Charles University

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HABILITATION THESIS

High-strength Mg alloys: toward low-alloyed alloys with layered structure

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"Learn from yesterday, live for today, hope for tomorrow.
The important thing is not to stop questioning" (AE)

"this is the right way out of the underpass" (KM)

Contents

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Preface

The progress in science and technologies achieved in the 19th and 20th centuries brought many theories and ideas on how the world around us works. It resulted in the development of many functional materials, mathematical patterns, quantum physics models, as well as great modernization of the methods and techniques up to the level that proposed earlier concepts can be confirmed or disproved. These advances include, for example, the development of electron microscopy tools and *in-situ* techniques that, in combination, allow not only detailed observation of microstructural changes but also the detection of dynamic processes in real time.

Advances in materials science can be demonstrated, for example, by the development of magnesium (Mg)-based lightweight alloys, where traditional alloys with conventional mechanical properties are being replaced by novel high-strength alloys. The improvement was partially achieved by introducing multimodal or/and layered microstructure. For instance, developed Mg alloys with a long-period stacking ordered (LPSO) phase exhibit superior mechanical properties at both room and elevated temperatures, making them promising candidates for broader applications. However, it is obvious that further design of safe structural elements, regardless of their intended use, requires a thorough knowledge of deformation mechanisms responsible for mechanical performance under various circumstances, incl. strain-path changes, aggressive environment, or elevated temperatures. Acquiring knowledge of the key mechanisms in novel Mg alloys became the primary motivation and objective of research cases addressed in this thesis, intended to contribute to knowledge-based and application-driven development of high-strength Mg alloys. The application of advanced experimental techniques in combination with computational tools turned out to be essential for obtaining vital information. Besides the scientific importance of the achievements, the selected research cases highlight capabilities and current limitations of applied experimental methods, supporting their progress in tackling modern materials-science challenges.

Besides well-known and widely used electron microscopy and diffraction methods, the acoustic emission (AE) has emerged as powerful, non-destructive *in-situ* technique offering integral information from the entire sample volume. AE is defined as transient elastic waves produced by a sudden release of energy due to a local dynamic change within the material. The capabilities of modern AE devices as well as resources to analyze large amount of data open a window to the detailed study of collective phenomena in condensed matter physics and materials science. For example, AE can shed light on dynamics of deformation processes in crystalline materials. Moreover, by combining AE with high-resolution microscopy, one can now both visualize and "listen to" deformation processes. Combination of tools with a high temporal (AE) and spatial (electron microscopy, diffraction) resolutions is a qualitative step in analysis of the deformation mechanisms, incl. dislocation slip, twinning and/or kinking in Mg(-LPSO) alloys. Modern techniques bring us to another level of understanding the dependence of dislocation activity on the microstructure features such as secondary phases or precipitates. Thus, we are on the way where proposed earlier models of dislocation activity can be confirmed using experimental data. Advanced simulations further complement and validate experimental findings.

This habilitation thesis summarizes selected studies exploring the fundamental processes of plastic deformation in both conventional and modern Mg alloys by means of advanced *in-situ* techniques (AE, diffraction), supported by *in-situ* and *post-mortem* microstructure analyses. Starting with revealing basic deformation mechanisms in pure Mg single crystals, the influence of solutes as well as presence of a few (α-Mg, LPSO) phases on deformation mechanisms (dislocation slip, twinning and kinking) in polycrystalline Mg alloys is discussed.

The thesis consists of three chapters. A brief introduction to the deformation mechanisms in Mg alloys considering microstructure and secondary phases can be found in Chapter 1. Chapter 2 contains my commentary on motivation, experimental procedures and main achievements published previously in 12 selected journal publications, which are thematically divided into three subchapters. The subchapter 2.1. explores twinning, detwinning, and twin-dislocation interactions in conventional Mg alloys with α-Mg lattice. The subchapter 2.2. is dedicated to investigation of deformation mechanisms in modern high-strength Mg alloys with multimodal microstructures containing α-Mg and LPSO phases. The subchapter 2.3. deals with research and development of low-alloyed high-strength Mg alloys prepared by rapidly solidified ribbon consolidation (RSRC) technique. The RSRC processing of low-alloyed Mg alloys results in the formation of individual solute segregated stacking faults (SFs) or their agglomeration in cluster arranged layers or nanoplates instead of completed LPSO phase. Several key aspects related to microstructure features and mechanical performance in these advanced Mg alloys are addressed. General conclusions and future perspectives are presented in Chapter 3. References and a list of publications selected for the habilitation thesis can be found at the end of the thesis, while their reprints are located in the Appendix.

1. Introduction to deformation mechanisms in Mg alloys

The dislocation slip and twinning are main deformation mechanisms covering the plasticity of metallic materials. The activation of ***slip*** systems strongly depends on loading direction with respect to slip plane and slip direction, temperature, and microstructure features. To initiate slip, the critical resolved shear stress (CRSS) has to be reached. The resolved shear stress is determined as the shear component of the applied tensile or compressive stress resolved along the slip plane. The relationship between the resolved shear stress τ_{RSS} and the applied stress σ was first established by Schmid [1] as follows:

$$\tau_{RSS} = \sigma \cdot m = \sigma \cdot (\cos \varphi \cos \lambda_A), \quad (1)$$

where m is the Schmid factor defined by the angles (φ) between loading axis and normal to slip plane and the angle (λ_A) between loading axis and the glide direction. Thus, the Schmid factor (m) is a geometric factor indicating how effectively applied stress is resolved into shear stress on a slip system. The resolved shear stress has a maximum when $\varphi = \lambda_A = 45°$. It is therefore obvious that CRSS for each dislocation slip system depends on the crystal orientation. Consequently, deformation in a single crystalline material initiates at different applied stresses with respect to its orientation [2, 3].

