

Twin roll casting of ZAX210 magnesium wire: Processing, microstructure, texture and mechanical properties

KITTNER Kristina^{1,a}, ULLMANN Madlen^{1,b*} and PRAHL Ulrich^{1,c}

¹Technische Universität Bergakademie Freiberg, Institute of Metal Forming, Bernhard-von-Cotta-Straße 4, 09599 Freiberg, Germany

^akristina.kittner@imf.tu-freiberg.de, ^bmadlen.ullmann@imf.tu-freiberg.de,

^culrich.prahl@imf.tu-freiberg.de

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Abstract. Wires of magnesium alloys are appropriate candidates for applications as filler materials, as biomaterials due to their good biocompatibility and excellent mechanical properties but also as materials for joining applications, such as welding wires or screws. Twin roll casting (TRC) provides an innovative and efficient technology for the production of semi-finished wires with a good property profile. TRC of wire is a comparatively new technology that has so far only been performed at the Institute of Metal Forming at Technische Universität Bergakademie Freiberg, Germany. In recent studies, the process was combined with the promising calcium-containing Mg alloy ZAX210 (Mg-2Zn-1Al-0.3Ca). The wire was produced with an oval cross section under variation of twin roll casting speed in order to investigate the influence of twin roll casting speed (2.5 m/min to 4.5 m/min) on microstructure and texture evolution. Microstructure after TRC consists of fine dendrites and locally deformed microstructure. Twinning and small globular recrystallized grains can be detected as well. Furthermore, it can be observed that higher twin roll casting speeds (4.5 m/min) result in more pronounced formation of intermetallic phases and segregations but also in a higher amount of recrystallized grains, which arise due to dynamic recrystallization during twin roll casting. Texture analysis reveals the development of strong basal textures at lower (2.5 m/min) twin roll casting speed and weakened textures with prismatic character at increased twin roll casting speed of 4.5 m/min.

Introduction

The twin-roll casting (TRC) processes, that combines both sollicitation and rolling in a single step, is known to allow the production of near net shaped products. In the last decades TRC for magnesium alloys was widely investigated in laboratory as well as in industrial scale. Compared to conventional processing routes for magnesium semi-finished products, twin-roll casting can save energy and capital costs and reduce material losses [1–3]. The research activities are mainly focussed on the production of sheets and strips. However, in 2017, a pilot plant for twin-roll casting of magnesium wire was installed at the Institute of Metal Forming (IMF) at Technische Universität Bergakademie Freiberg. The TRC wire pilot plant enables the production of wires with an oval cross sections 20 mm in width and 9.2 mm in height. First investigations were done with the magnesium alloy AZ31 [4].

Conventional production processes for magnesium wire consist of continuous casting combined by several steps of extrusion and heat treatment. It is well known, that extrusion can result in fine-grained microstructures that lead to high strength values of the rods or wires. Tong et al. (2015) [5] present a Mg-5.3Zn-0.6Ca alloy with an ultimate tensile strength (UTS) of 279 MPa after extrusion at 300 °C. Further results are shown for example by Bazhenov et al. (2021) [6] with 352 MPa ultimate tensile strength of a Mg-4Zn-1Mn-0.7Ca alloy or Du et al. (2013) [7] with 378 MPa UTS of a Mg-4.5Zn-1.13Ca alloy. However, deformation processes like extrusion contribute to the development of strong basal textures having a negative impact on ductility and formability of



magnesium alloys [8–10]. Strong basal textures result in poor ductility at room temperature due to insufficient number of available slip systems.

In order to overcome those drawbacks, research is focussed on addition of alloying elements to magnesium, which lead to weaker basal textures, and suitable processing routes, that also contribute to texture weakening by dynamic recrystallization (DRX). Tong et al. (2015) [5] showed the deviation and weakening of conventional basal textures after extrusion of a Mg-5.3Zn-0.6Ca magnesium alloy. DRXed grains exhibited inclined c-axis about 30° to 40° to extrusion direction. Zhang et al. (2012) [11] also showed texture weakening by Ca addition to a Mg-1Zn magnesium alloy during extrusion. A for Ca addition characteristic basal pole split developed, which leads to enhanced ductility and formability. Wang et al. (2017) [12] reported a split texture with double peaks of misorientation angle distribution by 30° to 60° in extrusion direction. The authors suggest that Ca addition reduced the generalized stacking fault energies of basal slip and prismatic slip and therefore promoted the activity of prismatic slip systems. Former investigations of Ullmann et al. (2019) [13] of twin roll cast and hot rolled sheet of the ZAX210 alloy also reveals that a texture with basal pole split and lower intensity develops. The texture weakening was attributed to the occurring recrystallization mechanisms, which were mainly identified as twin-induced dynamic recrystallization (TDRX).

