Microstructure and texture evolution of Mg-Zn-Al-Ca (ZAX210) during groove rolling

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Abstract. This study introduces the ZAX210 magnesium alloy wire, produced using groove rolling. The starting material, initially extruded, was successfully rolled from 12.0 mm to 6.7 mm diameter in eight passes using square-oval grooves, finishing with an oval and round shape. High strain rates and an optimized rolling temperature of 265 °C were found beneficial, avoiding hot cracks and promoting recrystallization. Microstructural analysis using light and electron microscopy revealed a fully recrystallized, fine microstructure with uniform precipitate distribution. Texture development is being assessed with EBSD, where a strong dependence of the respective groove shape on the expression of the texture was observed. Mechanical properties of the final wire were determined, whereas $R_{p0.2}$ is 308 MPa, R_m is 337 MPa and A is 17.8 %.

Introduction

Magnesium alloys can play a key role as innovative lightweight materials for reducing CO_2 emissions. This is mainly due to its low density, high specific strength and high life cycle assessment potential. The limited formability of magnesium due to its hexagonal crystal structure is considered a challenge, which is why the forming processes are mainly carried out at elevated temperatures for a sufficient number of sliding systems. Furthermore, an understanding and specific manipulation of texture during the forming process is crucial to achieve ductile material behavior. Long products such as rod and wire material made of magnesium can be produced by casting, extrusion, or groove rolling (also known as caliber rolling), with the latter standing out in terms of economic efficiency due to high production throughput combined with promising mechanical properties [1]. Groove rolling is known from the processing of steel, where the material is formed into a defined groove shape by a large number of rolling passes. The precise design of the groove shape has an influence on the microstructure development due to the locally acting force and the material flow behavior. Groove rolling promises a weakening of the basal texture in contrast to, for example, extrusion [2]. Previous studies on the groove rolling of magnesium alloys have concentrated mainly on conventional alloy concepts like AZ31 [1–8]. In comparison to steel alloys, a stronger spreading behavior can be observed in magnesium alloys, which indicates other material flow mechanisms [8,9]. On the process side, other groove shapes are therefore necessary for optimum production. The improved mechanical properties of the groove-rolled samples are primarily attributed to the effects on the microstructure development and in detail the grain refining effect. Doiphode et al. observed a strength-increasing effect due to grain boundary consolidation, and the effect is also attributed to the texture and formation of twins [1]. Heat treatments to improve the mechanical properties only make sense in the low temperature range (373-473 K) for increased ductility [10]. To the authors best knowledge, there are only few investigations into the processing of this alloy system using groove rolling. In a Mg-Al-Ca-Mn-Sr alloy, which was processed using groove rolling, elongations of up to 37 % at a strength of 140 MPa were achieved in tensile tests [11].

Another promising alloying system is the use of calcium, which shows similar effects to the use of rare earths regarding the weakening of the basal texture and a grain-reducing effect through additional precipitate formation. This increases formability, especially at low temperatures [12]. The current state of research into the Mg-Zn-Al-Ca system relates primarily to the processing of flat products.

This paper is the first to investigate the behavior of a calcium-containing magnesium (ZAX210) alloy during groove rolling. After successfully determining the process parameters for processing the alloy, the focus is on the development of the microstructure. The development of the texture is of particular interest. Furthermore, the mechanical properties of the wire are determined.

Methodology

The starting material was an extruded ZAX210 wire with a diameter of 12 mm containing 2 % Zn, 1 % Al, and 0.3 % Ca. The samples are heated to forming temperature in a convection oven and subsequently formed on a three-high rolling mill at Institute of Metal Forming, TU Bergakademie, Freiberg, Germany. A total of 8 rolling steps are carried out, the corresponding groove pass design can be seen in Fig. 1. A square-oval groove pass is used, whereby 12 mm round bars will be fabricated to a final cross-section of 6.8 mm round. The degree of elongation reaches from 1.38 to 1.05, the overall area reduction is 68.8 %. Before each pass, the billet was rotated 90° clockwise along rolling direction. For the rolling process, no lubricant or reheating was used. Following preliminary tests, a rolling temperature of 265°C and a roll speed of 120 rpm (corresponds to a forming speed of approx. 3.5 m/s and 3.7 m/s for the present roll diameter of 297 mm and 278 mm) was choose. For the present alloy, a high forming speed shows positive effects as a result of the increasing recrystallized fraction. The same applies to an increased rolling temperature; because of the occurrence of hot cracks (possible melting of MgZn precipitates) from 350 °C and the heating due to the forming, a temperature window at 265°C was obtained.

