Z050, Ulm, Germany) with a maximum applied force of 50 kN, whereas a strain rate of 10^{-3} s^{-1} was adjusted in all cases.

3. Results and Discussion

In Fig. 4 to 9, micrographs of the individual feedstock of longitudinal cuts in the center of the billets are shown. The microstructures of alloys AZ31, AZ61, AZ80, and ZM21 exhibit rather coarse grains with a grain size between 300 and 500 μm. Contrary to that, the wrought magnesium alloys ZK30 and ZE10 have smaller grains, with a diameter of approximately 40 to 60 μm (Fig. 8, 9). These alloys contain zirconium, which acts as a grain refiner (Ref 9).

The following extrusion trials were carried out using the process parameters described in Table 1, leading to extruded profiles with a smooth surface at an extrusion temperature of 100 °C, and 110 °C in the case of alloy AZ80 (Fig. 10).

It is worth mentioning that the maximum extrusion pressure of the press was not achieved for each material. This is illustrated in Fig. 11, where the extrusion pressure of the used magnesium alloys is shown for all alloys.

After extrusion trials at 100 °C, micrographs of longitudinal sections of the rods were prepared. They are illustrated in Fig. 12 to 17, where the horizontal direction of all micrographs represents the extrusion direction. It is obvious that the extrusion led to a complete recrystallized microstructure of each investigated alloy. It can be realized that the chosen parameters listed in Table 1 are suitable for supporting dynamic recrystallization at this extrusion temperature.

Investigations of the average grain size of each alloy (Fig. 18) show that for all hydrostatically extruded rods a significant reduction of the grain size was received. From the systematic study of the AZ series, it is obvious that the grain size of this family increases with an increasing content of aluminum. The smallest grain size in the range of 1.8 μm was measured for the wrought magnesium alloy ZE10, followed by the alloys AZ31, ZK30, and AZ61 with a mean grain size between 2 and 4 μm. The development of such small grains could be explained by the lower temperature rise of profiles during hydrostatic extrusion process (Ref 21), but further extrusion trials are necessary for verification. In the cases of alloys AZ80 (Fig. 13) and ZM21 (Fig. 14), the grain size was reduced up to approxi-