In this study, twin roll casting of wire was performed on the unique pilot plant at the IMF. The Ca-containing magnesium alloy ZAX210 (Mg-2Zn-1Al-0.3Ca) was used in order to investigate the influence of twin roll casting speed on microstructure and texture development. The responsible mechanisms, which may arise during TRC, were discussed.

Experimental Procedure

In this study, ingots of the magnesium alloy ZAX210 (Mg-2Zn-1Al-0.3Ca) with a chemical composition according to Table 1 was used. Twin roll casting was conducted at a pilot plant for TRC of magnesium wire at the IMF (Fig. 1a). The ingots were melted in an electrical furnace at temperatures from 690°C to 715°C under protective gas atmosphere. The melt was transferred into a headbox using a dosing tube and finally, the melt was forwarded into a casting nozzle, which feeds the melt into the water-cooled closed groove. Further information can be found in [14,15,4]. The twin roll casting speed was varied between 2.5 m/min to 4.5 m/min in 0.5 increments. The twin roll cast wire exhibits an oval cross section with 20 mm width and 10.5 mm to 11.7 mm height (Fig. 1b). The edges of the TRC wire exhibit burrs, which occur, when the melt flows into the narrow gap between the upper and the lower roll. If further processing is intended, burrs must be removed from the surface of the wire.

Table 1. Chemical composition of the ZAX210 magnesium alloy in mass percent measured via optical emission spectrometry.

Zn	Al	Ca	Si	Cu	Fe	Ni	Mg
1.89	0.78	0.28	0.011	<0.001	0.006	0.003	Bal.

The twin roll casting process was accompanied by the development of finite element-based model using Abaqus. This enables information to be obtained on the temperature profile within the rolling gap and the mechanisms during solidification. The following assumptions were made prior to the simulation: Melt is a Newtonian fluid, melt domain (nozzle and the region between rollers) tundish is a closed channel and the boundary condition at the walls is non-slip/shear. For density 1,750 kg/m³ was used. The viscosity varies linearly with the solid fraction. Velocities for inlet and outlet were set to 2.5 m/min and 4.5 m/min with regard to the experimental procedure. The heat flux to the rollers, the air and the nozzle were estimated to be 2,000,000 W/m², 50 W/m²

and 0 W/m^2 . Melt domain and boundary conditions as well as the used meshing systems are shown in Fig. 2.

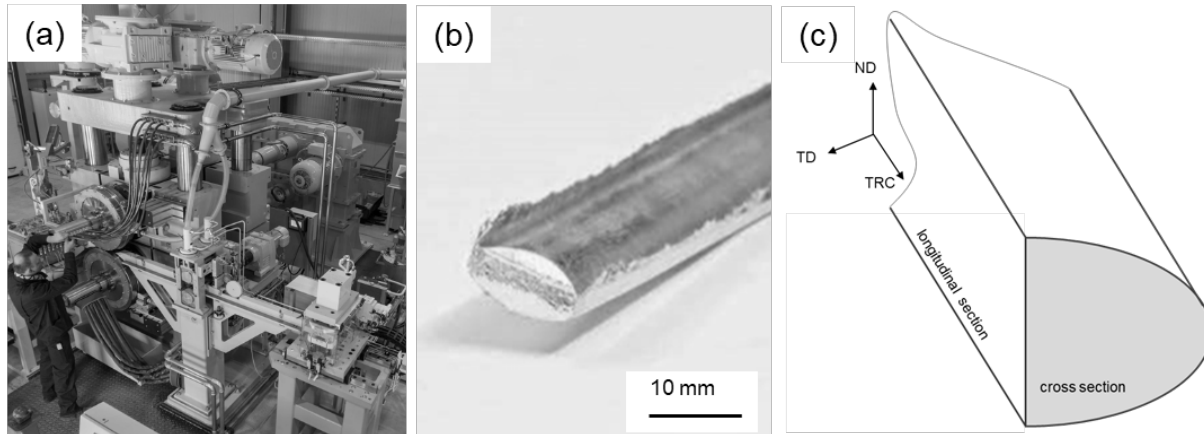


Fig. 1. (a) Pilot plant for twin roll casting of wire at the Institute of Metal Forming, (b) twin roll cast wire of the ZAX210 magnesium alloy and (c) schematic view of the wire for microstructure analysis.