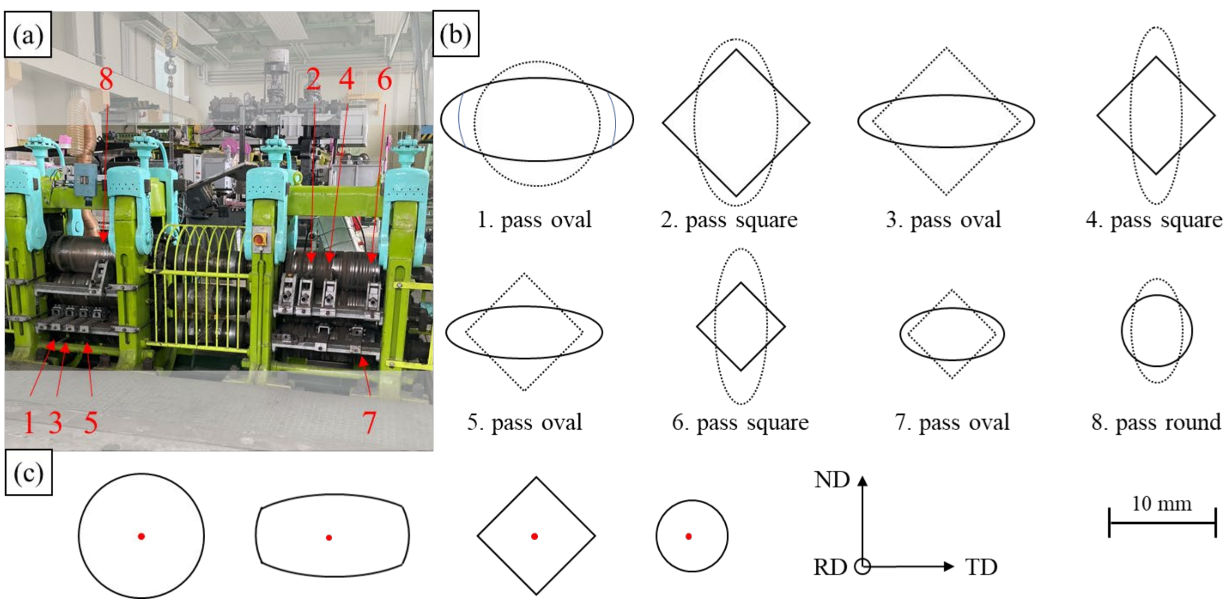

Fig. 1. Three high rolling mill (a) with the used square-oval groove pass design (b), where the dotted line is the input profile and the solid line groove pass. Blue Line in 1. pass represents an underfilling of the first groove pass. (c) Locations where microstructure is characterized (red dots).

Subsequently, microstructural images are taken from the initial extruded and rolled state using light and scanning electron microscopy to characterize the microstructure. For sample preparation, after cutting and grinding the samples with SiC-paper in five different steps, various polishing stages are carried out with MD-Chem OPS 300 magnesium polishing cloth and OP-chem, whereby attention is paid to regular rinsing with ethanol. The etching of the samples was conducted with a solution of picric acid (10 ml distilled water, 10 ml glacial acetic acid, 70 g ethanol and 4 g picric acid) for 15-30 seconds up to three times. The same samples were subsequently polished for scanning electron microscopy (SEM), whereby a ZEISS GeminiSEM 450 scanning electron microscope was used.

Texture analysis was performed using an electron backscatter diffraction detector (EBSD). The accelerating voltage was between 15 and 20 kV. A step size of 0.65 µm was selected. For the analysis of the EBSD data and the calculation of the pole figures, the free MTEX MATLAB toolbox [13] was used. A microstructural analysis was carried out on cross-sections in the center of the rod. Additionally, the samples were investigated by using energy dispersive X-ray spectroscopy (EDX).