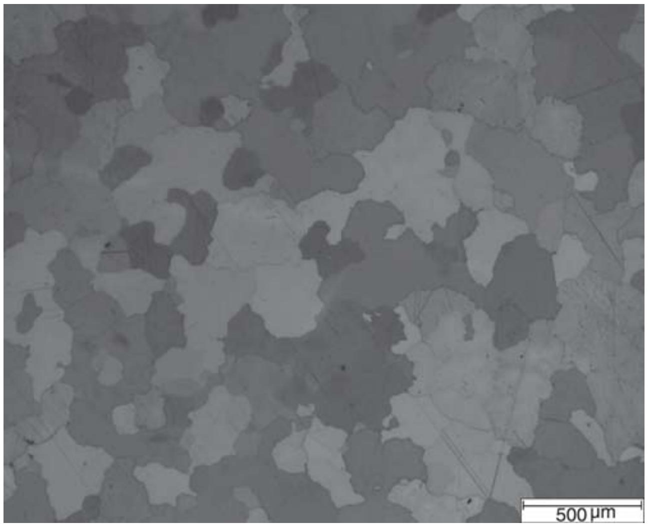

Fig. 4 Microstructure of alloy AZ31 in as-cast condition

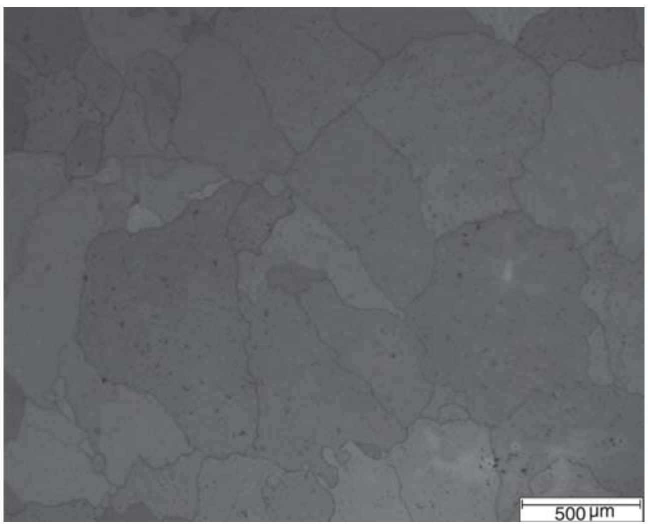

Fig. 5 Microstructure of alloy AZ61 in as-cast condition

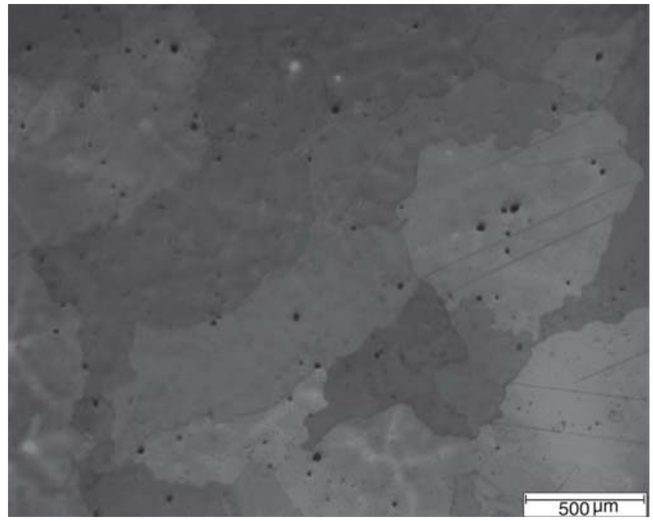

Fig. 6 Microstructure of alloy AZ80 in as-cast condition

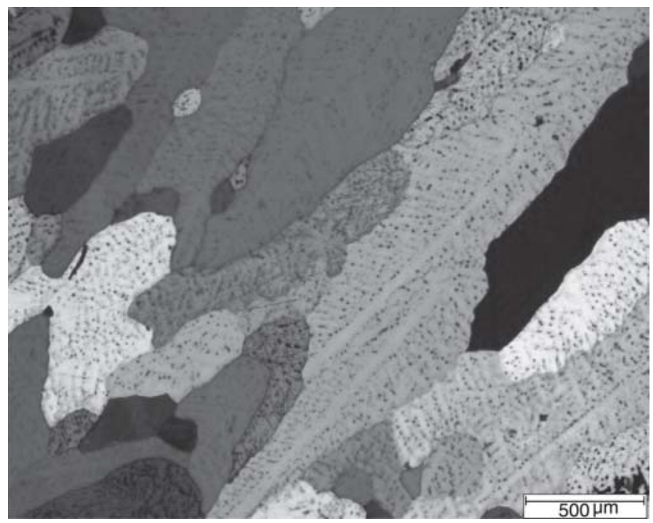

Fig. 7 Microstructure of alloy ZM21 in as-cast condition

Table 1 Parameters of the extrusion trials

Alloy	Chemical composition, wt.%	Extrusion temperature, °C	Billet diameter, mm	Profile diameter, mm	Extrusion ratio	Extrusion rate, m/min
AZ31	Mg-2.9Al-0.98Zn-0.29Mn	300, 200, 100, RT	80	15	28	8
AZ61	Mg-6.5Al-0.99Zn-0.20Mn	100	80	15	28	8
AZ80	Mg-8.5Al-0.51Zn-0.31Mn	110	80	15	28	8
ZM21	Mg-2.0Zn-0.98Mn	100	80	15	28	8
ZK30	Mg-3.0Zn-0.58Zr	100	80	15	28	8
ZE10	Mg-1.2Zn-0.4Zr-0.3RE	100	80	15	28	8

Note: RT, room temperature

mately 6 to 7 μm. With respect to the microstructure of the individual feedstock, the hydrostatic extrusion of the AZ series and ZM21 led to the most effective reduction of the grain size. Therefore, it can be assumed that the performance of this forming process is hardly dependent on the microstructure. Regarding the micrographs of the extruded alloys, it has to be pointed out that the grain sizes of alloys ZE10, AZ31, ZK30, and AZ61 obtained by these hydrostatic extrusion trials are comparable with that received by ECAE pressing (Ref 1).

