Variation of Extrusion Process Parameter for the Magnesium Alloy ME21

G. Kurz, M. Nienaber, J. Bohlen, D. Letzig, and K. U. Kainer

Abstract

Extrusion is an economic production process for the generation of semi-finished magnesium products that can be used for biomedical and automotive applications. This paper reports on the variation of process parameters (temperature and extrusion speed) in the aluminum-free magnesium alloy ME21 (Mg–2Mn–0, 6Ce–0,3) during extrusion in order to investigate their influence on strength and ductility of the produced profiles. The influence of the varied process parameters on the microstructure before and after heat treatment is shown. Furthermore, the mechanical properties of the extruded profiles are presented and discussed with respect to arising textures. The results of this work are used to discuss how to tailor the mechanical properties of the magnesium alloy ME21 during the extrusion process.

Keywords

Magnesium • Extrusion • Aluminum-free • Magnesium sheet • Rare earth-containing alloys

Introduction

In order to exploit the full potential of magnesium as a lightweight material, the use of thin-walled components, such as extruded flat profiles, is indispensable. The funding project for **Fu**nction-integrated **Ma**gnesium Lightweight Construction for Car **S**eat Structures (FUMAS), funded by the German Federal Ministry of Economics and Energy (BMWi), makes it possible for the first time to use magnesium in large quantities in highly stressed seat structures by the combination of extrusion and forming processes as well as by the implementation of new design concepts. The project covers the development of a material adapted design and the production process. The production process includes the extrusion and the forming process, joining, and the coating for corrosion protection. Finally, it is planned to manufacture a prototype seat and test the seat in a crash test. One workpackage of the Magnesium Innovation Centre (MagIC) is the development of a robust and reproducible forming process. The work in this funding project is aimed at reducing the weight of a vehicle seat structure by using extruded magnesium parts in the seat back. Vehicle seats are among the most mechanically stressed components of the vehicle interior and therefore make a considerable contribution to the vehicle mass. Magnesium parts have so far been cast into small production volumes in the seat area. Weight saving potentials of 30–40% compared to a steel reference structure were shown. Compared to magnesium casting alloys, wrought magnesium alloys exhibit higher mechanical strength and elongation at fracture. The simple substitution of steel components by magnesium components, however, fails on the one hand due to the lower strength of magnesium and on the other hand due to suitable joining and corrosion protection solutions.

This paper shows the results of the trials for the optimization of the extrusion process. From the company TWI commercially available magnesium alloy ME21 (2 wt% manganese and up to 1 wt% of rare earth elements) was chosen to realize the seat back component, because of its good hot strength and hot workability compared to other magnesium alloys [1]. The alloying element manganese is contained in small quantities in many technical magnesium alloys to increase corrosion resistance. In magnesium alloys, manganese forms stable intermetallic phases in which impurities such as iron or silicon are bound. In addition, manganese increases tensile strength, improves casting behaviour, and leads to a grain refining effect [2]. Another reason for choosing this alloy is the absence of a low melting eutectic in contrast to aluminium-containing alloys. During extrusion, manganese provides a large process window and

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allows relatively high extrusion speeds [1, 3], which makes this alloy economically interesting.

Typically, rare earth metals are used as cerium mischmetal (approx. 60% cerium, approx. 30% lanthanum, rest neodymium, praseodymium) as alloying additive. Rare earths increase the high-temperature strength and creep resistance [2]. In addition, studies show that the addition of rare earths has increased the activation of non-basal slip systems during the rolling process of binary magnesium alloys, which leads in combination with recrystallisation to a weakening of the basal texture component [4]. The effect of texture weakening of the basal component was also observed in extruded rare earth-containing magnesium alloys [1, 5]. Rolling tests with the alloy ME21 have shown that the mechanical properties do not change significantly depending on the RE content [6].

Experimental Procedure

The aim of the investigations for the FUMAS project at the MagIC is to show a process window in which the seat back of a car seat after the manufacturing process has a fine-grained, globular, and homogeneous microstructure with corresponding precipitation behaviour. In order to investigate the influence of the extrusion parameters such as billet temperature 300 °C $\leq T_B \leq$ 450 °C and extrusion speed 0.75 mm/s $\leq v_P \leq$ 7.5 mm/s which was varied, see Table 1. The extrusion speed is in all described trials the ram velocity.