Tensile tests are carried out at room temperature to determine the mechanical properties of the groove rolled samples. The specimen geometries correspond to DIN 50125 shape B with a specimen diameter of 4 mm and a test length of 20 mm.

Results and Discussion

Rolling process. The rolling could be carried out without cracking., it was found that the spreading behavior of the magnesium alloy was too strong for this schedule, resulting in overfilling. After adjusting the rolling gap, this was largely reduced, but overfilling still occurred in the final rolling pass. Furthermore, this was counteracted by deliberately underfilling the groove as the first rolling step. This can be seen in Fig. 1b with the blue line. The reason for the overfilling is the stronger spreading behavior in comparison to conventional steel alloys, for which the original groove pass design is intended. Compared to other magnesium alloys (see [9]), ZAX210 shows a similarly strong spreading behavior, which is, however, less dependent on temperature. Corresponding coefficients were included for the Freiberger model of spread [9] (Table 1) for the material coefficient $C_{W\beta}$ and the temperature coefficient, which is given by:

$$C_{T\beta} = a + b \cdot T \quad (1)$$

The Freiberg spreading model is used for the design of break down passes and includes all relevant influencing factors for groove rolling. The two coefficients for material and temperature behavior are decisive for transfer to other materials. Further explanations are given by [9].

Table 1. Coefficents of Freiberger model of spread [9] for different Mg wrought alloys.

Coefficient	$C_{W\beta}$ [ ]	a for $C_{T\beta}$ [ ]	b for $C_{T\beta}$ [ ]
ZAX210	1.4794	0.945894	1.38E-04
AZ31 (from [9])	1.0541	0.848512	5.13E-04
AZ81 (from [9])	1.0020	0.869500	4.28E-04
WE43 (from [9])	0.9119	0.886358	2.20E-04

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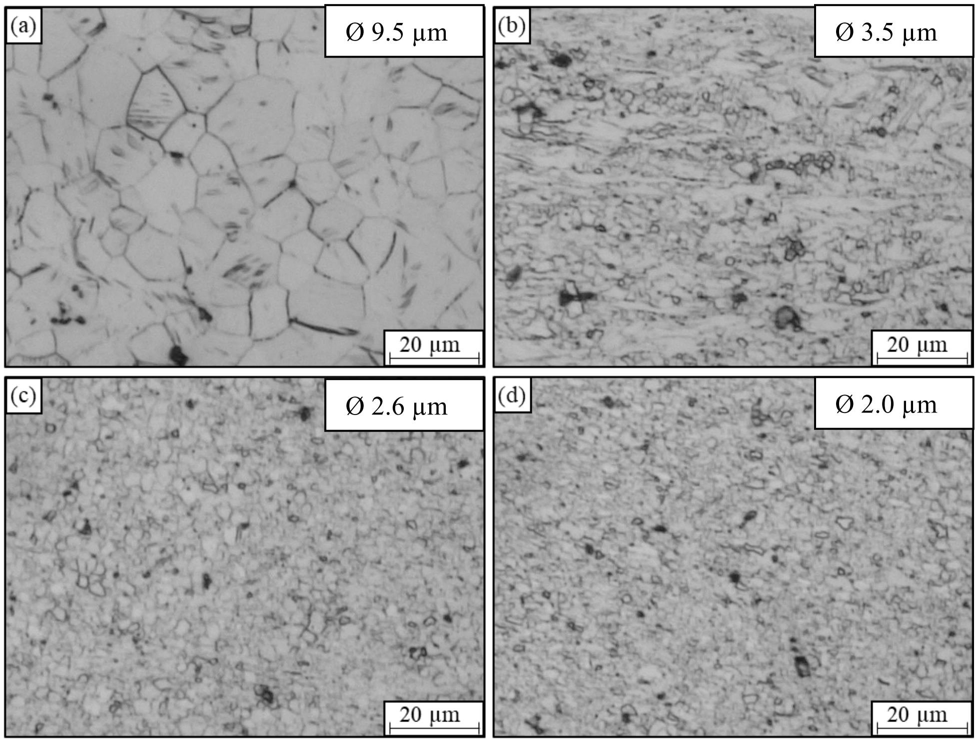

Fig. 2. Microstructure (center position) of the ZAX210 alloy in initial state (a), after 1. roll pass (b), 2. roll pass (c) and 8. roll pass (d). Average grain size via line intersection method on top right.