Figures 19 to 21 show the results of the mechanical properties of the extruded rods that have been received from tensile and compression tests performed at room temperature and with a constant strain rate of $\dot{\varepsilon} = 10^{-3}\ s^{-1}$. In Fig. 19, the ultimate tensile strength (UTS) of the materials is presented for all of the

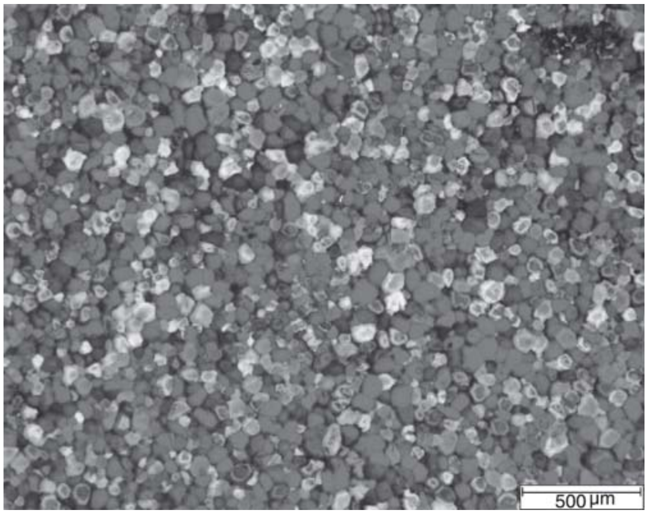

Fig. 8 Microstructure of alloy ZK30 in as-cast condition

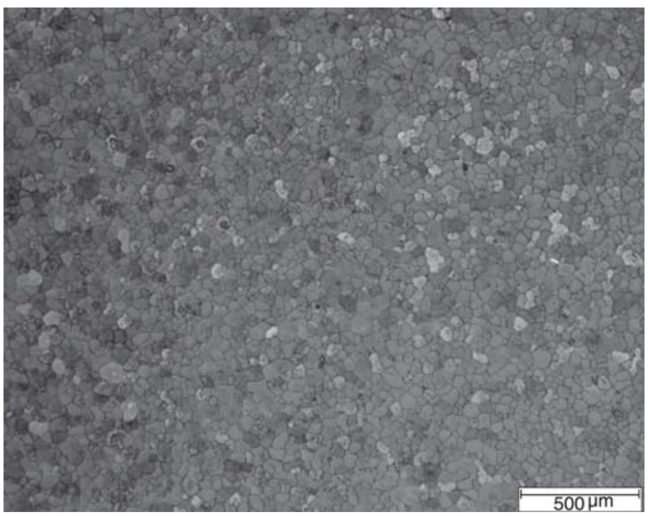

Fig. 9 Microstructure of alloy ZE10 in as-cast condition

Fig. 10 Received extruded profiles and reminders worked at low temperature. The surface of the material is affected neither by hot nor cold cracks. This is also valid for other profile dimensions, which were not the subject of this article.

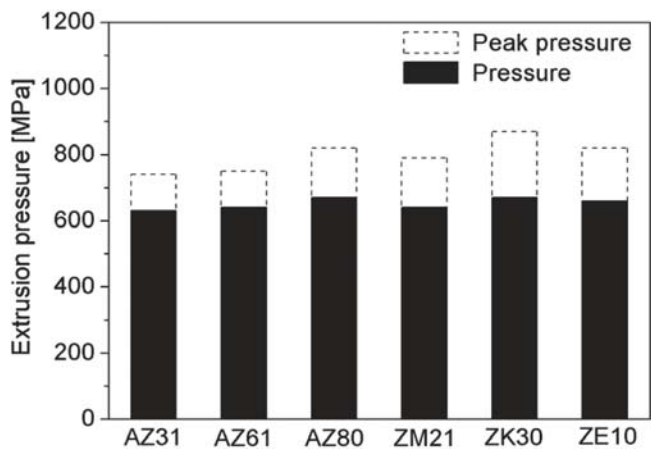

Fig. 11 Extrusion pressure for all extrusion trials at 100 °C (110 °C for alloy AZ80). The peak pressure is defined in Fig. 3.

magnesium alloys used in this study. The UTS of the alloys AZ31, AZ61, and ZK30 is significantly increased by 10 to 25% compared with the data from these alloys that were created by the conventional extrusion processes (Ref 10, 12). Typical UTS data were obtained for the wrought magnesium alloys AZ80, ZM21, and ZE10. With increasing aluminum content, a rise in UTS was measured, which was attributed to solid solution hardening being more effective for a higher fraction of aluminum.