The company TWI, a commercial magnesium supplier, delivered cast feedstock material used for the extrusion trials. All delivered billets were heat-treated to homogenize the microstructure and to reduce the amount of precipitates. The samples for the microstructure and EDX analyses were taken from the two half slices of the billets top. For the extrusion trials, the billets were machined to four smaller billets with 49 mm in diameter and approx. 130 mm in length. The machined billets were pressed on a 2.5 MN extrusion press, built by the company Müller, with an extrusion ratio of 49 : 1 in a direct extrusion process to strips of 20 mm in width and 2 mm in thickness. Graphite was used as lubricant. After the extrusion tests, the influence of the grain size and the heat treatment condition of the starting material on the extrusion behavior, the microstructure, the texture, and the mechanical properties of the extruded flat profiles were analyzed. In order to guarantee that sample was only taken from sections that experience homogeneous material flow, the first and last 1500 mm of the extrusion profile were not used. To compare the microstructure and texture development along the profile length, 20 mm sections were taken for each sample at the beginning (1500 mm), at the center (3000 mm), and at the end of the resulting strips (4500 mm). For microstructure analysis, standard metallographic sample preparation techniques were applied and an etchant based on picric acid was used to reveal grains and grain boundaries [7]. Texture measurements were done on the strip mid-planes using a Panalytical X-ray diffractometer setup. The pole figures were measured up to a tilt angle of 70° which allowed recalculation of full pole figures based on a MTEX software routine [8]. The (0001) and (10-10) pole figures of the sheets in as rolled and heat-treated condition are used in this work to present the texture of the strips at midplane. The mechanical parameters required 30 cm of the strip and the three dog bone-shaped tensile specimens with a measuring length of 18 mm were separated in the extrusion direction by wire erosion. The mechanical properties of the extruded strips were investigated by tensile tests with a constant initial strain rate of 1.0×10^{-3} s^{-1}.

Characterization of the Feedstock Material

The company TWI provided the ME21 material for the extrusion tests and six continuous cast sections. For chemical composition, see Table 2.

In contrast to typically RE-containing magnesium alloys, no cerium -based mischmetal was used as usual but only cerium and lanthanum. The delivered ME21 billets are characterized by a different macrostructure. Figure 1 shows the macrostructure and microstructure of three representative billets. This ranges from an extremely inhomogeneous coarse-grained structure with large elongated grains with an average grain size of 10^{-2} mm to a globular homogeneous fine-grained grain structure with an average grain size of around 2 µm. Because the manufacturer could not provide any process parameters, it can only be assumed that the differences in microstructure are due to varying holding times of the melt (nucleation leads to a finer microstructure) or different cooling rates of the billets (grain growth can be inhibited). A chemical influence on the macrostructure

Table 1 Process parameter of the extrusion trials

Extrusion temperature, T_B (°C)	Extrusion speed, v_P (mm/s)	Billet condition
450	0,75; 1,4; 2,8; 5,5; 7,5	Heat-treated at 500 °C for 8 h
400	0,75; 1,4; 2,8; 5,5; 7,5	Heat-treated at 500 °C for 8 h
350	0,75; 1,4; 2,8; 5,5; 7,5	Heat-treated at 500 °C for 8 h
300	1,4; 2,8; 5,5; 7,5	Heat-treated at 500 °C for 8 h

Table 2 Chemical composition of the billets in wt%

Specimen	Mn	Ce	La	Fe	Cu	Ni
Section 1	1,78	0,549	0,345	0,00389	0,00202	0,00098
Section 2	2,18	0,555	0,360	0,00064	0,00231	0,00103
Section 3	2,07	0,546	0,353	0,00030	0,00229	0,00110
Section 4	1,98	0,587	0,382	0,00150	0,00222	0,00108
Section 5	1,97	0,591	0,384	0,00125	0,00217	0,00113
Section 6	1,81	0,507	0,362	0,00223	0,00208	0,00119

development can be excluded, because all billets have with small tolerance the same chemical composition.

All microstructures are characterized by an inhomogeneous dendritic microstructure because of the multiphase microstructure. Furthermore, all billets have a fine-grained edge zone as a result of the faster solidification at the mould wall. The microstructures also show a very high amount of precipitates, which is due to the low dissolubility of manganese, cerium, and lanthanum in the magnesium solid solution.

The subsequent heat treatment should reduce internal stresses and achieve homogenization or partial solution of the precipitation phases. Figure 1 compares selected areas of the microstructure of three selected billets in the cast and heat-treated condition. The 8-h heat treatment at 500 °C resulted in a partial solution but also a coarsening of the precipitates between the dendrite arms. The coarse Mn precipitates are not evidently dissolved in the microstructure by the heat treatment.