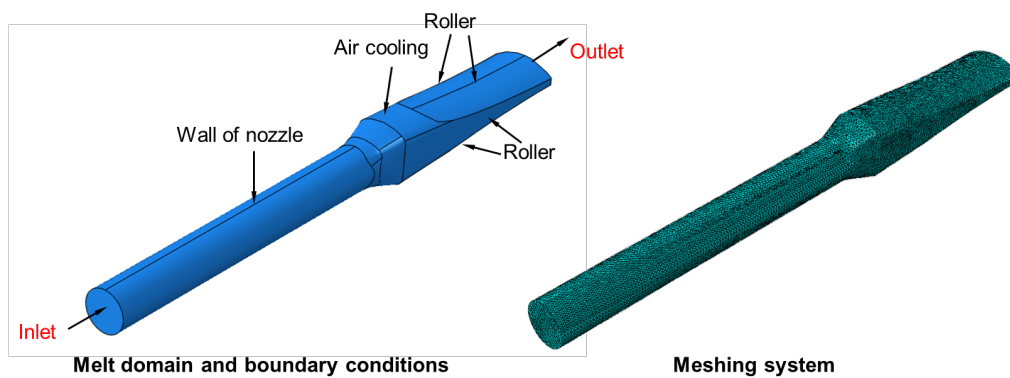


Fig. 2. Melt domain and boundary conditions and meshing systems of the applied twin roll casting system for the production of wire at the Institute of Metal Forming.

After twin roll casting samples for microstructural analysis were cut from the wire according to Fig. 1c with regard to the cross and the longitudinal section. Samples were metallographically prepared by several steps of grinding and polishing with oxide polishing suspension. After polishing and cleaning in an ultrasonic bath, the samples were etched with a picric acid solution for microstructural characterization. Optical and scanning electron microscopy (SEM) was conducted with the help of Zeiss GeminiSEM 450 in order to analyse microstructural characteristics with regard to the twin roll casting speed. Energy dispersive X-ray spectroscopy (EDX) was used to determine the chemical composition of individual microstructural constituents. Acceleration voltage of 20 kV and a working distance of 8.5 mm were applied. To investigate orientation relationships of the grains, electron backscatter diffraction (EBSD) analysis was used. The EBSD operating conditions were 15 nA probe current with 20 kV accelerating voltage and a step size of $0.5 \mu\text{m}$ to $2 \mu\text{m}$. For analysis of the EBSD data and the calculation of pole figures the free MTEX MATLAB toolbox [16] was used.

Tensile tests at room temperature were performed to determine the mechanical properties in TRC direction. Sample shape and dimension were selected according to DIN 50125-A5x25. Traverse speed was 0.625 mm/min .

Results and Discussion

Fig. 3 shows the microstructure of the twin roll cast wire in the polished and the etched state, exemplarily for the wire twin roll cast with a twin roll casting speed of 4.5 m/min. The polished state of the longitudinal and the cross section reveals, that fine and coarse intermetallic phases are evenly distributed, with fewer phases only occurring in the peripheral area. After etching a homogenous fine dendritic microstructure can be observed in the macroscopic micrographs. Strong centre line segregation, as it is reported for twin roll cast magnesium sheet [17,18], cannot be found in the twin roll cast wire of the ZAX210 magnesium alloy. Furthermore, the three characteristic areas, that can be found in TRC sheet (edge zone, dendritic columnar zone and central equiaxed zone) [19], are not obvious in the longitudinal section of the TRC wire. Because of the closed groove, cooling takes place evenly from all sides, so that the solidification conditions are different from those for the twin roll casting of sheet metal and consequently, the resulting microstructures differ from each other [19].