Microstructure analysis. The initial microstructure of the extruded state can be seen in Fig. 2a. The grain size distribution across the sample cross-section can be determined (Fig. 3a). There is also a partially recrystallized structure along the grain boundaries, which was produced by the forming process of extrusion. Fig. 2b - d show the microstructure after one, four and eight rolling passes respectively. A recrystallized microstructure can already be seen after the first rolling pass. This is also visible in the grain size distribution (Fig. 2b) with a decrease in grain sizes. The grain refining effect is mainly due to the dynamically recrystallized grains. The different force effect and thus degree of deformation distribution in the groove can be seen on the basis of light microscopic images of the first rolling pass at various points (Fig. 3). While a high proportion of recrystallization is already visible in the center of the sample, this cannot yet be observed in the edge areas. Initial recrystallization processes can be observed at the upper edge of the rolled sample, while in contrast there is hardly any change visible in the microstructure compared to the initial state at the edge without the effect of pressure. These differences can also be seen in the grain size distribution, whereby some larger grains in the area >10 µm exists. After the fourth or final eighth rolling pass, a completely recrystallized fine microstructure is present. Even after the fourth roll pass, no differences in the various position of the cross-section can be determined. Using EDX measurements, it was possible to determine that these are at least ternary intermetallic phases consisting of Al, Ca and Zn with different stoichiometric ratios (Fig. 4). The rolling processes have no influence on the number and distribution of the precipitates; similar precipitation tendencies could be observed in all samples at different points in the microstructure using EDX images. The exact determination of the precipitates present is difficult, as a large proportion of the precipitates are very fine and are therefore more difficult to detect using EDX.

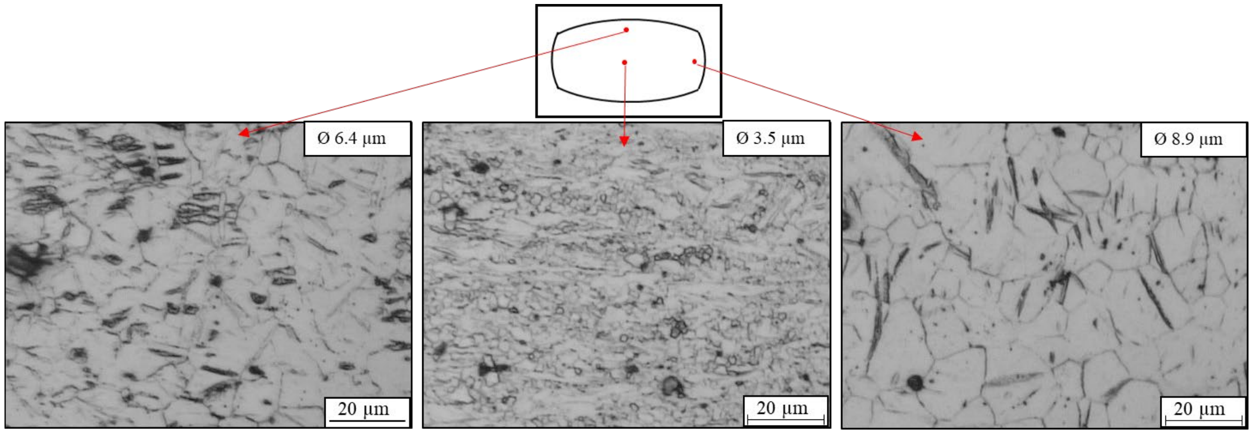

Fig. 3. Microstructure after the first roll pass at different positions. Average grain size via line intersection method on the top right.