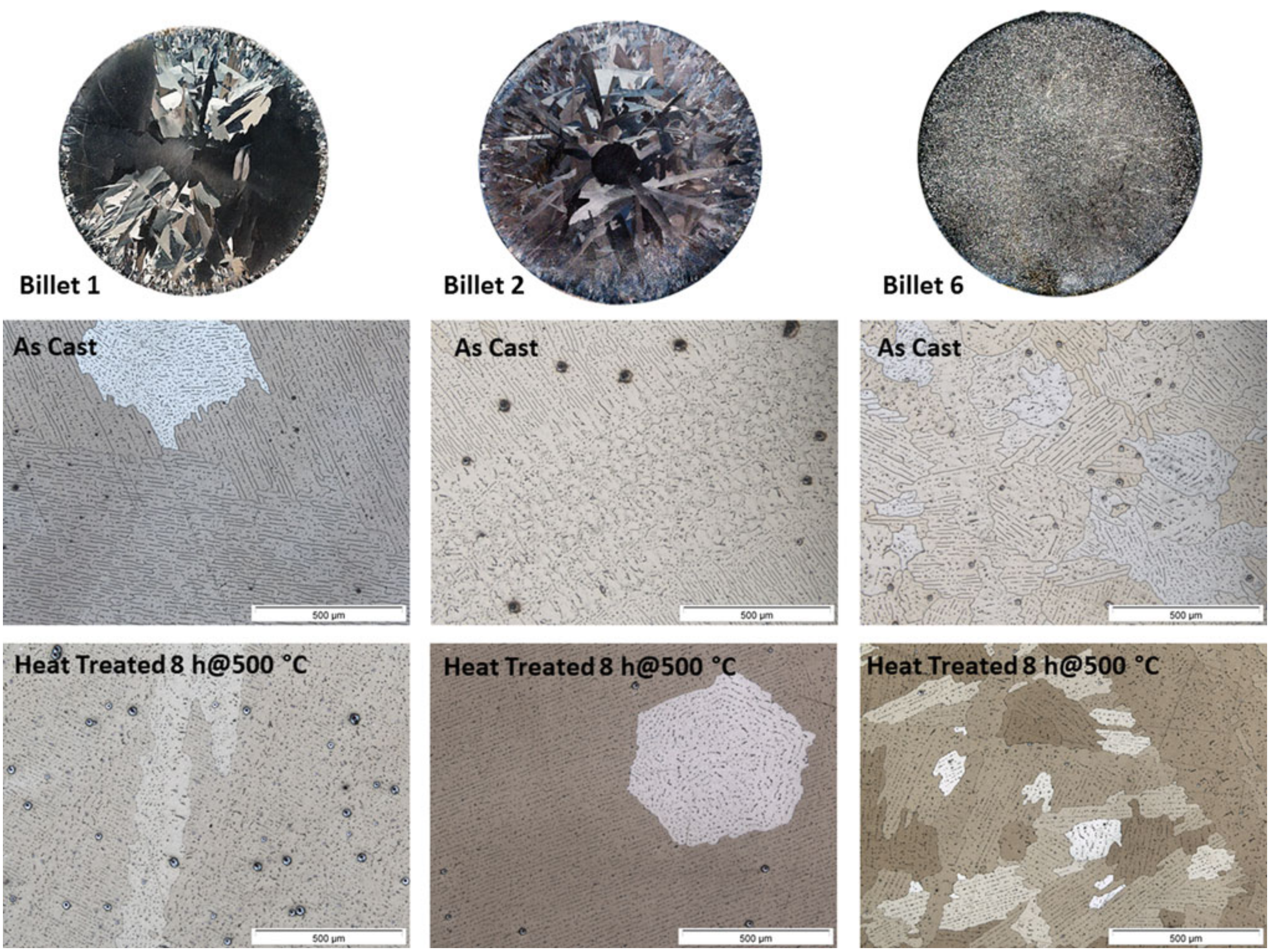


Fig. 1 Macrostructure and microstructure of three representative billets in as-cast and heat-treated condition

