Reduction of plastic anisotropy and improved formability of novel magnesium alloy sheets through utilization of Equal-Channel Angular Pressing (ECAP)

FINAL REPORT

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1 General Information

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Project title:	Reduction of plastic anisotropy and improved formability of novel magnesium alloy sheets through utilization of Equal-Channel Angular Pressing (ECAP)
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2 Summary

In diesem Projekt wurde die Auswirkung verschiedener Herstellungsrouten auf die Mikrostruktur, die Textur und das Umformverhalten einer Mg-Zn-Nd-Y-Zr-Legierung untersucht. Besonderes Augenmerk wurde dabei auf die thermomechanische Verarbeitung, die damit verbundene Charakterisierung (mikrostrukturelle und mechanische Eigenschaften der verarbeiteten Proben) und das Verständnis der beteiligten Verformungsmechanismen (mittels kristallplastischen (CP) Modellierungsansätzen) gelegt. Um dieses Ziel zu erreichen, wurde das Equal-Channel Angular Pressing (ECAP) Verfahren zur Deformation der Mg-Bleche eingesetzt. Dieser Prozess führt zu hohen plastischen Scherdeformationen im verarbeiteten Material, was es erlaubt die kristallografische Textur einem spezifischen Umformverhalten anzupassen. Die mittels ECAP bearbeiteten Proben zeigten eine Verbesserung des einachsigen Verformungsverhaltens mit ausgewogener Kaltverfestigung, guter Duktilität und geringerer planarer Fließasymmetrie. Trotz dieser Verbesserungen wurde festgestellt, dass das Umformverhalten im Tiefziehzustand im Vergleich zu anderen thermomechanischen Verarbeitungsmethoden schlechter sein kann. Im Rahmen dieses Projekts wurde die Auswirkung des Kaltwalzens auf die Texturgestaltung dieser Legierungsart systematisch untersucht. Dabei wurde festgestellt, dass eine basale Textur grundsätzlich die Zipfelbildung beim Tiefziehen dieses Legierungstyps reduzieren kann. Darüber hinaus wurde untersucht, wie die normale und planare Anisotropie (in Verbindung mit verschiedenen Zuständen dieser Legierung) die Zipfelbildung beeinflusst. Außerdem konnte durch eine CP-Simulation ein tiefes Verständnis für den Einfluss der Aktivierung verschiedener Deformationsmechanismen geschaffen werden. In den letzten Phasen des Projekts wurde auch der Effekt der Erholung analysiert.

In this project, the effect of different processing routes on the microstructure-texture and related deformation and forming behavior of an Mg-Zn-Nd-Y-Zr alloy was investigated. Special emphasis was placed on the thermomechanical processing, related characterization (microstructural and mechanical of processed samples) and understanding of involved deformation mechanisms (via crystal plasticity (CP) modelling approaches). To reach this goal, the use of Equal-Channel Angular Pressing (ECAP) to deform Mg sheets was the starting point. This technique was selected as the introduction of severe shear deformation allows to tailor the crystallographic texture for a specific deformation behavior. Samples processed via ECAP have shown an improvement in the uniaxial deformation behavior with well-balanced work hardening, good ductility, reduce planar yield asymmetry. Despite these improvements, it was found that the forming behavior under deep drawing state can be worse in comparison to other thermomechanical processing approaches. In the frame of this project, the effect of cold rolling

on the texture design of this type of alloy was systematically investigated. It was found that a basal texture can in principle reduce the earing behavior during deep drawing of this alloy type. Accordingly, an understanding of the meaning of the normal and planar anisotropy (related with different conditions of this alloy) is provided. Furthermore, through CP simulation, it was possible to provide a deep understanding of the influence of the activation of different deformation mechanisms. In the last stages of the project, also the effect of recovery has been implemented to this type of analysis.

3 Development of the work carried out

3.1 Initial questions and objectives of the project

Conventional magnesium alloy sheets normally exhibit limited ductility and formability if they are deformed at temperatures close to room temperature (RT). This is related to the limited number of active deformation systems, in particular, the predominant action of basal $\langle a \rangle$ slip and mechanical twinning. Potential activation of other non-basal slip systems is possible with the rise of deformation temperatures due to a reduction of the critical resolved shear stresses (CRSS) [Boh15]. Thus, slip systems, such as prismatic $\langle a \rangle$ slip and pyramidal $\langle c + a \rangle$ slip can be activated leading to an increase of the forming capabilities of conventional Mg alloys such as AZ31 alloy. The addition of rare earth elements lead to changes in the deformation and recrystallization mechanisms active during thermomechanical treatments and subsequent heat treatments, e.g. recrystallization annealing. As a result, distinctive crystallographic textures can be obtained after hot rolling and extrusion. For instance, hot rolled Mg-Zn-RE (e.g. ZE10) develops weak crystallographic textures, i.e. RE-textures, that show a broad spread of basal planes towards the transverse direction (TD). That off-plane inclination of the basal planes with respect to the sheet plane allows the activation of dislocation slip in the basal plane, which is the main deformation mechanism in Mg alloys. This leads to an enhancement of the RT ductility, reduction in the tension/compression anisotropy (in the case of extruded bars), a well-balanced strain hardening (especially along the direction with broader scatter of basal planes) and good stretch formability. In addition to weakening the texture, RE additions also have an influence on the activation of various deformation mechanisms in Mg alloys. For example, they can promote the activation of non-basal slip mechanisms, such as prismatic $\langle a \rangle$ slip [Vic19-1] [Ha19] and pyramidal $\langle c + a \rangle$ slip [San11] [Zha13] and, different twinning types [Suh16-1], which further improve the ductility and formability. After thermomechanical treatment, e.g. rolling, and subsequent static annealing there is the stable development of texture components that persist after static recrystallization, i.e. the so-called TD-split texture. This