Mg and its alloys, having hexagonal close packed (*hcp*) lattice and the c/a ratio close to the ideal value (1.624) exhibit different deformation behavior compared to materials with cubic lattices. The main reason is the limited number of slip systems available in *hcp* materials at room temperature (RT). It is generally accepted that the basal (0001), the prismatic $\{10\bar{1}0\}$ and pyramidal $\pi1$ $\{1010\}$ slip in the directions $\langle 11\bar{2}0\rangle$(the so-called <a> type slip systems) and pyramidal $\pi2$ $\{11\bar{2}2\}$ slip in the <c+a> direction can be activated during plastic deformation of Mg and its alloys [4, 5]. The CRSS value for the basal slip is significantly lower than that for the other slip systems at RT, and therefore, the basal slip is taken as the first activated mechanism [6-10]. The pyramidal $\pi2$ $(11\bar{2}2)$ <c+a> slip requires either higher applied stress and/or elevated temperatures to be activated. However, according to the von Mises criterion for a homogeneous plastic deformation of polycrystalline materials, at least five independent slip systems are required [11]. In this regard, an additional mechanism, mechanical ***twinning*** providing strain along *c*-axis, plays an important role in plastic deformation of Mg alloys. The twinning mechanism can be described as follows: when the shear stress on a certain crystallographic plane (twin plane) reaches a threshold, atoms at one side of the twin plane move to a new position, while the motion is parallel to the twin plane [12]. As a result, both sides of the twin plane have the same crystal structure but bear different orientations. In another words, twinning in crystalline material results in the formation of domain crystals inside their parent crystals (grains), however, both sharing the same crystal lattice in a symmetrical manner, and these domains are separated by twin plane – twin boundary [12]. While twinning can or cannot contribute to plastic deformation depending on the specific twinning mechanisms, it influences a substantial evolution of microstructure and results in the formation of twin boundaries [12]. Similar to grain boundaries, twin boundaries can effectively strengthen materials by impeding dislocation motion due to the slip discontinuity caused by the mirror symmetry.

Twinning as a process starts from twin nucleation, which is driven by local stress states and local atomic configurations at grain boundaries [13, 14]. Further twin growth is driven by long-range stress states across grains through the motion of twin boundaries. It is realized either by gliding of twin dislocation on the twin plane along the twin direction – ***propagation,*** or migration of twin boundary perpendicular to the twin plane via nucleation and gliding of twin dislocation on the twin plane – ***twin thickening***. Furthermore, when the reversed load is applied, ***detwinning*** (twin shrinkage) can occur. Multiple twins nucleated in the same grain interact with each other, form twin-twin junctions, influencing twin growth or shrinkage, as well as nucleation of new and/or secondary twins [15]. Twinning itself can accommodate up to 6.4% of strain [16]. In addition, the twins can change the orientations of the lattice to become more favorable for the activation of the dislocation slip (basal and prismatic), thus providing additional strain. The most common twinning system in Mg alloys is the $\{11\bar{2}0\}\langle 10\bar{1}1\rangle$ extension twinning, which is activated when external compression is applied perpendicularly to the *c*-axis, and newly created fraction (twin) is characterized by misorientation of 86° with respect to the original lattice [17]. Hence their activation depends on the loading direction and crystallographic orientations of the lattice or, generally speaking, texture (which is preferential orientation of the grains) in the case of polycrystalline materials. Due to this polar nature of twinning and difference in CRSS for activation of particular dislocation slip systems, wrought Mg alloys with strong texture (formed during processing) exhibit pronounced yield anisotropy and asymmetry, which pose challenges for their practical application. Moreover, the twinning-detwinning phenomenon has been found to be the main contributor to the specific strain hysteresis (anelastic behavior) observed during the loading-unloading process [18-20].

Obviously, the anisotropic deformation behavior, controlled by the activity of individual deformation mechanisms, is one of the main limiting factors for the wide application of wrought Mg alloys. Further significant shortcomings of wrought Mg alloys are insufficient strength values, especially at elevated temperatures. These limitations can be partly minimized in conventional Mg alloys by suppressing twinning activity through reducing texture intensity and/or grain size, achieved by using different alloying or advanced processing techniques. Particularly, the issue was partly resolved by developing Mg composites [21] which, however, exhibit a limited ductility. With the aim to further improve mechanical properties (incl. strength and ductility), a new class of Mg alloys with long-period stacking ordered (LPSO) phase has been recently developed (e.g. Mg-Zn-Y [22-27], Mg-Zn-Gd [23], Mg-Ni-Y [28], Mg-Y-Cu [29]). Types of the LPSO phase are defined by a periodic arrangement of the close-packed atomic layers enriched with rare earth (RE) elements and transition metals (TM) in the Mg lattice [30, 31], see **Fig. 1**. In this case, the hard elongated fractions of the LPSO phase provide a fibre-reinforced composite-like strengthening to the alloy. Due to the limited number of active slip systems in the LPSO phase, the ***kinking*** mechanism is activated when compressive load is applied perpendicular to *c*-axis. Importantly, this mechanism significantly contributes to the work hardening [32-35]. Thus, the volume fraction, orientation, and distribution of the LPSO phase have a remarkable influence on mechanical behavior [27, 36-41].