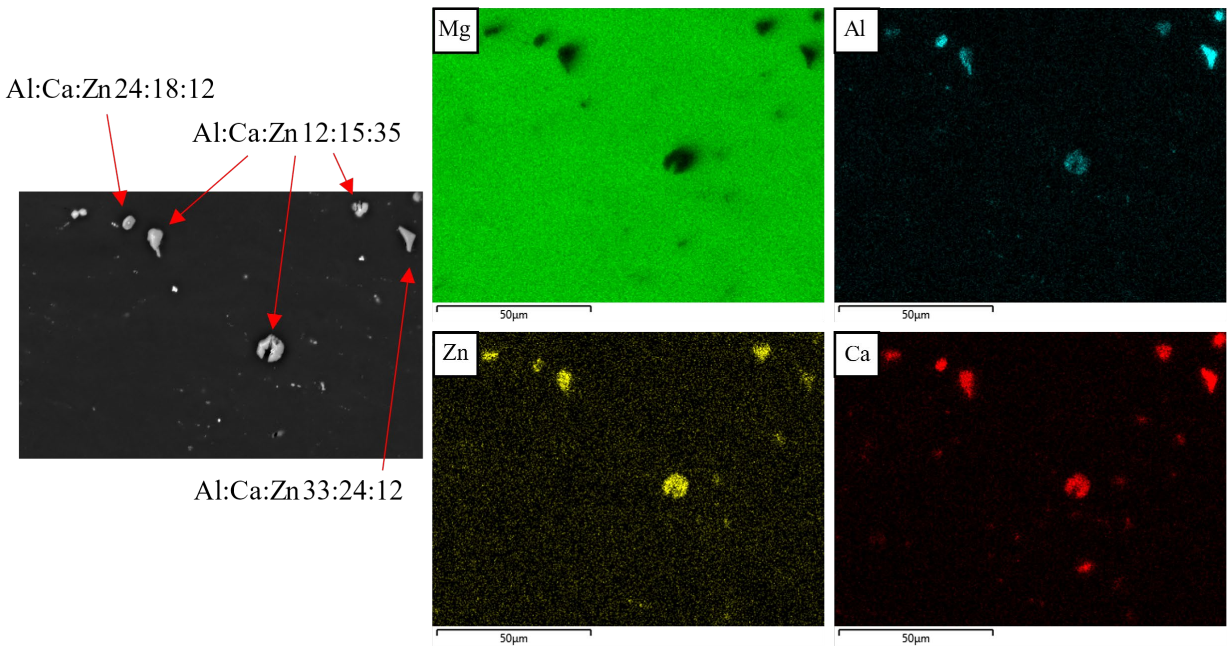

Fig. 4. EDX analysis of the intermetallic phases in the center after first roll pass.

Texture measurements. In the following, the EBSD pole figures calculated from the orientation distributions of grains in the rolled samples are discussed. An extruded texture is present prior to groove rolling, with a balanced orientation of the c-axes in the direction of the radial directions (here ND or TD) toward the outside. Furthermore, the peak of the misorientation angle at about 86° correspond to the $\{11\bar{2}0\}$ tensile twins. After the first rolling pass, a strong texture change is observed whereby the c-axis is oriented exclusively in the direction of the groove edges. These characteristics were confirmed by additional measuring points at the right edge of the rolled material and at the top contact point between the rolled material and the groove. A slight split form in the middle in the ND direction. A strong dependence of the texture on the corresponding groove shape is evident from the fourth pass. The sample was rolled from an oval to a square, with the first contact of the rolled material with the groove being off-center from the vertical line of symmetry. This can be seen in the texture as a slight shift of the maximum from the ND direction. The one-sided characteristic is technologically explained by a presumably slightly skewed entry of the rolled material, which shifted the maximum load to the right half of the groove. Similar observations can be made during the last rolling pass. Furthermore, the analysis of the twins shows

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that their importance in groove rolling appears to be secondary. Based on the misfit orientations, there is an increasing decrease in the twins, with slight peaks at 30° and 86°, which indicates $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twinning with 38° $\langle 11\bar{2}0\rangle$ rotation and $\{10\bar{1}2\}$ tensile twins with $\langle 11\bar{2}0\rangle$ orientation. These texture investigations were made in the center part of the rolled samples. Further investigations should deal with the microstructural development in different areas of the rolled material cross-section.