leads to a markedly difference on the work hardening rate (WHR), Lankford parameter (r-value), and ductility of the material with respect to the mechanical testing direction, e.g. uniaxial tension tests. This has deep implications on the anisotropic behavior of such alloys, which lead to undesirable defects during forming operations, e.g. earing behavior during deep drawing with concomitant inhomogeneous wall-thickness of the workpiece. Despite the huge efforts to understand the mechanical behavior of Mg alloys having off-basal textures, the topic dealing with the attenuation of planar anisotropy has not been deeply investigated. This is because most of the studies are aiming only to improve the formability neglecting this important issue.
Therefore, the ECAP technique has been used to process Mg sheets (ECAP tool has been developed in the DFG founded project (LE1395/3-1, HO 2165/47-1)) and to explore the effect of the introduction of shear deformation to alter the crystallographic texture of formable Mg-Zn-RE alloys. The main objective was to understand the physical mechanisms involved during warm deformation and to provide guidelines to reduce the anisotropy of Mg-Zn-RE alloys while maintaining its good formability.

3.2 Results of the project

In the development of this project, one important activity was to find an appropriate composition of the Mg-Zn-RE alloy that made it able to be deformed via ECAP. In the original hypothesis, a Mg-Zn-Ca (Mg-1Zn-0.2Ca wt.%) alloy was also considered to be investigated. This is because Ca can at some extend emulate the effect of RE in changing the crystallographic texture leading to a weakening effect and to promote a broad distribution of basal planes towards TD. However, the formation of a brittle Mg_2Ca laves phase leads to the formation/nucleation of cracks in this intermetallic phase during the ECAP process.
One of the first steps was to use a formable Mg-1.6Zn-0.52-Y-0.45Zr-0.2Nd-0.1Gd (wt.%) produced via twin-roll casting technology at Helmholtz-Zentrum Hereon. Despite this alloy showed good stretch formability at RT, after ECAP processing the ductility of the samples was significantly decreased [Böh23]. In a parallel work, a novel Mg-1.8Zn-0.2Nd-0.2Y-0.1Zr (ZEWK alloy) was developed in [Sch23] and produced via a modified direct chill casting method [Els11]. The preparation method ensures a clean alloy with low amount of impurities such as Mg oxides and pores. This alloy showed excellent formability prior and after ECAP process [Vic23]. Consequently, results from this alloy processed in different conditions, i.e. after hot rolling, cold rolling and ECAP, are to be provided in this report.

For the first stage of the project, the ZEWK alloy was hot rolled at 400 °C using 11 mm thick rolling slabs. Four rolling passes were performed using a thickness reduction of 0.1 strain.

Afterwards, seven additional rolling passes were imposed, each of which induced a thickness reduction of 0.2 strain to achieve a final thickness of 1.8 mm. In between rolling passes, the sheet was reheated for 10 min at the rolling temperature. After the last rolling pass, the sheet was air-cooled to RT. Recrystallization annealing was performed at 400 °C for 5 min to avoid excessive grain growth.
With the aim to find a way how to modify the crystallographic texture, i.e. TD-split texture, of the ZEWK alloy, additional rolling experiments were conducted. The rolling schedule described above was followed up to the 10^{th} pass. The remaining 0.2 strain of the last rolling pass was divided into two cold rolling passes. This led to the development of a basal-type texture of the ZEWK alloy in the as-rolled condition.

The ECAP process was simulated as part of the third work package. The FEM program Abaqus was used for this purpose. The result of the 3D simulation can be seen in Fig. 1. A dynamic explicit solver was used for the simulation.
The same channel angle and the same channel thickness were used as in the real ECAP tool. Only the width of the tool was reduced to save computing time. For further investigations, only the central part of the formed Mg sheet was considered to avoid any edge influences at the open areas of the tool. In this central area of the sheet, the extent of the plastic shear strain introduced was investigated. The deformation gradient was calculated from the degree of distortion of the elements and determined. This information was used as an input to predict the resulting ECAP texture of the ZEWK alloy via CP simulations using the Viscoplastic Self-Consisted (VPSC) model. As shown in Fig. 1, the VPSC model captured the main deformation texture components of the simulated alloy. Yet, the intensity is slightly higher than the experimental texture. This can be related to texture weakening effect promoted by the dynamic recrystallization. Moreover, there is a reduction of the shear deformation introduced by the real ECAP process due to friction effects as described in [Vol22] leading to this discrepancy in the deformation texture intensities.