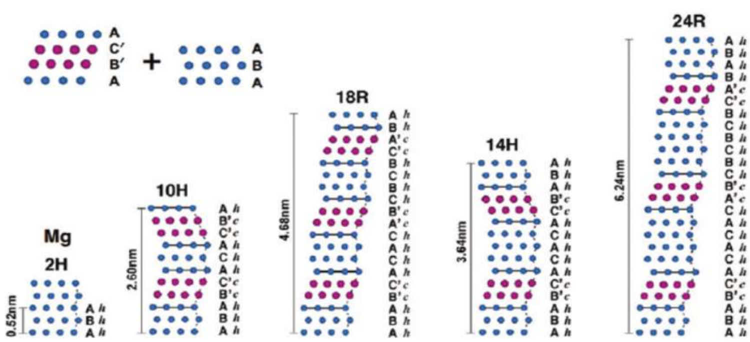

Fig. 1. *Classification of LPSO phases [30].*

Besides introducing the LPSO phase, other strengthening strategies emerged in Mg-TM-RE alloys, including, for example, formation of ultrafine-grained (UFG) microstructure by powder metallurgy [42, 43], extrusion of recycled chips [44, 45], equal channel angular pressing [44], or rapid solidified (RS) ribbon-consolidation [46-48]. In the latest case, individual solute-rich stacking faults (SFs) or their agglomerations are formed besides fine LPSO particles. The solute-enriched SFs, their agglomerations, much like the LPSO phase, inhibit the dislocation motion (similar to dislocation locks, jogs, precipitated particles, twinning, grain boundaries), thereby contributing to the strengthening of the material [33].

Due to the complex microstructure and large variety of possible deformation mechanisms, investigation of deformation behavior of Mg-LPSO alloys is a non-trivial task. Hence advances in currently developed techniques, incl. *in-situ* methods, have to be used, for obtaining desired information about activation and dynamics of deformation mechanisms. In particular, diffraction methods can be used to reveal the evolution of the overall dislocation density [49-51] and fractions of dislocations in particular slip systems [52] by analyzing the diffraction line broadening. Twinning process can be also evidenced by the change of particular diffraction peaks [19, 53, 54]. Modern techniques for *in-situ* microstructure observations, including direct imaging during loading inside the scanning electron microscope chamber or high-speed video recording, can provide real-time information about microstructure changes. The resolution achieved by scanning electron microscopy (SEM) imaging offers observations of microstructure details of underlying processes, such as dislocation slip, (de-)twinning, or kinking [55, 56]. Moreover, employing the *in-situ* AE technique in combination with *in-situ* SEM imaging and diffraction methods offers a powerful complementary approach for studying deformation mechanisms in metallic materials. Notably, the dislocation motion along the slip planes and twinning differ significantly in their source dynamics and, therefore, they generate AE with different waveforms (low-amplitude waveforms fluctuating in a wider range of frequencies, which are produced by dislocation slip, can be compared to high amplitude transients for twinning [57, 58]). To discriminate quantitatively between the sources of different origin in random AE time series, the *adaptive sequential k-means* (ASK) signal clustering algorithm proposed by Pomponi and Vinogradov [59] has been proven effective on many occasions, including our works on Mg alloys (see e.g. [60-63]), Zr alloys [64] or rocks [65]. Most prominent, the AE technique enables characterization of the dynamics of deformation mechanisms with millisecond time resolution.

Altogether, the above-mentioned *in-situ* and *post-mortem* techniques can provide comprehensive information for multiscale analysis of the deformation process in fine temporal and spatial details. The understanding of the deformation processes is of a key importance for the design of novel Mg alloys with superior properties and a high potential for applications.

2. Overview of the results achieved

2.1. Interplay of dislocation slip and twinning in conventional Mg alloys

This subchapter addresses findings related to the activity of main deformation mechanisms in conventional Mg alloys: dislocation slip and twinning. Particular attention is paid to twinning mechanism, its reverse operation (twinning-detwinning) and interaction of twins (and their boundaries) with dislocation slip. The dynamics of these deformation mechanisms, uncovered by *in-situ* SEM observations and advanced statistical analyses of the AE signal, is discussed in **[A1]**. Investigation of the twinning activity resulting in anelasticity of Mg alloys using *in-situ* SEM imaging, neutron diffraction and AE recording is summarized in **[A2]**. The twin boundaries mobility, as a part of twinning-detwinning mechanism, has been also analyzed with respect to intermediate heat treatment **[A3]**. To get more knowledge about twinning mechanisms, the 3D crystal plasticity model of the distribution of dislocation slips and shear stress inside and around an individual twin has been proposed and supported experimentally using high-resolution (HR) EBSD **[A4]**.