The anisotropy of mechanical properties is always a concern when working with magnesium and limits its application. It is expected that, based on the hexagonal lattice structure of magnesium alloys causing a strongly orientation-dependent deformation, mechanisms would lead to a distinct difference in tensile and compression strength (Ref 19, 20). Therefore, the tensile yield strength (TYS) and compression yield strength (CYS) for each material were measured (Fig. 20). It can be observed that the yield strength of the alloys with small grain sizes tends to result in higher values, which is in agreement with the Hall-Petch relationship (Ref 3). In all of the cases that are illustrated in the present work, it was found that the TYS was similar or only slightly higher compared with the corresponding compressive one. This behavior can be attributed to the absence of twinning being a dominant orientation-dependent deformation mechanism in magnesium alloys. For extremely small grains, twinning seems to become less important (Ref 22), which results in the received reduction of the anisotropy. Only for the alloy ZM21, having the largest grains after extrusion, was a stronger anisotropic behavior measured, although twins did not occur in the associated microstructure (Fig. 15). But, it can be assumed that besides the interaction of grain size and twinning the chemical composition also has an influence on anisotropy because despite the similar grain sizes of the microstructures of alloys ZE10, AZ31, and ZK30, the TYS and CYS are different. In Fig. 21, the elongation to fracture is presented for each investigated material. The highest elongation to fracture data were measured for alloys ZE10, AZ31, and ZK30, showing the smallest grain sizes of all of the alloys in this study. This mechanical property is also dominated by the grain size of the extruded rods.

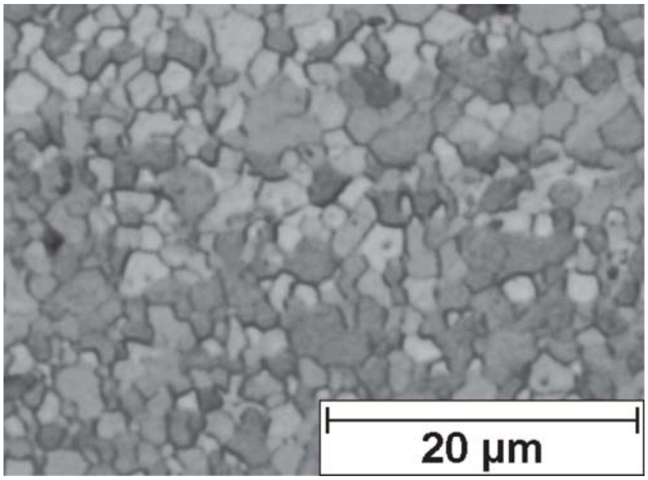

Fig. 12 Microstructure of alloy AZ31 extruded at 100 °C (extrusion direction →)

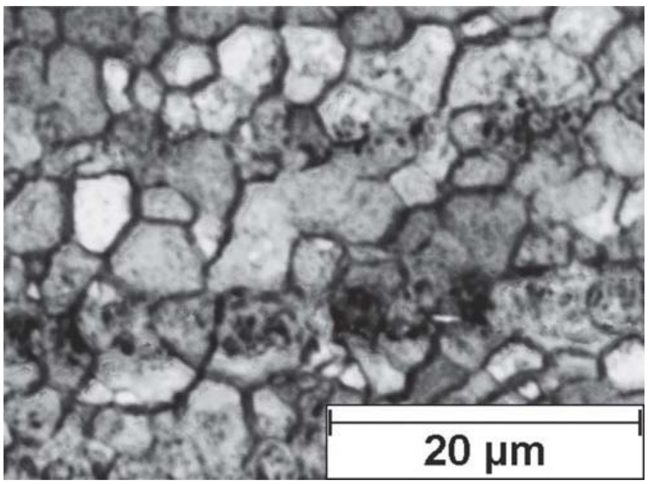

Fig. 13 Microstructure of alloy AZ61 extruded at 100 °C (extrusion direction →)