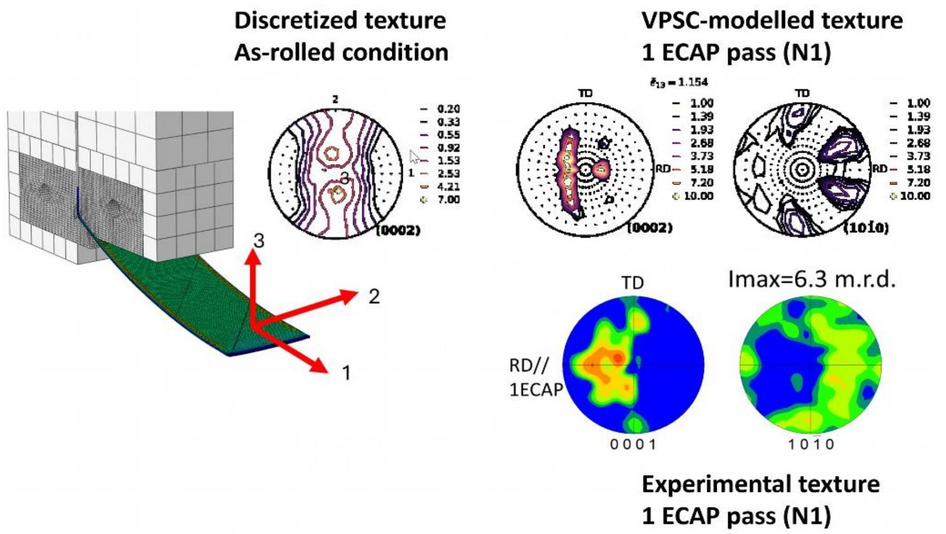

Fig. 1: FEM simulation of the ECAP process and VPSC simulated texture of the ZEWK alloy at 225 °C.

A schematic sketch of the ECAP tool used is shown in Fig. 2 (left). In the ECAP process, the sheet material is pressed through an angled (Φ = 120°) and heated (T = 225 °C) tool channel. Shear strains are introduced in the forming zone, which lead to plastic deformation of the Mg sheets. The discontinuous ECAP process allows the sheets to be pressed several times. The routes investigated as part of this project are shown in Fig. 2 (right). In route N1, the sheet passes through the ECAP tool once in such a way that the ECAP direction (ED) is parallel to the rolling direction (RD). The sheets of routes C2 and DC2 can be produced by pressing the sheets a second time and rotating the sheets in a different manner. Due to the ECAP process, the Mg sheets exhibit a slight curvature after the ECAP process. To reduce this curvature, the sheets were heat-treated after each ECAP process (30 min at 200 °C) to allow the straightening of the samples without a significant change in the microstructure and texture [Suh15]. In addition to curvature reduction, the heat treatment also leads to an improvement in ductility while maintaining strength [Val 00].

It was also investigated how the temperature in the Mg sheets develops during the ECAP process. For this purpose, small holes were drilled into the side of some sheets and temperature sensors were threaded into them. After inserting the sheets into the tool channel, a 60 s wait was always carried out until the pressing process was started. During this phase, the sheets heated up to an average of 80 °C. Directly in the forming zone, the sheets briefly reached temperature peaks of up to 218 °C, which is very close to the set tool temperature of 225 °C.

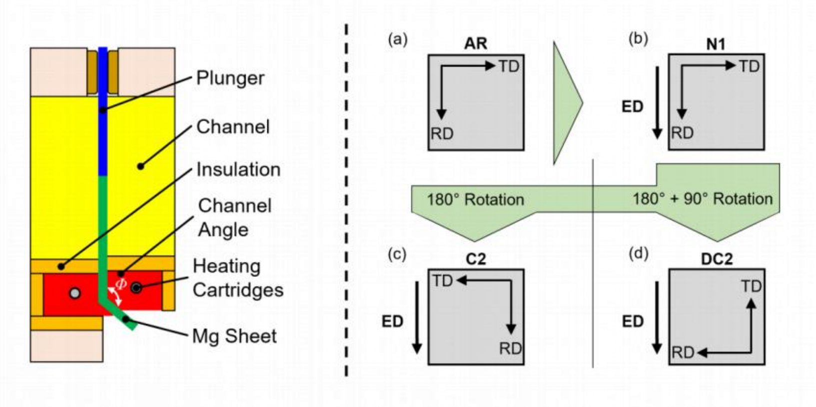

Fig. 2:Schematic representation of the ECAP tool (left) and investigated ECAP routes (right): (a) initial sheet (AR), (b) N1 route, (c) C2 route and (d) DC2 route based on [Böh23].