Influence of the Feedstock Properties

As shown in the previous paragraph, the billets of the starting material have a very different macrostructure. Therefore, it was first examined how the macrostructure affects the extrusion result. To do this, a billet with a diameter of 49 mm was selected from the initial billets 1, 2, and 6 in the cast and heat-treated condition. These billets were extruded at a temperature of 400 °C and a punch speed of 0.75 mm/s. Figure 2 shows the extrusion diagrams and punch pressure versus strip length. All curves show the typical shape for the direct extrusion process. As the profile length increases, the pressing pressure decreases as the friction force decreases as the billet length decreases. The billets in the cast condition show a very inhomogeneous plastic flow and require a higher maximum pressure than the billets in the heat-treated condition (Fig. 2). Because in the as-cast condition the precipitates Mg_{12}(Ce, La) are mainly located at the grain boundaries (Fig. 1), this could be the reason for the higher pressure. The heat-treated billet could be extruded with a more homogeneous plastic flow and lower punch pressure. It can be assumed that the heat treatment relieves internal stresses and allows the material to flow more easily and homogeneously. Due to the heat treatment, the precipitates are strongly agglomerated and were no longer so dominant located at the grain boundaries (Fig. 1). Figure 2 displays also the microstructures of the extruded strips. It can be clearly seen that the differences in the microstructure of the three strips extruded out of the heat-treated billets are marginal. In the microstructure of all three strips, it can be observed that the material has a homogeneous microstructure. Globular bimodal grain structures are clearly visible here. In some cases, it also has grains that are longitudinally stretched along the extrusion direction. The average grain size of the three extruded profiles varies between 5.9 and 6.5 µm. The precipitates also lie horizontally in the microstructure. Very large precipitates are visible, which are probably the (Mn) particles detected in the casting material. As a result, in the heat-treated condition, the macrostructure and the grain size have no significant influence on the extrusion result. So, it has been demonstrated that heat treatment can significantly improve extrusion performance.

Influence of the Extrusion Temperature and Speed on Microstructure

In order to investigate the influence of process parameters speed and temperature extrusion trials with in Table 1 listed parameters have been performed. At 350 °C and a punch speed of 0.75 mm/s, a process limit has been reached, because the material flows inhomogeneous. Consequently, in the extrusion tests at 300 °C, the test with a punch speed of 0.75 mm/s was not carried out. An inhomogeneous material flow was also observed at 300 °C and 1.4 mm/s, but

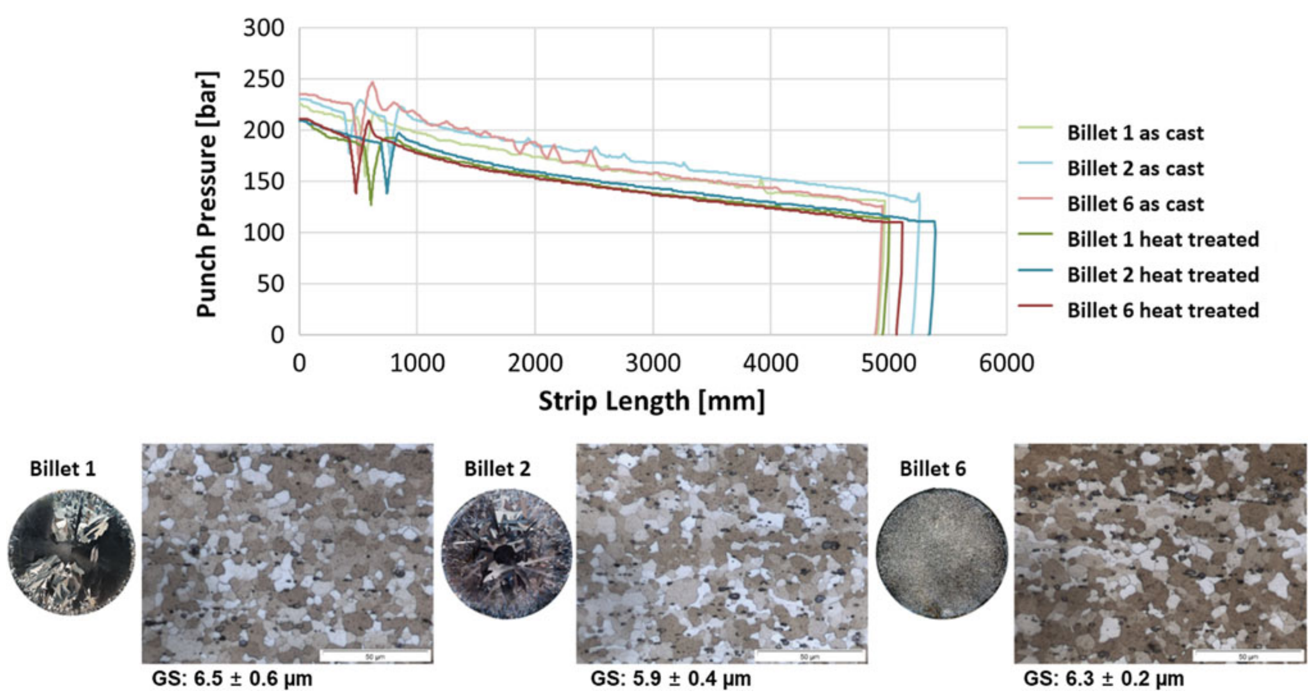


Fig. 2 Extrusion diagrams of billet 1, 2, and 6 pressed at a temperature of 400 °C and a punch speed of 0.75 mm/s and the resulting microstructure of the extruded strips