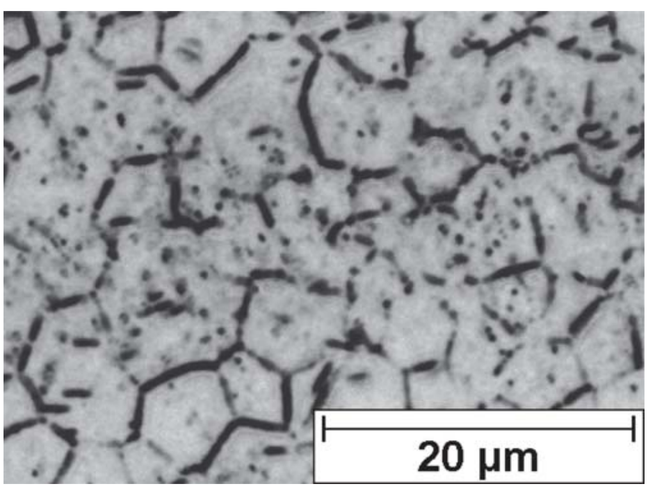

Fig. 14 Microstructure of alloy AZ80 extruded at 110 °C (extrusion direction →)

It has to be emphasized that the hydrostatic extrusion of all alloys with an extrusion rate of 8 m/min was successful and led to similar or even improved mechanical proper-

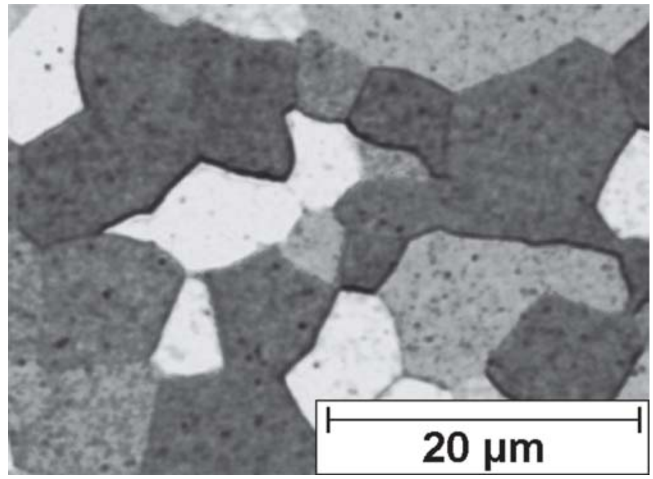

Fig. 15 Microstructure of alloy ZM21 extruded at 100 °C (extrusion direction →)

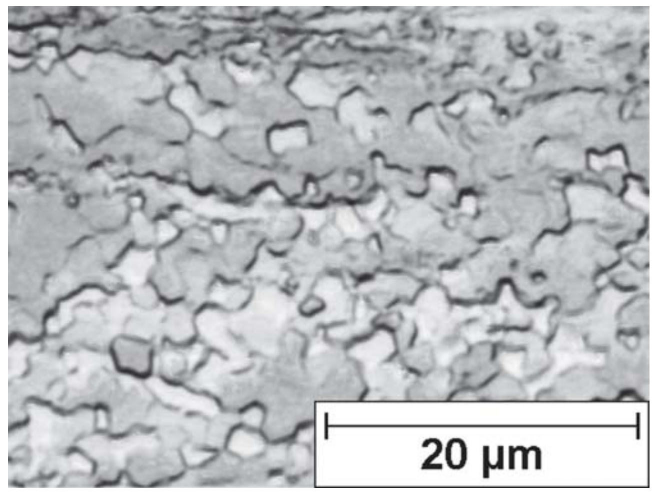

Fig. 16 Microstructure of alloy ZK30 extruded at 100 °C (extrusion direction →)

ties. Especially for the magnesium wrought alloy AZ80, an extrusion rate that was fourfold higher than that for conventional processing (Ref 17) could be received without any problems.