The effects of the ECAP process on the microstructure can be seen in Fig. 3. As route C two introduces severe shear strain only in the rolling direction (RD), the formation of micro-cracks in the surface of the sample was noticed. This led to a detrimental effect of the forming experiments. Therefore, two processing routes were investigated in more detail, i.e. one ECAP pass parallel to RD (ECAP N1) and two ECAP passes following the route DC2 (ECAP DC2) (see Fig.2). In the as-rolled condition, the ZEWK alloy sample exhibited a mixture of recrystallized and deformed grains inherited from the rolling-annealing processing. Initial microstructure was nearly free of twinned areas, and an average grain size of 17±6 µm was observed with a distinctive texture consisting of the $(0002) \perp \mathrm{RD}$ with a broad spread of basal planes towards TD and $(10\bar{1}0) \parallel \mathrm{RD}$ components. After the first ECAP pass, there the processed sample exhibits a deformed grain structure and the formation of multiple twins. It is important to highlight that the degree of recrystallization is limited. The texture, however, shows a slight weakening in the texture intensity and the main texture component is tilted towards RD. The DC2 processed sample is characterized by a more fragmented grain structure with the appearance of more dynamically recrystallized grains. The deformation texture of the sample shows the (0002) component parallel to TD. To enhance the mechanical behaviour and the formability of the ECAP processed samples a short recrystallization annealing at 350 °C for 30 min was performed. As observed in Fig. 3, the grain structure is more equiaxed and there is a significant weakening of the texture intensities and a noticeable grain size refinement.

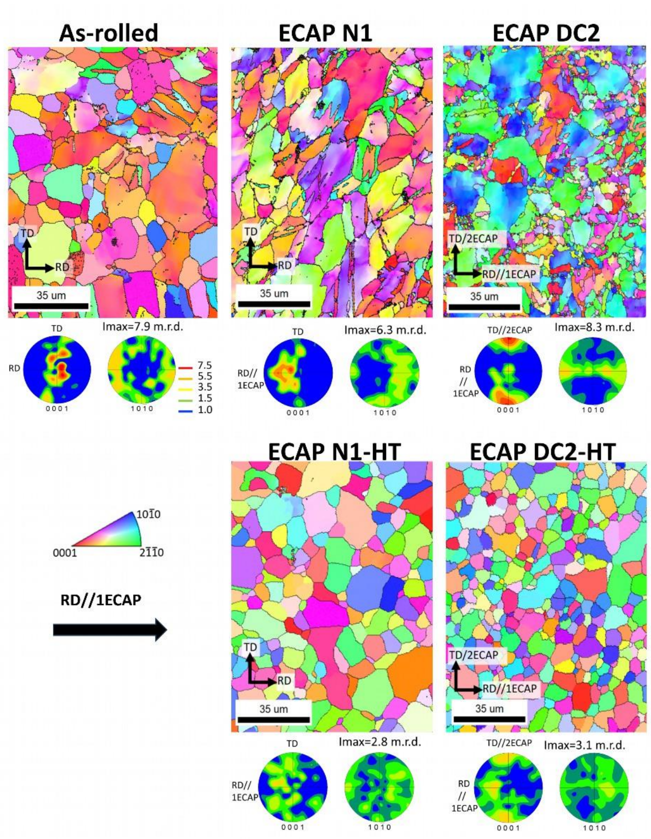

Fig. 3: Inverse pole figure (IPF) maps of a rolled sample and ECAP processed sheets following the route N1 and DC2 in as-received and annealed conditions respectively.

To determine the influence of the ECAP process on the mechanical properties of the Mg sheets, tensile tests were carried out at RT (see Fig. 4). Samples were tested in rolling direction (RD) and transverse to rolling direction (TD). As observed in Fig. 4, the yield asymmetry of the ECAP samples is significantly reduced in comparison to the rolled material. Also, it is important to highlight the increase in ductility of the ECAP samples.

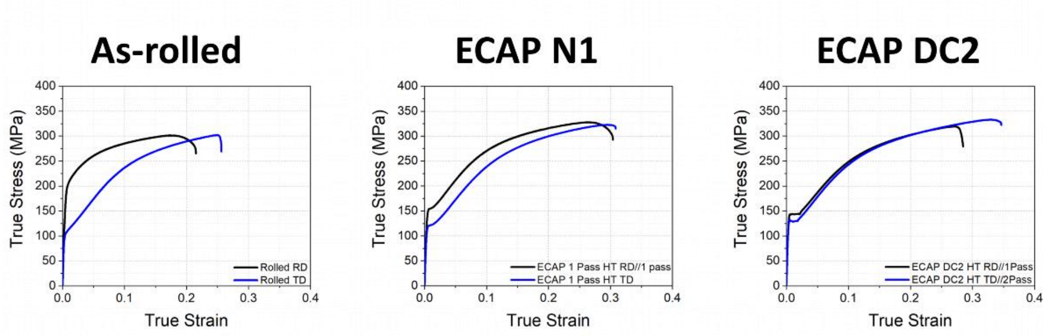

Fig. 4: True stress-true strain at RT of rolled, ECAP N1 and ECAP DC2 processed samples.

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Fig. 5 shows the results of ex-situ tensile testing results used to characterize the relative activity of dislocation modes in samples cut along RD and TD, respectively. Exemplarily, the results of the ECAP DC2 sample are presented. The frequencies of various slip modes were assessed by tracing the associated slip lines after a strain of 10 %. The Euler angles of grains with visible slip traces were used as input to a Matlab™ code [Cha16] that provides potential slip lines pertaining to individual slip modes. An example is shown in Fig. 5. Regardless of the processing route, the basal slip showed the highest frequency, followed by prismatic $\langle a \rangle$ and pyramidal $\langle a \rangle$ slips.