[A1] E. Agletdinov, **D. Drozdenko**, P. Harcuba, P. Dobroň, D. Merson, A. Vinogradov, *On the long-term correlations in the twinning and dislocation slip dynamics, **Mat Sci Eng a-Struct*** 777 (2020) 139091. IF: 5.234.

For simplifying the case of polycrystalline materials, the study **[A1]** was performed on Mg single crystals, eliminating the contribution of grain boundaries. In our previous work on single crystals [66], the classical (threshold-based parametrization) approach for the AE data analysis in combination with X-ray diffraction measurements was successfully used for the description of dynamics of individual deformation mechanisms. The experiments were realized on single crystals oriented in the most suitable way to facilitate particular deformation mechanisms under compressive loading. In this way we addressed the operation of solely basal slip or pyramidal slip, and interplay of prismatic slip and twinning. This work became a basis and motivation for further research. With developed possibilities for the data analysis, the AE technique powered by the advanced signal categorization technique was employed in **[A1]** to probe the dynamics of two primary deformation modes: basal dislocation slip and twinning. These mechanisms determine majority of possible scenarios of plastic deformation in structural metallic materials.

The classification power of the ASK algorithm for the AE data analysis was demonstrated to allow predictions of deformation mechanisms with very high confidence, as verified by direct *in situ* SEM observations. While AE provides integral information from the entire volume of the material, the *in-situ* SEM imaging can confirm AE results and visualize even unexpected processes. Particularly, signatures of twinning were revealed by AE in the single crystal preferably oriented for solely basal slip and prompted detailed microscopic observations confirmed the appearance of a few tiny extension twins with secondary basal slip inside twinned region (**Fig. 2**). In this case, twins do not appear to contribute much to global deformation, but they are rather developed to compensate crystal lattice rotation induced by dislocation slip. The independent and non-supervised ASK cluster analysis of AE time series is thus proven to be capable of predictive discrimination between underlying deformation mechanisms, the accurate timing of their activation and evolution (**Fig. 3**).

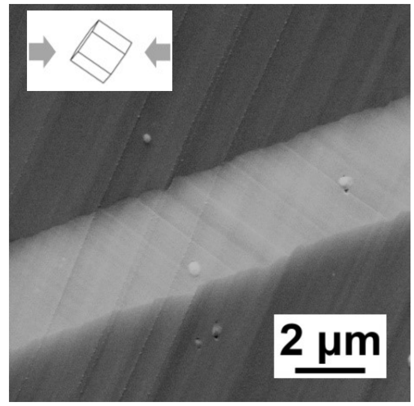

Fig. 2*. Magnified view of the twin revealing traces of the secondary slip in the twinned region. [A1].*

Moreover, statistical analysis of arrival times of AE events in time series has been performed. It is based on the idea that AE signal released in the solids from localized sources due to rapid stress relaxation resembles seismic time series. Following the data processing strategy proposed in [67] for the assessment of possible correlations in the behavior of deformation-induced defects, the AE time series reduces to a point process which is characterized solely by a set of arrival times of events $\{t_0, t_1 \ldots t_i \ldots t_N\}$. However, it is more convenient to investigate the distribution of time intervals between the successive events $\Delta t_i = t_{i+1} - t_i$. Assuming that the sources of AE events are independent, a Poisson-type time series [68] is anticipated in the form of a train of Dirac δ-impulses $\delta(t - t_k)$ with amplitudes U_k:

$$A(t) = \sum_{k=1}^{N} U_k \delta(t - t_k). \quad (2)$$

The inter-arrival time intervals Δt_ishould, therefore, obey an exponential distribution with the probability density function (PDF):

$$\rho(\Delta t) = \frac{1}{\Delta t} exp(-\Delta t/\overline{\Delta t}) = \lambda\, exp(-\lambda \Delta t), \quad (3)$$

where $\overline{\Delta t}$ is the average time interval between the pulses in a time-series (averaging is done over the time interval T) and $\lambda = 1/\overline{\Delta t}$ is the average intensity (also termed as count rate or activity) of the pulse flow on the chosen time interval T. Since the properties of any random process are fully determined by the PDF of the descriptive random variable, the AE stream was sectioned into a set of successive realizations and the PDF was obtained for each realization. Then, the statistical goodness-of-fit test was applied to probe the agreement between the inter-arrival times Δt and the Poisson distribution (3) for each realization.

Statistical analysis of AE signals unambiguously demonstrated that twinning behaved as an intermittent process with the temporal correlation between the events having a relatively long (sub-millisecond and millisecond range) memory of the past. In other words, twinning is a process with a memory of the past, where twinning events affect the occurrence of successive events. In contrast, the basal dislocation slip manifested itself as a substantially random intermittent process comprising of independent elementary slip events without long-term correlations between gliding dislocation(s) or slip bands emerging caused by the collective dislocation slip (on basal planes). **Fig. 3** illustrates the correlation of statistical analysis of arriving time series of AE events (**Fig. 3a**) with ASK clustering analysis (**Fig. 3b**) of AE signal recorded during deformation of Mg single crystal in orientation prone for concurrent activity of twinning and slip. (It should be emphasized that these two AE data processing surveys are independent in its nature). The dashed line indicates a cross-over between the two primary deformation modes, which coincides with the inflexion point on the deformation curve.