4. Summary

The forming of magnesium alloys at a temperature below 200 °C is considered to be extremely unproductive due to the insufficient number of activated gliding systems and is avoided in conventional technical forming processes (e.g., rolling, direct extrusion, and forging). Also, other forming techniques like ECAE pressing are performed at higher temperatures.

The behavior of magnesium wrought alloys during hydrostatic extrusion was investigated, and showed that the hydrostatic process offers the possibility of decreasing the extrusion temperature to 100 °C for alloys AZ31, AZ61, ZM21, ZK30, and ZE10, and to 110 °C for alloy AZ80.

An extremely fine-grained microstructure, excellent ductility, as well as enhanced yield strength of the investigated

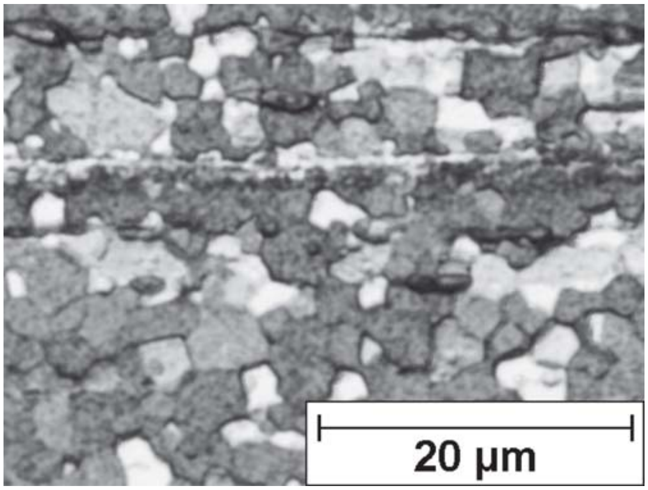

Fig. 17 Microstructure of alloy ZE10 extruded at 100 °C (extrusion direction →)

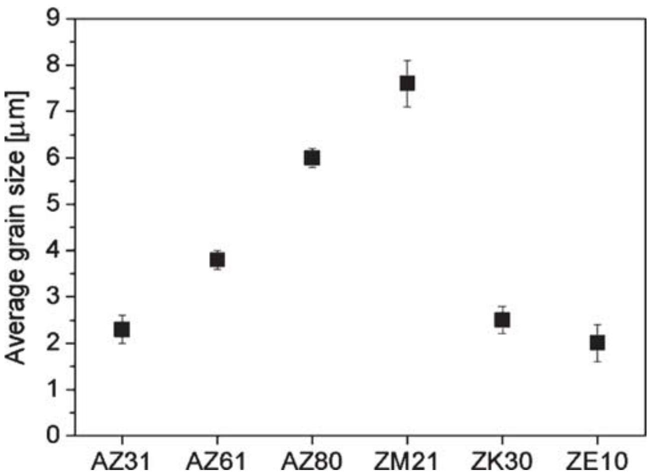

Fig. 18 Average grain size after extrusion at 100 °C (110 °C for alloy AZ80)

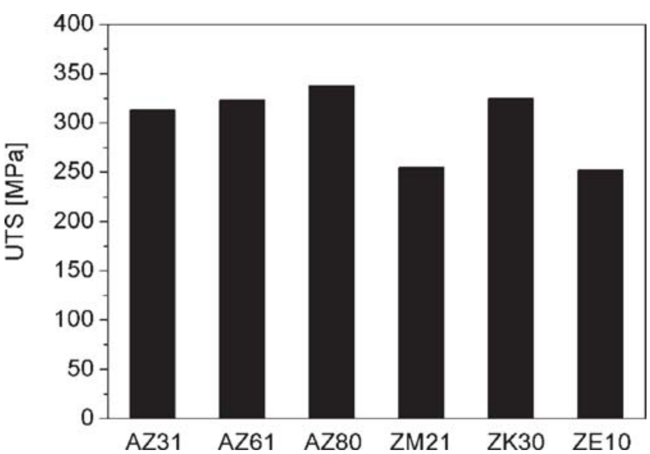

Fig. 19 Comparison of UTS during hydrostatic extrusion at 100 °C

magnesium wrought alloys could be achieved by this extrusion method. Also, the expected difference of yield strength and CYS (anisotropy) was significantly reduced after extrusion at 100 °C. With respect to the alloys used in this study, the