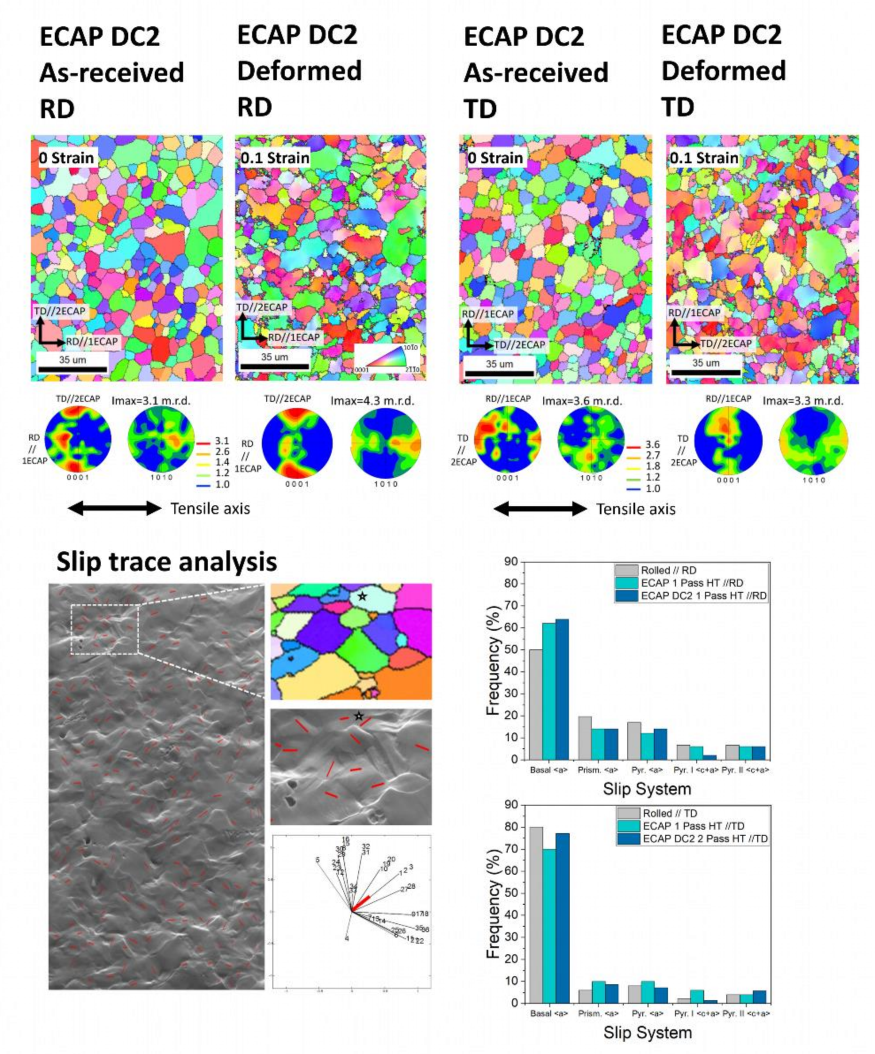


Fig. 5: Ex-situ IPF maps of RD and TD samples uniaxially deformed in tension up to 10 % strain to assess the activity of slip modes via slip trace analysis.

The previous experimental results were used to validate the CP simulation using the VPSC model. In this case, the hardening behaviour and texture evolution have served to adjust the model parameters. The initial input data, such as CRSS and other fitting parameters, were taken from [Vic22]. The model can simulate to a good agreement the hardening behaviour and the experimental texture development of the different processing routes (Fig. 6). Regarding the relative activity of deformation modes, the model predicts well the dominant activity of basal $\langle a \rangle$ slip, followed by prismatic $\langle a \rangle$ and pyramidal $\langle a \rangle$ slip. It is important to see that ECAP can significantly modify the activity of basal $\langle a \rangle$ slip and $\{10\bar{1}2\}$ extension twins leading to a reduction of the yield asymmetry and a similar hardening behaviour in RD and TD tested samples.