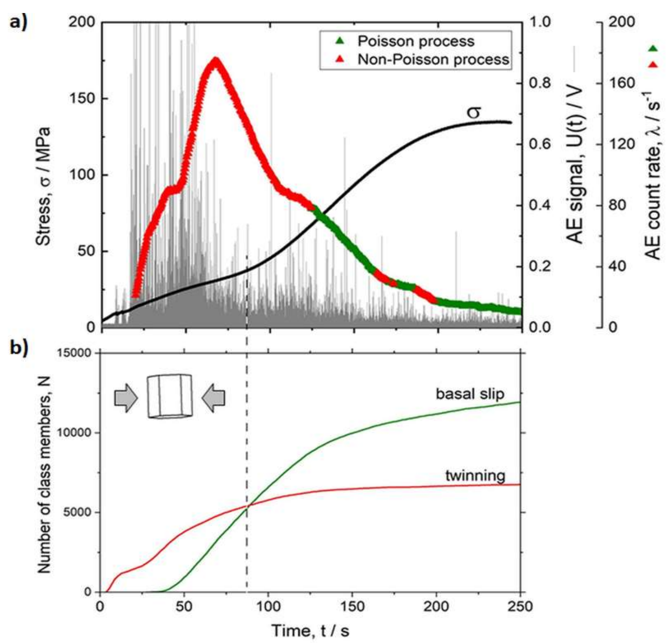

Fig. 3. *AE activity revealing deformation mechanisms in the Mg single crystal [A1].*

[A2] **D. Drozdenko**, J. Čapek, B. Clausen, A. Vinogradov, K. Máthis *Influence of the solute concentration on the anelasticity in Mg-Al alloys: A multiple-approach study* ***J Alloy Compd*** 786 (2019) 779-790. IF: 4.65.

The anelastic mechanical response, caused by twinning-detwinning processes and interaction with dislocation slip, is ubiquitous for deformation behavior of Mg and its alloys. In the paper **[A2],** the complex twinning-detwinning processes have been studied using a rare blend of advanced *in-situ* techniques with different spatiotemporal resolutions.

Particularly, the AE monitoring (with following ASK analysis as was performed in [A1]), neutron diffraction (ND) measurements, *in-situ* SEM and high-speed microscopic video imaging enabled access to otherwise inaccessible fine microstructural details of the underlying deformation mechanisms. Within this study, the influence of the solute content (in this case Al) on the anelastic behavior during cyclic loading was characterized, as well. From practical point of view, the applicability, advantages, and disadvantages of applied *in-situ* techniques for investigation of the deformation mechanisms in metallic materials were discussed. The work was partly motivated by our previous studies [69, 70], where AE technique and *pseudo in-situ* EBSD analysis were performed for studying twinning-detwinning mechanisms with respect to bimodal or homogeneous microstructure.

The *in-situ* SEM and high-speed imaging helped to visualize in **[A2]** the microstructure processes underlying the AE behavior in different stages of the loading-unloading hysteresis loops. The strongest AE response was observed in Mg alloys with higher alloying concentration (Mg-9wt.%Al). Notably, it was revealed that the reduction of the twin length during unloading is a high-speed process accompanied by detectable AE transients (**Fig. 4**). On the reloading stage, the nucleation of new twins contributes to detectable AE. As opposed to this, in pure Mg and Mg-2wt.%Al alloy, only the slow movement of existing twin boundaries (twin thickening and thinning) occurs, and this process does not generate detectable AE (red arrows in **Fig. 4**). These findings help to complete the existing knowledge of twinning as AE source. Previously, it was specified that twin nucleation and propagation (increase in lateral length) is a strong source of AE, while in/decrease in thickness does not contribute to AE [71]. However, the situation with detwinning with final collapse (i.e., complete disappearance) of twin was not clear (**Fig. 4**).

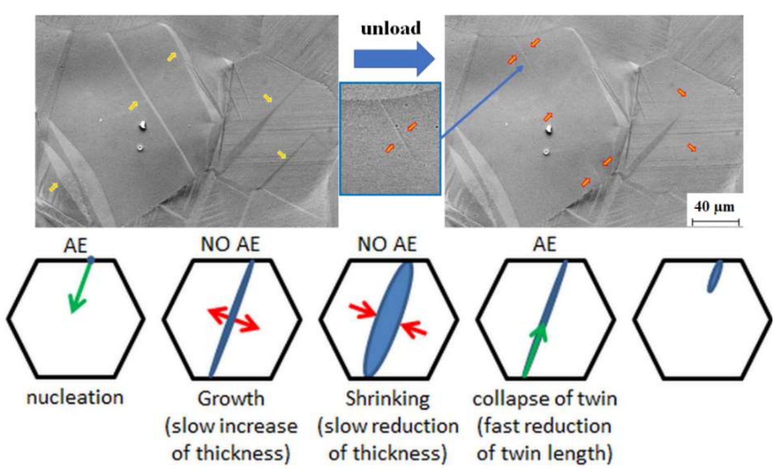

***Fig. 4**. In-situ SEM imaging of Mg9Al alloy and illustration of different stages of twinning-detwinning process accompanied by AE [A2].*