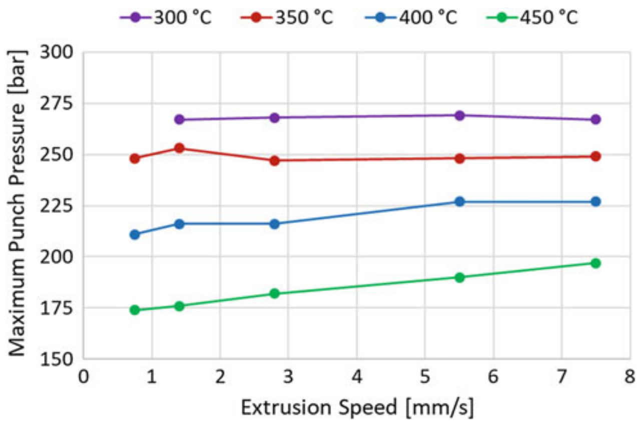


Fig. 3 Maximum punch pressure versus extrusion speed of all extrusion trials of ME21

this inhomogeneous flow was not as pronounced as at 350 °C and 0.75 mm/s. For all trials, the punch pressure versus strip length was recorded. All extrusion diagrams show the typical curves mentioned above for direct extrusion. Figure 3 shows the maximum punch pressures as a function of the punch speed. The punch pressure drops with rising temperature, because the strength of the magnesium alloy ME21 decreases with higher temperature. The maximum punch pressure increases again with increasing punch speeds.

The microstructures shown in Fig. 4 were taken in the middle (300 cm) of the profiles, because the microstructures do not show any significant differences depending on the position where they were taken. The average grain size was determined by the line intersection method on three polarized images at 1000x magnification. To demonstrate the tendency of all results, only the results of the extruded profiles at 350 and 450 °C are presented in the following. Figure 4 shows the microstructures for the process temperatures of 350 and 450 °C as a function of the punch speed. After extrusion, all profiles extruded have a fine-grained completely recrystallized structure. The only exception is the microstructure of the strip extruded at 350 °C at the slowest speed of 0.75 mm/s; here is the microstructure only partly recrystallized. As a reason for this, it can be assumed that the temperature of 350 °C and the forming energy introduced at the extrusion speed of 0.75 mm/s do not allow dynamic recrystallization processes to take place completely. Depending on the extrusion speed, a clear influence of the billet temperature on the grain growth can be seen. In general, it can be observed that grain growth can be observed with increasing temperature. All microstructures exhibit a high density of precipitates, which are predominantly distributed horizontally in the direction of extrusion.