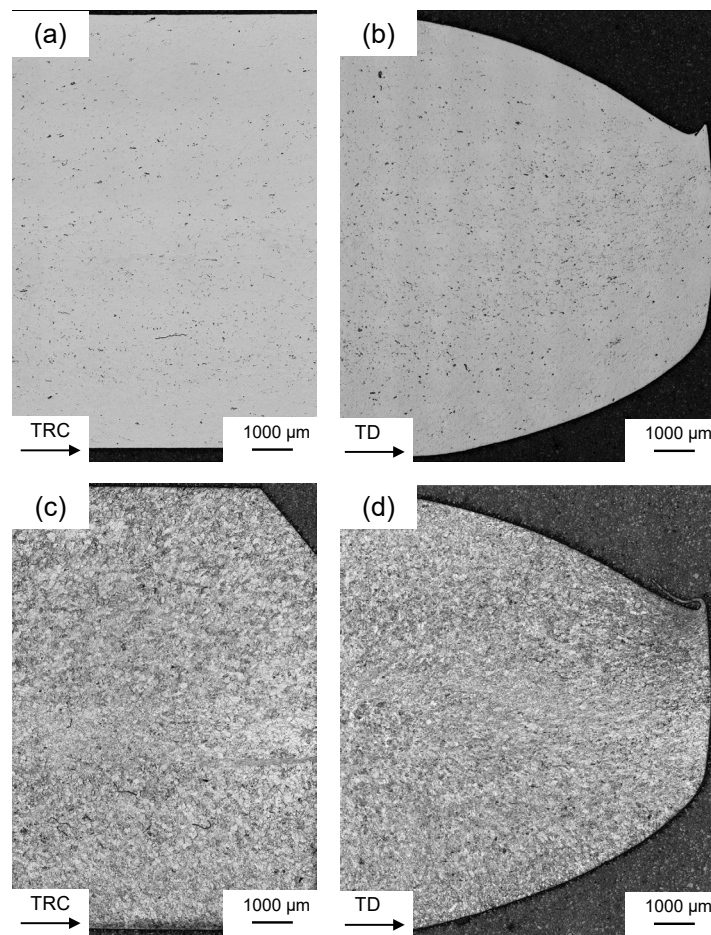


Fig. 3. Optical micrographs of the twin roll cast wire, exemplarily shown for a twin roll casting speed of 4.5 m/min in the polished state (a) longitudinal and (b) cross section and in the etched state (c) longitudinal and (d) cross section.

Detailed views of the microstructure are shown in Figure 4 for twin roll casting speeds 2.5 m/min, 3.5 m/min and 4.5 m/min, respectively. The micrographs refer to the middle and the edge of the cross section of the wire. Especially in the middle section, high amounts of twins can be observed in all samples. However, lower twin roll casting speeds seem to lead to a more pronounced formation of twins. Beetles et al. (2012) [20] reported that twins can be easily activated in Ca-containing magnesium alloys and may be responsible for an accelerated dynamic

recrystallization (DRX) due to twin-induced dynamic recrystallization (TDRX). Basis for this mechanism is the formation of double twins. Double twinning is defined as the simultaneous or partly nucleation of secondary twinning at parent twin-twin interfaces. Most commonly reported double twin structures are parental $\{1011\}$ or $\{1013\}$ primary twins and secondary $\{1012\}$ twins [21,22]. For the magnesium alloy ZAX210, the formation of double twins after hot deformation was presented by Kittner et al. (2022) [23].