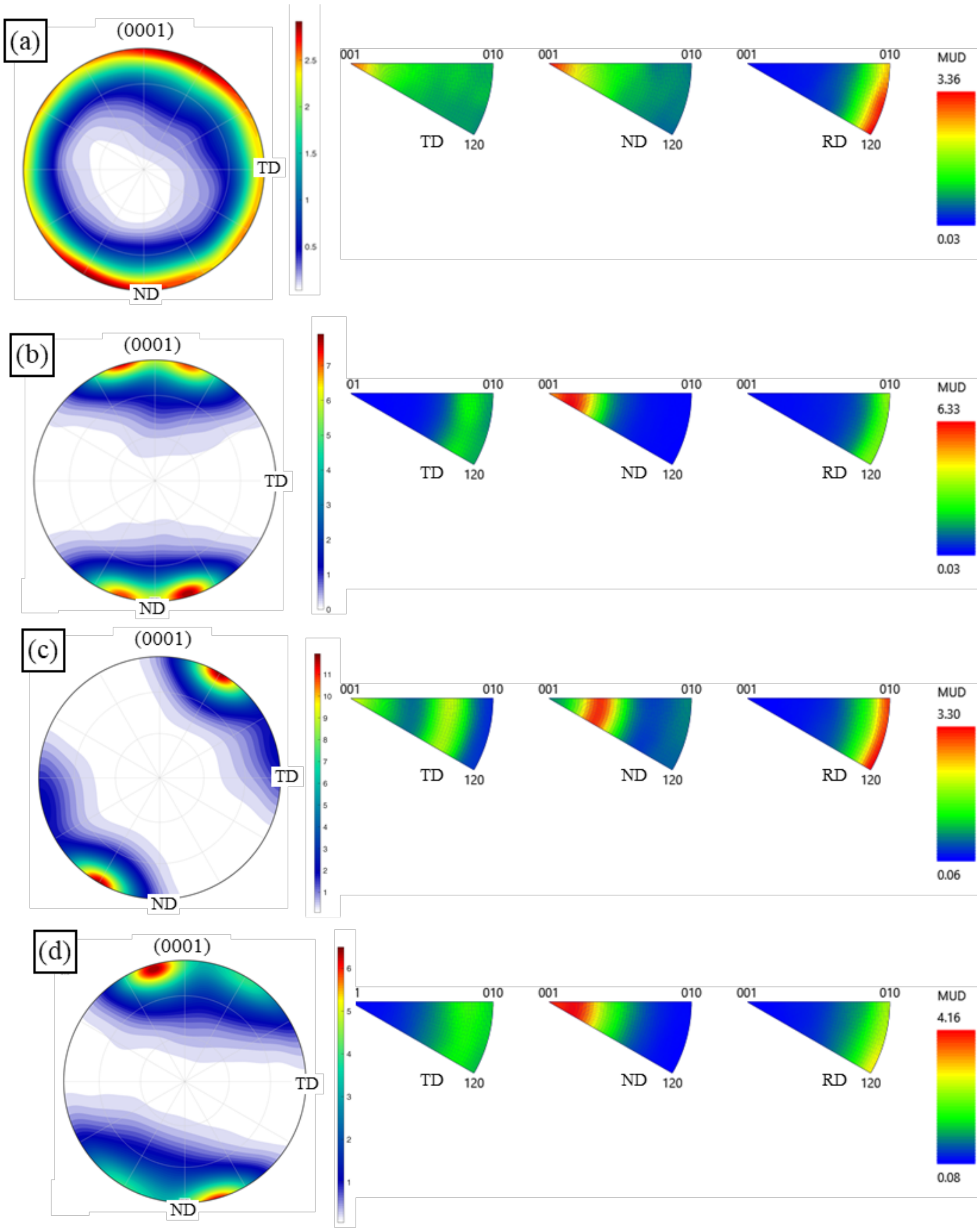

Fig. 5. {0001} and inverse pole figures of ZAX210 alloy in (a) initial extruded condition and after one (b), four (c) and eight passes (d) of groove rolling.