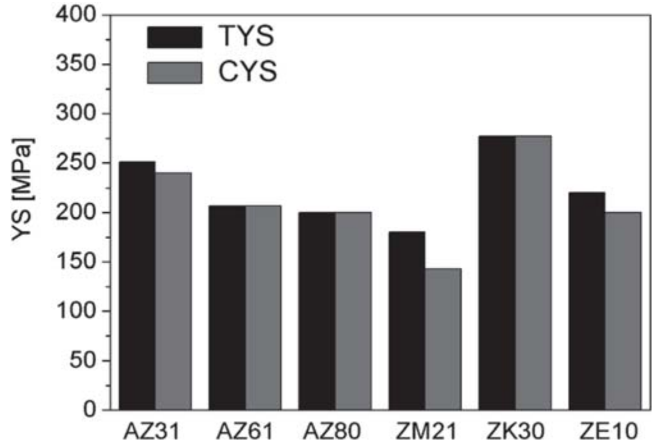

Fig. 20 Comparison of TYS and CYS during hydrostatic extrusion at 100 °C

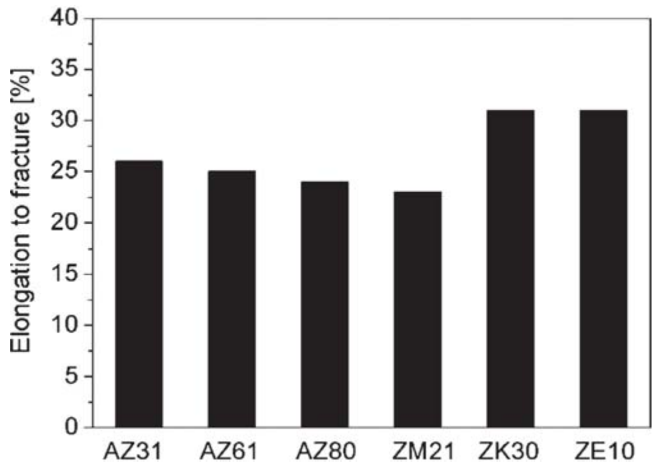

Fig. 21 Comparison of elongation to fracture during hydrostatic extrusion at 100 °C

wrought magnesium alloys AZ31, AZ61, ZK30, and ZE10 are of special interest for low-temperature hydrostatic extrusion. Further investigations at different extrusion parameters are necessary to verify the assumptions according the relationship between microstructure and the resulting mechanical properties.

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References

1. H. Watanabe, T. Mukai, K. Ishikawa, and K. Higashi, Low Temperature Superplasticity of Fine Grained ZK60 Magnesium Alloy Processed by Equal-Channel-Angular Extrusion, *Scripta Mater.,* 2002, **46,** p 851-856
2. A.M. Galiyev, R.O. Kaibyshev, and G. Gottstein, Grain Refinement of ZK60 Magnesium Alloy During Low Temperature Deformation, *Magnesium Technology 2002*, *TMS Annual Meeting,* H.I. Kaplan, Ed., February 17-21, 2002 (Seattle, WA), The Materials Society, 2002, p 181-185
3. C. Sanchez, G. Nussbaum, P. Azavant, and H. Octor, Elevated Temperature Behaviour of Rapidly Solidified Magnesium Alloys Containing Rare Earths, *Mater. Sci. Eng., A,* 1996, **221,** p 48-57