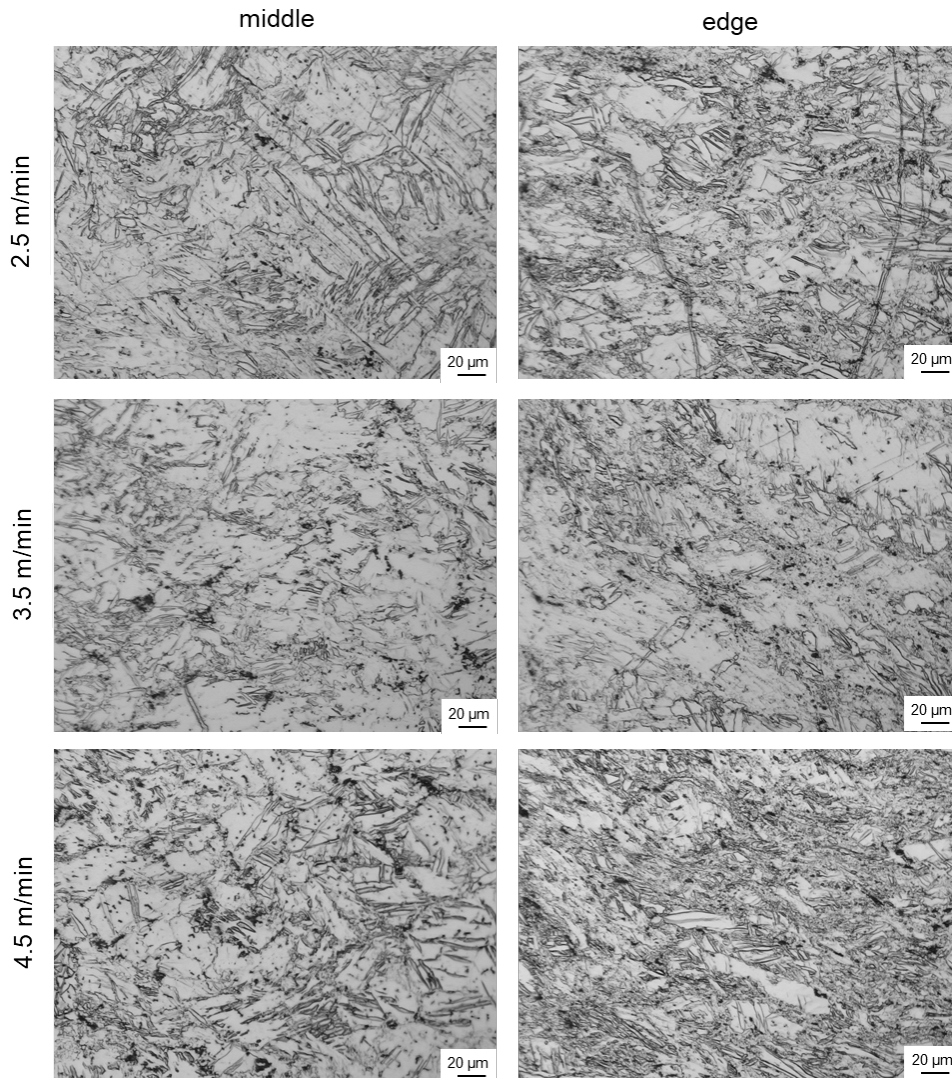


Fig. 4. Optical micrographs in detailed view of the middle and the edge of the cross section of the twin roll cast wire at twin roll casting speed 2.5 m/min, 3.5 m/min and 4.5 m/min.

Twinning analysis in Fig. 5 shows the frequency of misorientation angles in a range of 5° to 95° . There were basically three recognizable peaks. These can be assigned to double twinning (30°), twinning with twin plane $\{-2111\}$ (75°) and extension twinning with twin plane $\{10-12\}$ (86°). Commonly reported double twin types are primary twins of the $\{10-11\}$ or $\{10-13\}$ planes with $\{10-12\}$ twinning within the original twin interior [24]. The mentioned twin types have already been identified in magnesium alloys by other research groups [25]. The occurrence of double twinning is known to have a favourable effect on DRX of Ca-containing magnesium alloys [23]. Higher number of twins at 2.5 m/min twin roll casting speed can be assigned to the existing solidification conditions.

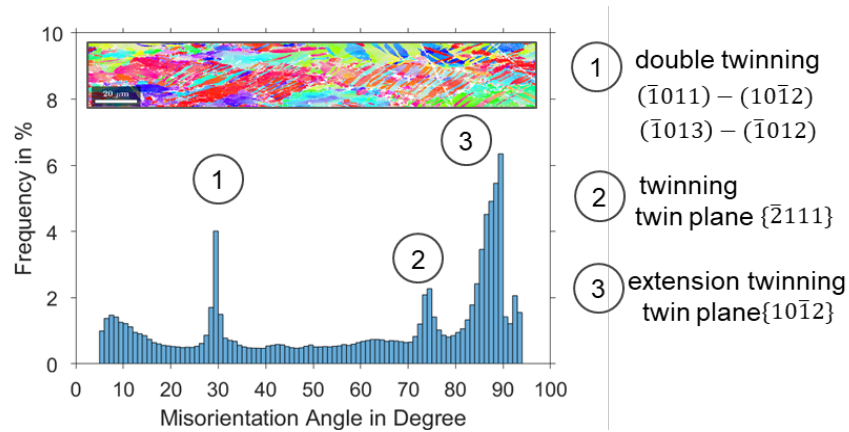


Fig. 5. Twinning analysis based on EBSD data for the twin roll cast magnesium wires.

The temperature distribution was simulated at a low (2.5 m/min) and a higher (4.5 m/min) twin roll casting speed (Fig. 6). At both speeds, it is clear that the edge area cools rapidly as the melt contacts the rolls. The temperature profile shows a more uniform cooling of the melt at the slower twin roll casting speed, which in turn leads to a more uniform solidification. Increasing twin roll casting speeds result in a delayed cooling of the melt at the centre of the wire and therefore to a change in the solidification and deformation conditions. From the FE-based model, it can be suggested, that at lower twin roll casting speeds (2.5 m/min), the maximum temperature of the wire when leaving the roll gap is nearly 200°C in the centre and 100°C at the edges. Consequently, an almost uniform temperature distribution over the cross-section can be achieved. Twinning can easily be activated at lower temperatures owing to the low critical resolved shear stress (CRSS) [26,27].