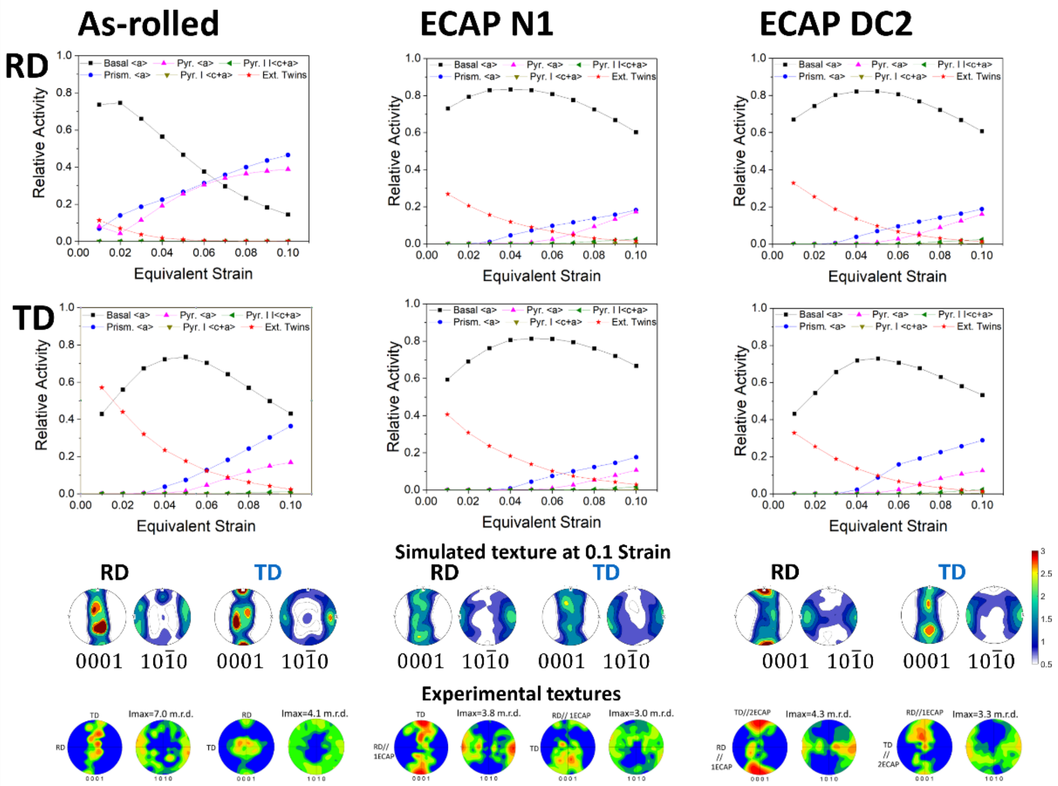
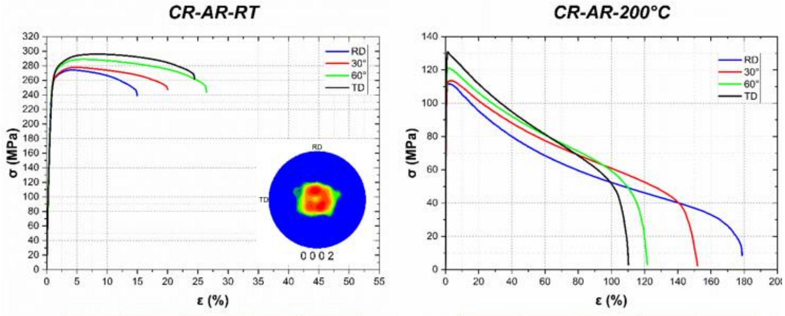


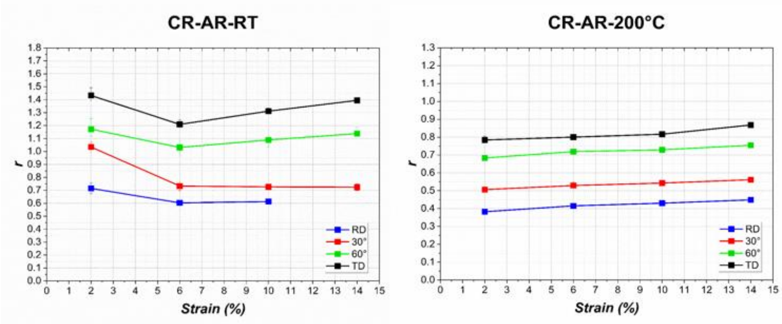
Fig. 6: VPSC modelling of as-rolled, ECAP N1 and ECAP DC2 processed samples. Exemplarily the hardening behaviour of the DC2 sample is presented. The experimental and simulated texture development are also presented.

In the case of the determination of the Lankford (r) parameter, tensile tests have been carried out at RT and at 200 °C, whereby 200 °C is also the selected temperature for the deep drawing experiments in this work. Tensile samples cut parallel to the RD, 30°, 60° and 90° (TD) with respect to RD were tested until facture. Furthermore, additional samples were used to characterize the r-value at strains of 2, 6, 10 and 14 % strain. As mentioned above, experiments using a cold rolling strategy were performed and they showed the best results in terms of a reduction of anisotropy and increase ductility. Fig. 7 shows the typical engineering stress-strain curves of these samples. These results are worth to show as the lowest earing behavior has been achieved using these samples. In the as-rolled condition (CR-AR) tested at RT, there is low yield asymmetry in all tested directions while the TD direction is the one showing the highest strength. The opposite is the case, if the sample is subjected to a recrystallization annealing. Even in the as-rolled condition following the cold rolling scheme, the studied ZEWK alloy shows good ductility. The ductility is significantly improved at 200 °C, where the alloy can reach elongations as long as 178 %. About the r-value, the RD direction shows the lowest value among the other directions. This can be related to the basal texture which shows a double peak tilted towards the RD.