It is important to note that ND significantly contributes to such research by providing statistically relevant datasets from (large) sample volume, what can be complemented by (rather local) 2D SEM and high-speed camera observations. The change in the twinned volume fraction measured by ND was found to be directly proportional to calculated anelasticity of investigated alloys, which confirms a connection of the twinning-detwinning process with anelasticity effects. At the same time, the *in-situ* SEM imaging remains a powerful method for tracking and visualization of the dislocation slip activity, providing a high spatial resolution of the surface topography. High-speed microscopic video imaging brings particular benefits in estimating the speed of twin propagation (in length) and growth/shrinkage (in thickness). A detailed study on twin propagation rate has been reported in our work [72], where experimental observations are well supported by molecular dynamic simulation, indicating that twin propagation rate is of the order of 10^1 m/s. The findings are in agreement with a previous work [56]. Moreover, it was shown in [72], that at common strain rates ($\sim 10^{-3}$ s^{-1}) twin propagation rates decrease with increasing alloying (in that case Gd) concentration. The molecular dynamics simulations revealed that the twin boundaries can be pinned by Gd solutes, leading to intermittent movement of the twin front, in which the slow propagation periods are followed by quick jumps between the obstacles.

[A3] D. Drozdenko, P. Dobroň, S. Yi, K. Horváth, D. Letzig, J. Bohlen *Mobility of pinned twin boundaries during mechanical loading of extruded binary Mg-1Zn alloy* ***Mater Charact*** 139 (2018) 81-88. IF: 3.220.

During twinning(-detwinning) process the mobility of twin boundaries and its restriction due to pinning on solute atoms or precipitates play a remarkable role. Moreover, Nie et al [73] showed significant increase in strength by segregation of Gd, Y, Nd at grain and twin boundaries. However, the question remained unsolved: can we stop or at least control twin boundaries during ongoing or reverse loading after they are pinned (e.g., by application of intermediate heat treatment). Our earlier works on the twinning-detwinning process without isothermal aging ([69, 70] as well as [A2]) gave a good general basis for study of twinning-detwinning process in terms of twin boundary mobility reported in **[A3]**.

In **[A3]**, the mobility of twin boundary pinned by solute segregation and precipitates (introduced by isothermal aging) was studied during subsequent tensile or compressive loading of a binary Mg-1wt.%Zn alloy. During re-compression of pre-compressed and heat-treated samples, i.e., re-twinning, both migration of pinned twin boundary as well as nucleation of new twins were observed. In case of applying opposite direction of loading and, thus, stimulating reverse twin boundary migration, the detwinning is forced to proceed either by (i) nucleation of new twins inside already existing twin lamellae or by (ii) migration of pinned twin boundary, similarly to the case of no heat treatment applied. The migration of pinned twin boundaries can be realized either simultaneously from both boundaries or, as a special case, by the motion of only one twin boundary. It can be concluded that segregation of solute atoms along twin boundaries initiated by annealing at 200 °C for 8 h does not completely disrupt the mobility of twin boundaries. Twin boundaries maintain the ability to propagate in both directions, for twin thickening or shrinkage. However, new twin nucleation inside the grain during reverse tensile loading is significant, which highlights the importance of distribution of internal strains and presence of potential seeds for activation of new twins at the expense of moving pinned twin boundary.

Similar microscopy approach to study twin boundaries mobility has been applied on ternary Mg alloys with 1wt.%Zn and up to 1wt.% of Ca or Nd [74], where similar trends in twin boundaries mobility have been observed. Moreover, it has been reported [74, 75] that the AE technique is also sensitive for unpinning the twin boundaries.

[A4] F. Siska, **D. Drozdenko**, K. Máthis, J. Cizek, T. Guo, M. Barnett, *Three-dimensional crystal plasticity and HR-EBSD analysis of the local stress-strain fields induced during twin propagation and thickening in magnesium alloys,* ***J Magnes Alloy*** 11 (2) (2023), 657-670. IF: 17.6.

This work was motivated by works on Mg micropillars [76], bulk single crystals **[A1],** and polycrystalline materials (e.g., **[A2, A3]** or [69, 70, 77]), where dynamics of twinning process were addressed. Particularly, it was reported that twins are limited in their thickening, and, at certain stress level, the nucleation of new twins is promoted instead of further thickening of already existing twins (see right grain in **Fig. 4)**. Thus, families of twins of the same type are formed inside a grain interior. Twin propagation is initially driven by stress release inside the parent grain; and it stops once the twin reaches a grain boundary, obstacle, or an end of volume inside grain that has a sufficiently high stress to drive twin propagation [78-80]. Twin thickening increases induced back stress inside the twin, which can be relieved by an increase of applied stress or by increased plastic slip around the twin [81, 82]. Thus, an existing twin significantly changes the stress state in the grain, affecting the nucleation, propagation, and thickening of other twins.

Present work **[A4]** was designed to provide a picture of the shear stress and plastic slip distribution inside and around the twin as a function of twin shape, CRSS for dislocation slip and grain size. An individual $\{11\bar{2}0\}\langle 10\bar{1}1\rangle$ tensile twin, represented as an ellipsoidal inclusion surrounded by the matrix, has been analyzed using a 3D crystal plasticity model. This analysis provides information on the propagation and growth of twins at the mesoscale (grain size scale) and complements the nanometer scale insights gained from the molecular dynamic models. Systematic work includes stress/strain analysis for five different twin thicknesses and three different lateral twin lengths. To verify numerical results, the distribution of geometrically necessary dislocations and stress fields inside and around a single twin in AZ31 alloy (selected as a model material) have been investigated using the HR-EBSD analysis. In the core of this analysis is a cross-correlation of small shifts in many regions of interest of the Kikuchi patterns, which determines the variation of elastic strain and lattice rotations across the EBSD map.