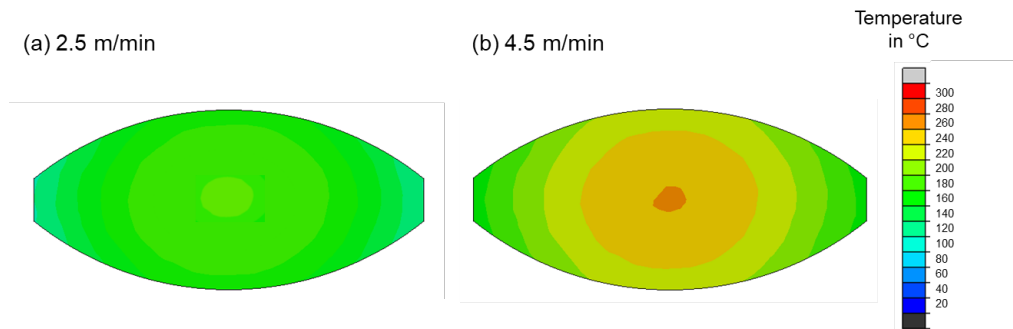


Fig. 6. Temperature distribution of the twin roll cast wires at 2.5 m/min and 4.5 m/min at the outlet of the roll gap.

With increasing twin roll casting speed, the temperature difference from edge to centre also increases and the maximum centre temperatures rise to values of 260°C to 280°C. A temperature of 220°C extends to the very edge of the wire. Those temperatures during the deformation, when the wire passes the closed groove, contribute to the initiation of dynamic recrystallization processes. For a twin roll casting speed of 4.5 m/min the formation of fine grains in the centre as well as at the edges can be observed. The amount of fine grains at the edges was determined to be 18 %, 29 % and 43 % for twin roll casting speeds of 2.5 m/min, 3.5 m/min and 4.5 m/min, respectively and is therefore higher compared to the core of the wire (< 10 %). This can be attributed to the higher strain that arises during deformation at the top and the bottom of wire, where the largest reduction takes place. The ZAX210 alloy exhibits a promoted recrystallization, especially at high strains [23,19]. Dynamic recrystallized grains are mainly located along the grain boundaries of the original microstructure or at twin boundaries. Therefore, continuous dynamic

recrystallization (CDRX) and TDRX are assumed to be the main softening mechanisms during twin roll casting of ZAX210 wire at higher twin roll casting speeds.

Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) analysis provide information about the composition of the microstructure. Fig. 7 presents SEM images of the twin roll cast wire at 2.5 m/min and 4.5 m/min twin roll casting speed. At lower twin roll casting speeds, the intermetallic phases (light grey) are finely distributed in the interdendritic areas. Because of their small size ($< 2 \mu\text{m}$) determination of the chemical composition via EDX is difficult and not reliable. X-ray diffraction (XRD) analysis of twin roll cast ZAX210 sheets conducted by Kittner et al. (2019) [19] indicates the occurrence of Mg_2Ca , MgZn -phases and $\text{Ca}_2\text{Mg}_6\text{Zn}_3$. Predominantly, the α -magnesium matrix was detected. Due to the more homogenous solidification, the intermetallic phases are finely distributed especially compared to the wire cast at higher twin roll casting speeds. Time for precipitation and diffusion processes is restricted. Locally, enrichment of residual melt with alloying elements can occur and small segregation areas developed. Overall, the intermetallic phases are unevenly distributed. In the segregation areas, the size of the intermetallic phases is larger than $2 \mu\text{m}$ and the EDX analysis provides a chemical composition of 11 % Ca, 35 % Zn and 54 % Mg, which corresponds to the phase composition of $\text{Ca}_2\text{Mg}_6\text{Zn}_3$ [28]. The chemical composition of the α -magnesium matrix exhibits no variations as a function of twin roll casting speed. Alloying elements contain a mass fraction of 1.4 % to 1.65 % Zn, 0.6 % Al and < 0.1 % Ca.