CR-AR	RD	30°	60°	TD
σ_0 (MPa)	261.84	267.74	268.86	273.51
ε_{Max} (%)	14.97	20.00	26.42	24.43
UTS (MPa)	275.45	278.70	289.23	296.38

CR-AR	RD	30°	60°	TD
σ_0 (MPa)	111.89	113.07	121.21	130.84
ε_{Max} (%)	178.27	151.88	121.63	110.27
UTS (MPa)	111.89	113.57	121.21	130.84



CR-AR	2%	6%	10%	14%
$\bar{r}$	1.093	0.890	0.926	0.853
Δr	0.165	0.200	0.237	0.370

CR-AR	2%	6%	10%	14%
$\bar{r}$	0.590	0.618	0.631	0.658
Δr	0.125	0.127	0.126	0.134

Fig. 7: Engineering stress-strain curves at RT and 200 °C of ZEWK alloy samples processed by cold rolling scheme. The measured r-values at different strains are also plotted.

Fig. 8 presents the earing behaviour of samples deformed by deep drawing in an Erichsen™ 145-30 sheet metal testing machine using a punch with a diameter of 50 mm at 150 or 200 °C. The test speed and blank holder force were 20 mm/min and 5 kN. Blank diameters of 85 and 100 mm were cut from the rolled and ECAP sheets for two different deep drawing ratios (DR) of 1.7 and 2.0. In Fig. 8 results of 100 mm diameter (DR=2) deformed at 200 °C are presented. To measure the earing development in the deep drawn cups 3-D scanning measurements were performed. As seen in Fig. 8, there is a clear development of ears in the hot rolling scheme in as-rolled condition (HR-AR) and after recrystallization annealing (HR-HT). Interestingly, the earing behavior is significantly reduced in the cold rolling scheme in the CR-AR condition. This can be related to the distinctive basal type of texture with a doble split peak towards the RD. The ease activation of the basal $\langle a \rangle$ slip due to the higher Schmid factor was a key factor to compensate the deformation in RD, leading to an almost ear free cup. As soon as the TD-split texture is developed in CR-HT sample, a stronger earing behavior is observed. Counterintuitive to the balanced work hardening behavior observed in the uniaxial tensile tests, a stronger tendency to develop cup ears is observed in the ECAP processed alloys.

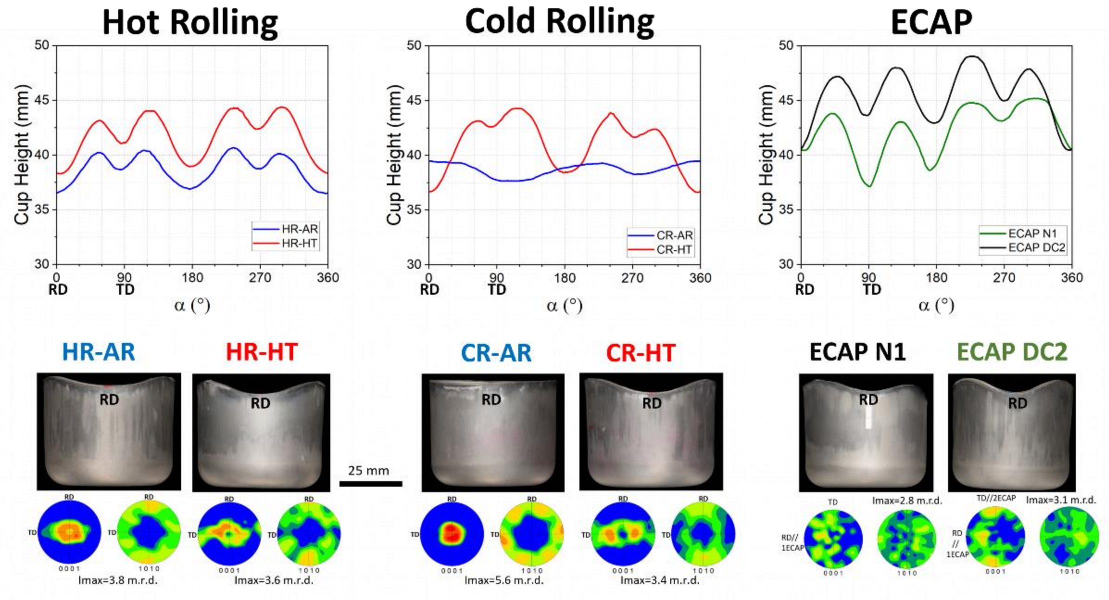


Fig. 8 presents the results of deep drawing experiments of hot-rolling, cold rolling and ECAP pro-cessed samples at 200 °C using 100 mm diameter blanks (DR=2).

The degree of earing behaviour is presented in Fig. 9 using the expression of [Yi10]. As in the ECAP processed samples the earing behaviour is significantly worse than after conventional hot rolling and cold rolling, the last two processing routes were used to draw an overview on the effect of the initial texture of the formable ZEWK alloy, i.e. basal type texture and TD-split texture, relating the normal and planar anisotropy. Contrarily to cubic metals, in this project, as the ears developed around 60° from the RD, the general expression to calculate the anisotropy were used. The results of the r-values and planar anisotropy measured at 200 °C can be observed in Fig. 9. For comparison results from deep drawing experiments using a conventional AZ31 are plotted. Yet, the deep drawing behaviour of the AZ31 alloy was poor at 200 °C and the deformation temperature had to be raised to 250 °C obtain cups without fractures. With regard to the normal anisotropy, it can be inferred that the different CRSS for the activation of different slip modes of the ZEWK and the AZ31 alloy (both having a similar basal type texture) is considerably different. However, a clear tendency is that a basal type texture leads to a larger planar anisotropy, which can result in weaker earing behaviour during deep drawing operations.