It was shown that the plasticity induced inside the twin is mainly caused by the prismatic dislocation slip and has no influence on the twin back stress, which is identical to that of a purely elastic twin. Basal slip around the twin influences twin thickening while prismatic and pyramidal slip influence lateral twin propagation. Furthermore, it was found that twins can reach a maximum thickness that is driven by the CRSS values for dislocation slip with the significant influence of basal slip. Obtained results complement knowledge regarding dynamics of twins and their growth in three directions. The molecular dynamics simulations predict that the fastest growth is in a lateral direction (in the twinning plane and perpendicular to twinning shear). The second fastest one is the forward propagation (in the twinning plane and parallel with twinning shear) and the slowest process is the growth perpendicular to the twinning plane (i.e. twin thickening), see, e.g., [83].

Summary remarks on the publications [A1-A4]

Publications **[A1–A4]** have significantly contributed to the understanding of deformation mechanisms, incl. twinning and dislocation activity, in single- and polycrystalline Mg alloys using statistical analysis of the AE data and SEM observations. Particularly, work on single crystals **[A1]** unambiguously demonstrated that twinning behaved as an intermittent process with a relatively long memory of the past (i.e., twinning events affect the occurrence of successive events), while the basal dislocation slip manifested itself as a substantially random intermittent process consisting of independent elementary slip events without long-term correlations. For the investigation of the anelastic mechanical response of Mg-Al alloys **[A2]**, we were the first who applied a rare blend of advanced *in-situ* techniques with different spatiotemporal resolutions, which enabled access to otherwise inaccessible fine microstructural details of the underlying deformation mechanisms. Particularly, it was for the first time shown that the reduction of the twin length (complete detwinning or collapse) produces detectable AE signal, which completes previous knowledge of twinning as AE source. In the context of the twinning-detwinning process, the mobility of twin boundary and its possible restriction due to pinning on solute atoms or precipitates has been discussed in **[A3]**. Despite of the fact that other research groups reported the opposite, we showed that a segregation of solute atoms along twin boundary does not completely disrupt its mobility. It opened more questions regarding the importance of distribution of internal strains, what was partly solved in **[A4]**, where a 3D crystal plasticity model in combination with HR EBSD experimental observations provided an insight into the shear stress and plastic slip distribution inside and around the twin. Nevertheless, remaining issues in this area stimulate further research, while the ongoing advancement of experimental and computational tools may offer valuable support, thereby keeping this topic among my key scientific interests.

Statement of the author's contribution to the publications [A1-A4]

In scope of research summarized in **[A1-A4]** I have significantly contributed to the proposals of all experiments and to physical interpretations of the results. My responsibilities included complex microstructure characterization **[A1-A4]** and use of advanced microscopy techniques (including *in-situ* SEM imaging during mechanical loading inside SEM chamber **[A1, A2])** for identification of the interplay of dislocation slip and twinning(-detwinning) process in single crystals **[A1]** or polycrystalline materials **[A2-A3]**. As to the results reported in **[A4]**, I contributed by performing HR-EBSD analysis using cross-correlation algorithm in order to provide an experimental validation of the 3D crystal plasticity model of the local stress-strain fields induced during twin propagation and thickening. Besides microstructure analyses, I accomplished deformation experiments **[A1, A3, A4]**, including *in-situ* AE measurements with following participation in statistical analysis of the AE data (*post-mortem*) **[A1]**. The papers **[A2, A3]** were written by me and I significantly contributed to manuscripts preparation of **[A1, A4]**.

2.2. New generation of high-strength alloys: Mg alloys with LPSO phase

The present section is dedicated to identification of deformation mechanisms in high-strength Mg-LPSO alloys at room and elevated temperatures. Wrought processing (e.g., extrusion) of Mg-LPSO alloys leads to the formation of a multimodal microstructure with a fine and coarse α-Mg grains and block-shaped LPSO phase fractions elongated along the extrusion direction (ED), see **Fig. 5**. Such coexistence of the fine dynamically recrystallized (DRX) α-Mg grains and coarse non-DRX grains were found to be especially effective in enhancing mechanical properties: fine DRX grains contribute to ductility, while coarse worked grains and an LPSO phase improve the strength of the alloy [22]. The performance of Mg-LPSO alloys significantly depends on volume fraction of LPSO phase [38, 84, 85] as well as deformation temperature [86].

Fig. 5. *Microstructure of extruded Mg-LPSO alloy (Mg-2at.%Y-1at.%Zn).*

Papers **[B1-B3]** provide summarized information about plastic deformation of Mg-LPSO with respect to orientation of LPSO phase, its volume fraction and deformation temperature. The physical aspects of deformation mechanisms were investigated using a combination of advanced *in-situ* methods and techniques, including *in-situ* SEM observation, diffraction measurements, and AE monitoring.