4. K. Savage, J.F. King, and A. van Kooij, Hydrostatic Extrusion of Magnesium, *Magnesium Alloys and Their Applications,* K.U. Kainer, Ed., Wiley-VCH, Weinheim, Germany, 2000, p 609-614
5. J. Bohlen, J. Swiostek, W.H. Sillekens, P.J. Vet, D. Letzig, and K.U. Kainer, Process and Alloy Development for Hydrostatic Extrusion of Magnesium, *Magnesium Technology 2005, TMS Annual Meeting,* N.R. Neelameggham, H.I. Kaplan, and B.R. Powell, Ed., February 13-17, 2005 (San Francisco, CA), The Metals Society, 2005, p 241-247
6. A.J. Den Bakker, W.H. Sillekens, J. Bohlen, K.U. Kainer, and G. Barton, The Magnextrusco Project: Process and Alloy Development for Hydrostatic Extrusion of Magnesium, *Magnesium Alloys and Their Applications,* K.U. Kainer, Ed., Wiley-VCH, Weinheim, Germany, 2003, p 324-330
7. J. Göken, J. Swiostek, D. Letzig, and K.U. Kainer, Damping Measurements of the Magnesium Wrought Alloys AZ31, AZ61 and AZ80 after Indirect and Hydrostatic Extrusion, *Materer. Sci. Forum,* 2005, **482,** p 387-390
8. A. Beck, *Magnesium und seine Legierungen,* Julius Springer Verlag, Berlin, 1939, in German
9. E.F. Emley, *Principles of Magnesium Technology,* Pergamon Press, Oxford, 1966
10. K.B. Müller, Direct and Indirect Extrusion of AZ31, *Magnesium Technology 2002, TMS Annual Meeting,* H.I. Kaplan, Ed., February 17-21, 2002 (Seattle, WA), The Materials Society, 2002, p 187-192
11. N. Ogawa, M. Shiomi, and K. Osakada, Forming Limit of Magnesium Alloys at Elevated Temperatures for Precision Forging, *Int. J. Machine Tools Manufact.,* 2002, **42,** p 607-614
12. M.M. Avedesian and H. Baker, Ed., *ASM Specialty Handbook, Magnesium and Magnesium Alloys,* ASM International, 1999
13. J. Bohlen, F. Chmelik, P. Dobron, and F. Kaiser, Orientation Effect on Acoustic Emission During Tensile Deformation of Hot Rolled Magnesium Alloy AZ31, *J. Alloys Compd.,* 2004, **378,** p 207-213
14. J. Swiostek, J. Bohlen, D. Letzig, and K.U. Kainer, Comparison of Microstructure and Mechanical Properties of Indirect and Hydrostatic Extruded Magnesium Alloys, *Magnesium Alloys and Their Applications,* K.U. Kainer, Ed., Wiley-VCH, Weinheim, Germany, 2003, p 278-284
15. J. Chapman and D.V. Wilson, The Room-Temperature Ductility of Fine-Grain Magnesium, *J. Inst. Met.,* 1962-1963, **91,** p 39-40
16. K. Müller, *Grundlagen des Strangpressens,* Expert-Verlag, 2003, in German
17. G. Fischer, *Documentation of the Conference* "*Magnesium im Fahrzeugbau,*" IIR Deutschland GmbH, Sindelfingen Germany, 2003
18. V. Kree, J. Bohlen, D. Letzig, and K.U. Kainer, The Metallographical Examination of Magnesium Alloys, *Pract. Metallogr.,* 2004, **5,** p 233-246
19. C.H.J. Davies, F. Xiong, and M. Battaini, Anisotropic Mechanical Properties of Extruded Magnesium Alloy AZ31, *Magnesium Alloys and Their Applications,* K.U. Kainer, Ed., Wiley-VCH, Weinheim, Germany, 2003, p 433-438
20. P.G. Partrige, The Crystallography and Deformation Modes of Hexagonal Close-Packed Metals, *Met. Mater.,* 1967, **1,** p 169-194
21. W.H. Sillekens, J.A.F.M. Schade van Westrum, A.J. den Bakker, and P.J. Vet, Hydrostatic Extrusion of Magnesium: Process Mechanics and Performance, *Mater. Sci. Forum Vols.,* 2003, **426-432,** p 629-636
22. M.R. Barnett, Z. Keshavarz, A.G. Beer, and D. Atwell, Influence of Grain Size on the Compressive Deformation of Wrought Mg-3A1-1Zn, *Acta Mater.,* 2004, **52,** p 5093-5103