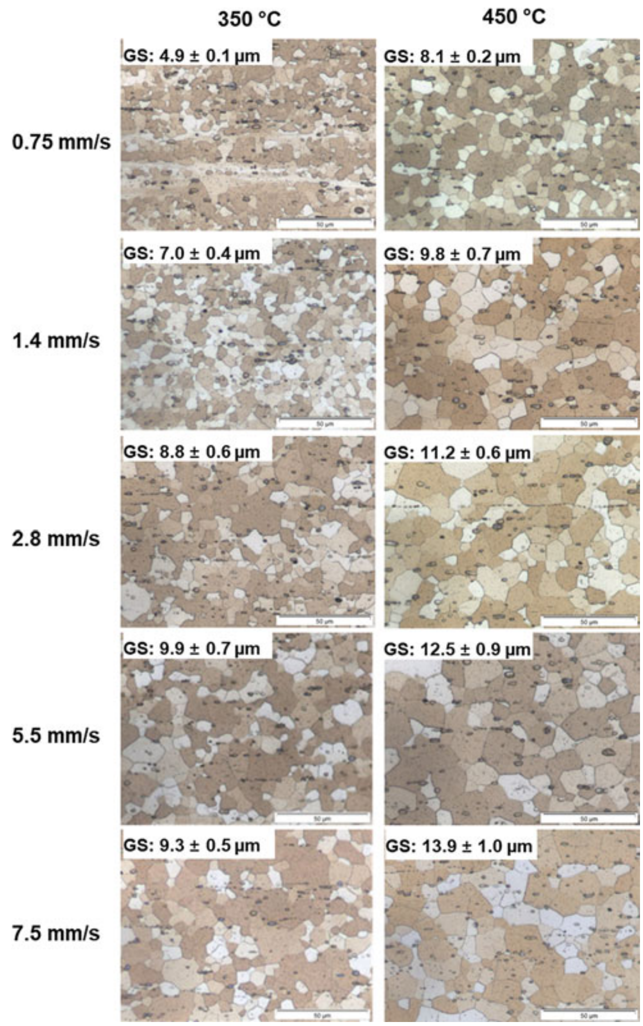


Fig. 4 Microstructures of the ME21 strips extruded at 350 and 450 °C

The microstructure develops, depending on the extrusion parameters, temperature, and speed, from partially recrystallized to fine-grained to bimodal and finally to a coarse-grained microstructure. Despite the wide process window, the mean grain size varied only from 4 µm (300 °C and 1.4 mm/s) to 14 µm (450 °C and 7.5 mm/s). As already mentioned, the strongly suppressed grain growth can be attributed to the alloying of rare earths. Due to the rare earths, the grain boundary mobility is changed and restricted and consequently the grains can only grow to a limited extent [9]. The higher the applied deformation (low extrusion temperature and speed), the smaller the grain size. Because the energy is lowest at low temperature and speed, the dynamic recrystallization during the extrusion process and the static recrystallization during the cooling process can only take place to a limited extent. Due to the crystal structure, certain grain fractions can grow better, which leads to the formation of the bimodal microstructure.

Influence of the Extrusion Temperature and Speed on Texture

Figure 5 displays the (10-10) and (0002) pole figures of the profiles extruded at 450 and 350 °C. The extrusion tests carried out at 450 °C in Fig. 6 all show a very similar texture. Only the maximum pole figures' intensities vary. Basically, the pole figures show a strong basal (0002) component, which is tilted by approx. 25–30° from the normal plane into the opposite extrusion direction. As the extrusion speed increases, the maximum pole figure intensity increases from 8.8 to 12 m.r.d.

With increasing extrusion speed also a widening of the {11-20} <10-10> by 30° from transverse to normal direction tilted component is recognizable. This component correlates with the maximum intensity in the (10-10) pole figure. Here, the maximum intensity increases from 2.4 to 4.4 m.r.d. All textures show (10-10) fiber components in extrusion direction, which become more pronounced or rather more complete with increasing extrusion speed.

Figure 6 also shows the textures for the extrusion tests at 350 °C. In the maximum intensities of the (0002) pole figures, there is no significant influence of the punch speed. It fluctuates between 6.4 and 7.0 m.r.d. It is concise that from a extrusion speed of 5.5 mm/s no {0001} <10-10> component deflected by 25° in the transverse direction is visible. All textures show a <10-10> extrusion direction fiber; in addition, a component is tilted by 30° from transverse in normal direction with varying intensity {11-20} <10-10> . The maximum intensity of this component, i.e., the (10-10) pole figure, increases from 6.8 to 10.0 m.r.d. as the extrusion speed increases. In ME20 sheets [10] and extruded ME21 thin strips [11], which have a very similar alloy composition to the ME21 alloy used in this paper, textures were measured