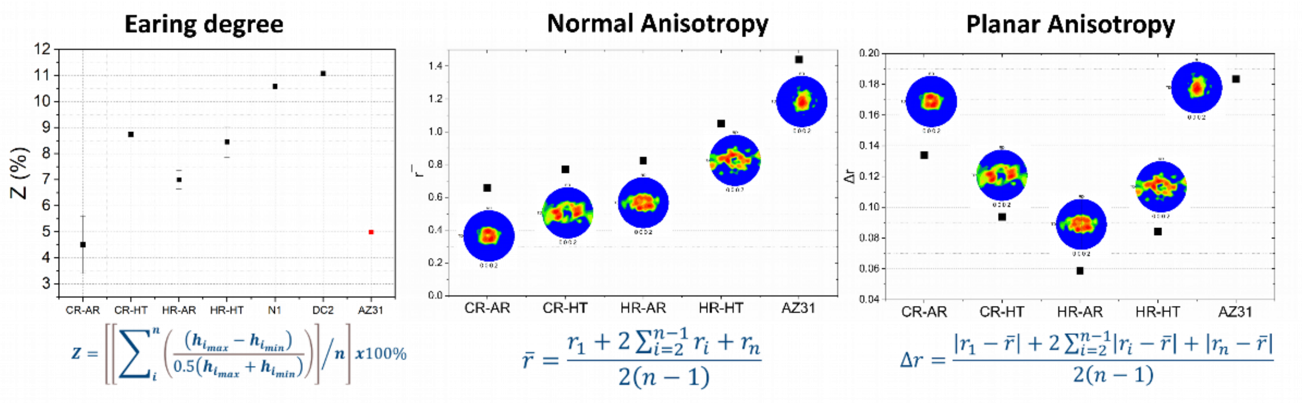


Fig. 9 presents the earing degree, the normal anisotropy and the planar anisotropy of hot-rolling, cold rolling and ECAP processed samples at 200 °C using 100 mm diameter blanks (DR=2). The r_{10}-values obtained from tension test at 200 °C using strain rate of $1x10^{-3}$ s^{-1}.

3.3 Brief summary

In summary, this research project has investigated the effect of different processing conditions on the microstructure and texture development of a novel Mg-Zn-Nd-Y-Zr alloy. One of the important aspects to evaluate was the effect of the introduction of shear strain via ECAP processing to design materials with suitable crystallographic texture for lower anisotropy during forming operations. In this case deep drawing was used as an example. It was found that despite the optimal mechanical behavior, i.e. well-balanced hardening behavior in different directions, the reduction of the planar yield asymmetry, the ZEWK ECAP samples showed an increase in the earing behavior. This can be attenuated by forcing the development of a basal type of texture of this formable alloys by implementing cold rolling. In addition, it has been clearly shown that a larger planar anisotropy would, contrarily to cubic metals, reduce the development of ears for round samples deformed in deep drawing conditions.

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4 Published Project Results

4.1 Publications with scientific quality assurance

- Viktor Böhm, Maximilian Gruber, Elias Abele, Cordula Steinbauer, José Victoria-Hernández, Dietmar Letzig, Noomane Ben Khalifa, Wolfram Volk (2023), Influence of Equal-Channel Angular Pressing on the Microstructure and Texture of Mg-Zn-Y-Zr-RE Alloy Sheets, doi: 10.1007/978-3-031-41341-4_47
- José Victoria-Hernandez, Guadalupe Cano-Castillo, Viktor Böhm, Maximilian Gruber, Wolfram Volk, Noomane Ben Khalifa, Dietmar Letzig (2023), Effect of ECAP Process on the Activation of Deformation Mechanisms During Subsequent Uniaxial Tension of Mg-ZEWK2000 Sheets, doi: 10.1007/978-3-031-42093-1_72

4.2 Other publications and published results

- Dissertation: Maximilian Gruber (2024), Equal-Channel Angular Pressing für industriell anwendbare Aluminiumblechwerkstoffe, ISBN: 978-3-911206-01-3

- Semester thesis (as part of a preliminary investigation to this project): Cordula Steinbauer (2021), Numerical and experimental investigation of equal-channel angular pressing for magnesium sheet metal
- Master thesis: Cordula Steinbauer (2023), Increasing the industrial applicability of magnesium sheets through the use of equal-channel angular pressing
- Bachelor thesis: Elias Abele (2023), Experimental investigations to determine the yield point of rolled magnesium sheets
- Bachelor Thesis: Joseba Larrinaga (2024), Reduction of the anisotropic forming behavior of Mg sheets by using severe plastic deformation
- Presentation in 89st International Magnesium Association Conference Fukuoka, Japan José Victoria-Hernández (2024), Recent Mg sheet development towards high formability and lower anisotropy

4.3 Patents (applied for and granted)

None