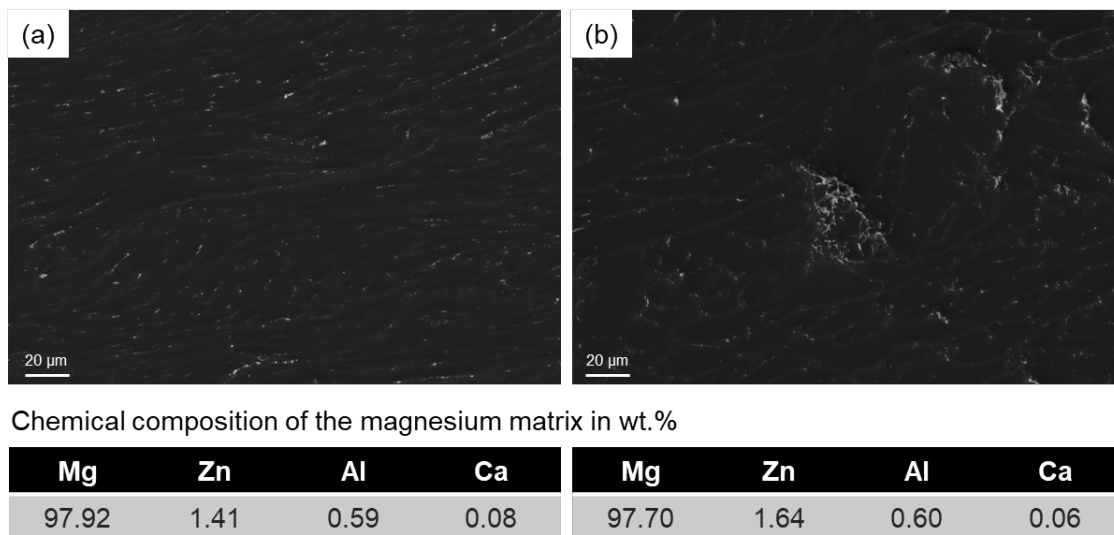


Fig. 7. SEM figures and chemical composition of the magnesium matrix, determined via EDX, of the twin roll cast wires at (a) 2.5 m/min and (b) 4.5 m/min.

EBSD analysis was performed to determine orientation relationships of the grains and to calculate pole figures. Fig. 8 presents the microstructure of the twin roll cast wires in inverse pole figure notation together with the calculated (0001) and (10-10) pole figures. It can be observed, that increasing twin roll casting speeds leads to the formation of small recrystallized grains, which exhibit a non-basal orientation (Fig. 8c). At low twin roll casting speed (2.5 m/min) no characteristics of DRX can be found. Resulting texture is dominated by a basal character with a maximum core intensity of 7.5 mrd (multiples of a random distribution) (Fig. 8a). Those textures also develop during deformation processes like rolling or extrusion. Especially deformed microstructures without dynamic recrystallization exhibit strong basal textures [9]. With increasing twin roll casting speed, the maximum core intensity is split in multiple maxima, which are tilted away from ND to TRC direction and texture with prismatic character is developed. The

maximum intensity decreased to 4.5 mrd at 3.5 m/min and 3.6 mrd at 4.5 m/min twin roll casting speed. At 4.5 m/min the prismatic character of the texture is more pronounced. The texture weakening by increasing twin roll casting speed of the Ca-containing ZAX210 magnesium alloy is attributed to the dynamic recrystallization. Comparable results were reported for magnesium alloys with Ca addition [11,12].