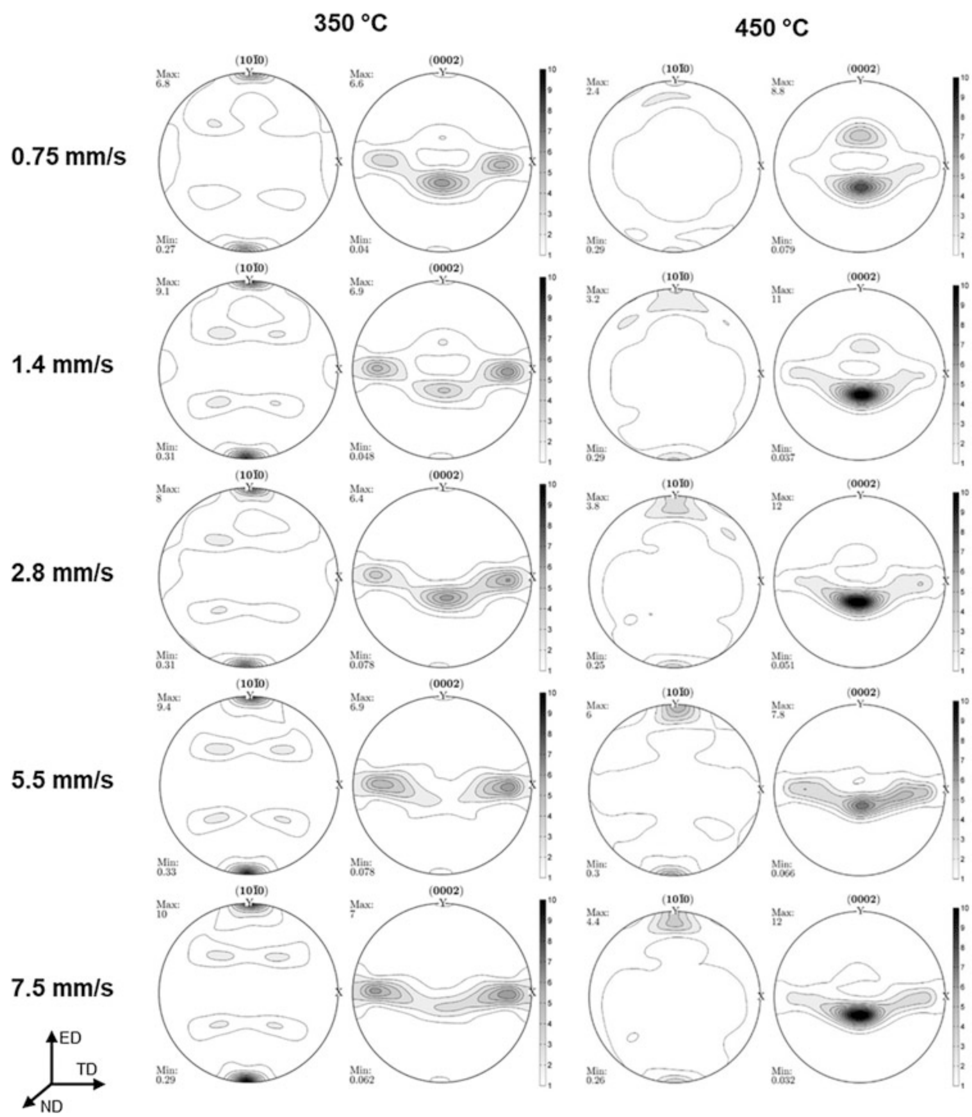
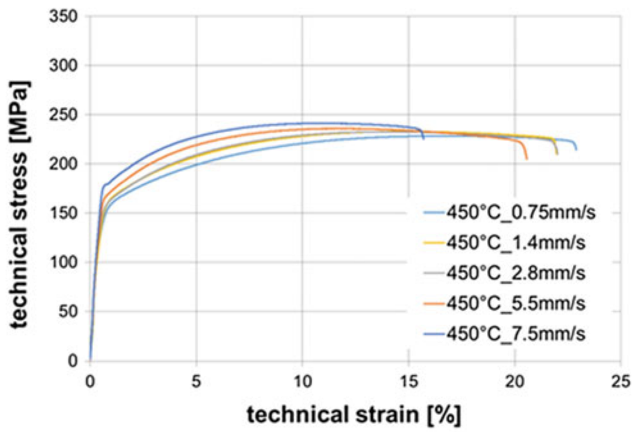
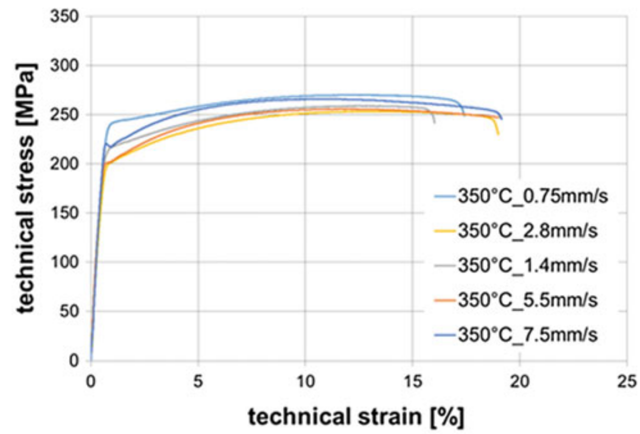


Fig. 5 Textures of the ME21 strips extruded at 350 and 450 °C





v_P [mm/s]	TYS [MPa]	UTS [MPa]	E [%]
0,75	155 ± 0,6	229 ± 0,3	21,5 ± 0,7
1,4	159 ± 0,4	233 ± 0,2	21,2 ± 0,2
2,8	158 ± 0,1	233 ± 0,1	20,5 ± 1,1
5,5	167 ± 0,3	235 ± 0,4	19,6 ± 0,9
7,5	180 ± 0,4	241 ± 0,2	14,9 ± 1,1

v_P [mm/s]	TYS [MPa]	UTS [MPa]	E [%]
0,75	238 ± 2,1	270 ± 1,6	16,5 ± 0,5
1,4	213 ± 2,2	259 ± 1,7	15,3 ± 0,3
2,8	200 ± 0,3	254 ± 0,7	17,9 ± 0,2
5,5	199 ± 1,6	255 ± 1,1	19,8 ± 1,0
7,5	220 ± 1,6	266 ± 0,5	19,0 ± 1,1