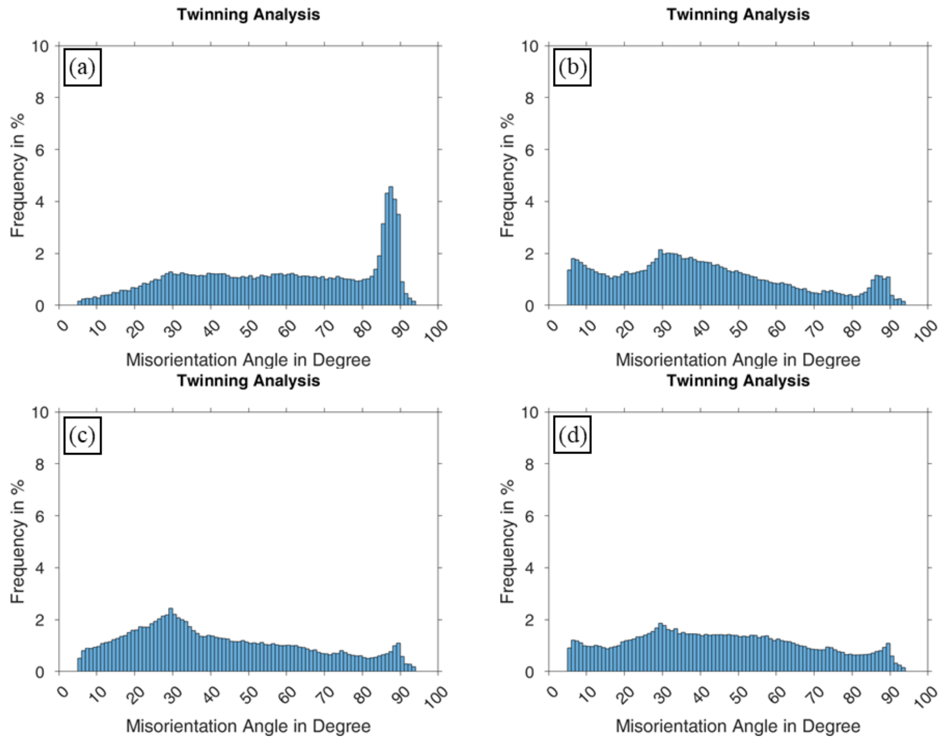

Fig. 6. Misorientation angle for (a) initial extruded condition and after one (b), four (c) and eight passes (d) of groove rolling.

Mechanical properties from tensile test. The results of the tensile tests are shown in table 2. The extruded material has a yield strength of 181 MPa, a tensile strength of 274 MPa and an ultimate strain of 17.2 %. After eight passes of groove rolling with a start rolling temperature of 265 °C, the material has a yield strength of 308 MPa, which is an improvement by 70.1 % compared to the extruded condition. For the tensile strength, an improvement of 23 % to 337 MPa can be determined. The elongation at break is almost identical to the extruded state at 17.8 %. The improvement of the tensile strength and especially the yield strength are due to the induced strain, which leads to dynamic recrystallization.

Table 2. Mechanical properties of the extruded and rolled alloy at room temperature.

Alloy	State	$R_{p0.2}$ [MPa]	R_m [MPa]	A [%]	Ref.
ZAX210	Extruded (initial)	181 ± 16.1	274 ± 6.4	17.2 ± 1.0	
ZAX210	Extruded and rolled (265 °C)	308 ± 13.0	337 ± 5.3	17.8 ± 1.3	
AZ31	Hot rolled (7 passes)	313	336	20.0	[10]
AZ80	Extruded and rolled (14 passes)	264 ± 3	365 ± 1	17 ± 1	[14]

Conclusion

In the present investigation, groove rolling of the calcium-containing magnesium alloy ZAX210 was carried out for the first time and microstructurally characterized and the mechanical properties determined. The following findings can be summarized:

a) The magnesium alloy ZAX210 containing calcium was groove rolled at 265°C without cracking, whereby a stronger spreading behavior was observed like other magnesium materials.

b) The finer microstructure improved the mechanical properties compared to the original material. After the 8th rolling pass, an average $R_{p0.2}$ of 308 MPa, R_m of 337 MPa and a total elongation of 17.8 % is achieved.

c) Groove rolling achieves significant grain refinement, with a completely recrystallized, fine-grained structure after the final rolling pass. Furthermore, a strong dependence of the respective groove shape on the expression of the texture was observed.

To gain a better understanding of the material during the groove rolling process, the microstructural development in different areas of the rolled material cross-section has to be investigated in the future. In particular, this will address the different stress and deformation states during groove rolling. Different texture development is also expected. This will be followed by further investigations into the material flow behavior in order to achieve the best possible groove filling and when designing the corresponding tools. In combination with an in-depth investigation of the process window, the effect and further improvement of the mechanical properties is also of interest.

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