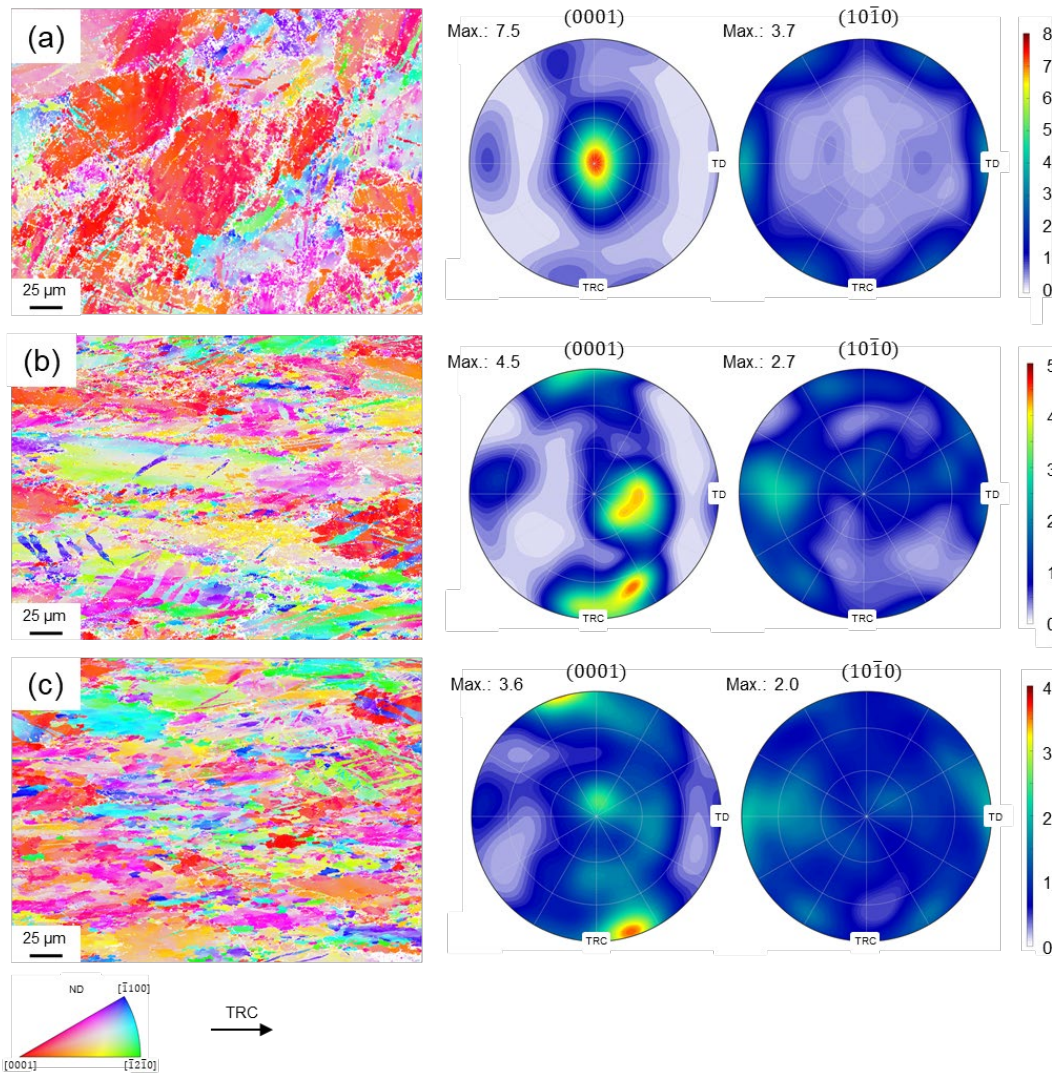


Fig. 8. Inverse pole figures and calculated (0001) and (10-10) pole figures of the twin roll cast ZAX210 wires produced with twin roll casting speed of (a) 2.5 m/min, (b) 3.5 m/min and (c) 4.5 m/min.

In order to determine the mechanical properties of the twin roll cast wires of the ZAX210 magnesium alloy, tensile testing was performed at room temperature. The results of proof stress, ultimate tensile strength (UTS) and elongation at fracture are listed in Table 2. It seems that twin roll casting speed has significant impact on the strength values of the twin roll cast wires. Proof stress and UTS range from 130 MPa to 135 MPa and 245 MPa to 257 MPa respectively. Strong basal textures may lead to limited plasticity because of operative basal slip and extension twinning [27]. However, at higher twin roll casting speed the UTS is lower. This may be due to the softening processes that occur during twin roll casting. The drop of the UTS seems to be less pronounced, as the small new recrystallized grains contribute to an increase in strength according to the Hall-Patch-relationship [29]. However, a significant increase in elongation at fracture can be observed with increasing the content of DRXed grains. Grain refinement can also effectively improve the

plasticity of the material. Texture weakening promotes the activity of basal < a > slip systems as well as non-basal slip systems, which can enhance the plasticity significantly [27].

Table 2. Mechanical properties of the twin roll cast ZAX210 wires determined via tensile testing at room temperature.

Twin roll casting speed in m/min	Proof stress in MPa	Ultimate tensile strength in MPa	Elongation at fracture in %
2.5	135	257	9.2
3.5	133	250	13.1
4.5	130	245	21.6

Conclusions

In this study, twin roll casting of the Ca-containing magnesium alloy ZAX210 was conducted to produce wire under variation of the twin roll casting speed from 2.5 m/min to 4.5 m/min. Microstructural and texture development as well as mechanical properties were analysed with regard to the twin roll casting speed. The main results can be summarized as follows:

- (1) Microstructures of twin roll cast wires exhibit several types of twinning: double twinning (30°), twinning with twin plane $\{-2111\}$ (75°) and extension twinning with twin plane $\{10-12\}$ (86°). Twinning formation is less pronounced when twin roll casting speed rises.
- (2) Increasing twin roll casting speed leads to initiation of dynamic recrystallization. Because of the occurring solidification kinetics, higher temperatures remain in the solidified wire before passing the closed groove. Deformation during TRC results in DRX via CDRX and TDRX.
- (3) Because of the occurring DRX processes, texture weakening can be observed at high twin roll casting speed. The texture changes from a strong basal texture with high core intensity (2.5 m/min) to a texture with a prismatic character and basal pole split with lower intensities (4.5 m/min).
- (4) The twin roll cast wires offer good mechanical properties with 135 MPa proof stress and 257 MPa UTS as maximum strength values at 2.5 m/min. Increasing twin roll casting speed results in a slight decrease of the UTS to 245 MPa. However, due to the recrystallization processes, which lead to grain refinement and texture weakening, plasticity can be improved. Finally, the elongation at fracture enhances from 9.2 % to 21.6 % when twin roll casting speed was increased from 2.5 m/min to 4.5 m/min.

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