Fig. 6 Stress–strain diagrams of the ME21 strips extruded at 450 and 350 °C and the values of tensile yield stress (TYS), the ultimate tensile stress (UTS), and fracture strain (E)

which also have a strongly pronounced {0001} <10-10> component of similar shape tilted by 25° in rolling/extrusion direction. The three slip systems <a> basal, <a> prismatic, and <c+a> pyramidal and the twin systems are necessary for the initiation of deformation in polycrystalline materials.

An additional reason for the formation of this texture component may be the different influence of dynamic recrystallization on grain growth in rare earth-containing magnesium alloys.

Influence of the Extrusion Temperature and Speed on Mechanical Properties

The stress–strain diagrams obtained from the tensile tests performed at room temperature are exemplary shown for the strips extruded at 450 and 350 °C in Fig. 6. The averaged values of the measured variables and their standard deviation are shown in Fig. 6. Depending on the temperature, various tendencies can be observed as to how the mechanical properties change as the grain size increases. At 450 °C, an increase in the process speed, resulting in a coarsening of the grains, a decrease in elongation at break from 21.5 to 14.9%, an increase in yield strength by 25 MPa to 180 MPa, and an increase in strength by 13 MPa to 241 MPa are shown.

In contrast to the mechanical properties of the strips extruded at 450 °C, the mechanical properties resulting from the at 350 °C extruded strips have no tendency in the measured values depending on the extrusion parameters. It is noteworthy that at a punch speed of 0.75 mm/s the curve has only a slightly pronounced convex slope. At the moderate punch speeds (1.4–5.5 mm/s), the maximum strength varies between 253 and 259 MPa and the tensile yield stress between 212 and 200 MPa. The elongation increases by 5% to almost 20%. A higher yield strength (220 MPa) and strength (265 MPa) are observed by the test at 7.5 mm/s.

Compared to mechanical properties of the at 450 °C extruded strips, the results of the strips extruded at 350 °C suggest that grain size is not always the dominant factor in plastic deformation. Also the texture, i.e., the inclination of the c-axis, correlates with the possibility of plastic deformation. At 450 °C the dominant texture component is the {0001} <10-10> component tilted 25° in the extrusion direction. When applying a load in the extrusion direction, basal <a> sliding systems can be activated very easily. The background is Schmid's shear stress law, which describes the critical shear stress required to move the dislocations on the most densely packed planes. In the optimum case, the planes are below 45° to the load direction. An explicit alignment of the preferred orientation along the load direction, i.e., a high maximum intensity of the {0001} <10-10> by 25° in extrusion direction, increasingly favors the deformation process and results in low strength or high elongation at fracture along the alignment [12].

Summary

The results of the extrusion trials for the FUMAS project reveal that the process parameters temperature and speed have not a very significant influence on the resulting ME21 strip properties. Heat treatment of the billets prior to extrusion greatly improves extrusion performance. Because of this reason, in the heat-treated condition, the macrostructure and the grain size have no significant influence on the extrusion result. There is a temperature and a partial speed influence on the flow behaviour and the maximum pressing pressures during extrusion. The variation of the process parameters in the investigated process window indicates a small influence on the grain size (5–14 µm) and no detectable influence on the morphology of the precipitates. Partial grain growth leads to a bimodal structure with increasing temperature and/or speed. With regard to the resulting texture, changes can be observed as a function of temperature. At higher temperatures, a strongly pronounced {0001} <10-10> component appears, which is tilted by 25° in extrusion direction; at lower temperatures, the texture dominates a {11-20} <10-10> component, which is tilted by 30° from transverse in normal direction. In general, the maximum intensity of the texture increases with increasing speed.

Looking at the mechanical properties of all strips, it can be seen that despite the large process window, the variation in properties is relatively small. It is also remarkable that the yield strength and tensile strength are lowest at 450 °C and 0.75 mm/s with TYS = 154 MPa and UTS = 228 MPa and highest at 300 °C and 1.4 mm/s with YTS = 221 MPa and UTS = 266 MPa. Consequently, the colder the extrusion temperature, the higher the strength of the profile and the lower the ductility. The results make clear the extrusion process of ME21 is very robust and parameter variation has only a small influence on the resulting material properties. This behavior is very beneficial for the production of the seat back, because the process control is variable.

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