[B1] G. Garcés, K. Máthis, J. Medin, K. Horváth, **D. Drozdenko**, E. Oñorbe, P. Dobroň, P. Pérez, M. Klaus, P. Adeva *Combination of in-situ diffraction experiments and acoustic emission testing to understand the compression behaviour of Mg-Y-Zn alloys containing LPSO phase under different loading conditions* ***Int J Plasticity*** 76 (2018) 166 –185. IF: 5.8.

This work addresses the effect of orientation of the non-DRX grains and the LPSO phase on the plasticity of Mg-2at.%Y-1at.%Zn alloy. The combination of *in-situ* synchrotron diffraction, AE experiments, SEM imaging, and *post-mortem* EBSD analysis was used to describe deformation mechanisms and reinforcing capacity with respect to the loading direction of the alloy with anisotropic microstructure given by directional processing (in this case, extrusion). As common for extruded Mg-LPSO alloys, the microstructure consisted of fine and coarse α-Mg grains and LPSO fractions elongated along ED. The specimens were compressed along ED, transverse direction (TD) and along direction tilted by 45° from ED to TD. The volume fraction of the LPSO phase calculated from SEM images and tomography observations was 19 and 25%, respectively. Moreover, basal planes of non-DRX grains and LPSO phase were oriented preferentially parallel to ED, which promoted activation of twins in non-DRX grains and kinking in LPSO phase during compression along ED. The activities of deformation mechanisms during loading were elucidated based on the evolution of elastic strains calculated from synchrotron diffraction data. In general, the lattice strain ε_{hkl} can be calculated as:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}}, \tag{4}$$

where d_{hkl} is the lattice spacing for a plane with the given Miller indices when the crystal is under deformation and $d_{0,hkl}$ is the planar spacing for a crystal in initial state. However, the kinking process cannot be easily observed in *in-situ* diffraction experiments due to low intensity diffraction peaks corresponding to the LPSO phase. Therefore, the use of the AE technique, which is sensitive to kinking, became beneficial in this case. In addition, the *in-situ* SEM imaging and *post-mortem* observation were used to complete the research. The combination of experimental techniques made it possible not only to distinguish particular deformation mechanisms, but also to correctly determine the onset of their activation and the subsequent development of their activity.

It was shown that, independently on the loading direction, the beginning of macroscopic plasticity is always controlled by the activation of the basal slip in the DRX grains. However, due to preferential orientation, non-DRX grains and the LPSO phase have a strong influence on the compressive deformation of the alloy. Particularly, the {10-12} <10-1-1> extension twinning occurs in non-DRX grains before reaching the macroscopic yield stress. These twins were found to be most significant during compression along ED. In the other two loading directions, TD and 45, the activation of non-basal slip systems took place. The AE results indicated the activations of non-basal slip systems after the macroscopic yield stress has been reached. It was also revealed that the reinforcing effect of the elongated LPSO phase is the most effective when the loading axis is aligned with the LPSO fibers direction (|| ED) since the load transfer mechanism is most effective. It leads to the highest compressive strength in ED by comparison to other loading directions. However, at a high stress level (above 400 MPa), the LPSO phase can be plastically deformed by kinking. This deformation mechanism has been determined by the ASK procedure and confirmed by *in-situ* SEM imaging. The Mg-LPSO alloy hence behaves as metal matrix composite where the LPSO phase acts as the reinforcement leading to the high strength.

[B2] K. Fekete, G. Farkas, **D. Drozdenko**, D. Tolnai, A. Stark, P. Dobroň. G. Garcés, K. Máthis, *The temperature effect on the plastic deformation of the Mg88Zn7Y5 alloy with LPSO phase studied by in-situ synchrotron radiation diffraction,* ***Intermetallics*** 138 (2021), 107321. IF: 4.075.

The activity of deformation mechanisms in Mg alloys are strongly dependent on temperature, and in case of Mg-LPSO alloys, also on the fraction of the LPSO phase. The research reported in **[B2]** addresses the temperature dependence of the compressive deformation behavior of the Mg-LPSO alloy with a high-volume fraction (~85%) of the LPSO phase. The LPSO phase, identified as the 18R polytype, is represented by wavy lamellae elongated along ED. It has a pronounced basal texture like non-DRX grains. For identification of the deformation mechanisms, the combination of *in-situ* synchrotron radiation diffraction with *post-mortem* SEM microstructure analysis was used in analogy to **[B1]**.

The investigated alloy compressed along ED at room temperature shows a superior yield strength (480 MPa). With increasing deformation temperature, the yield strength is reduced (by 15% at 200 °C and by 46% at 300 °C), however, it is still superior by comparison to conventional Mg alloys. It was found that independently of the test temperatures, the basal slip is activated in the α-Mg matrix far below the yield strength. The synchrotron radiation diffraction data indicated that shortly before the macroscopic yielding, the activation of the basal and pyramidal (1st and 2nd order) slip on the [42.8}$_{\mathrm{LPSO}}$ planes, and the non-basal slip on the [42.5}$_{\mathrm{LPSO}}$ planes creates sufficient conditions for the activation of the deformation kinking. Thus, in contrast to alloy described in **[B1]**, the macroscopic yielding of the alloy is controlled by the activation of deformation kinking in the LPSO phase due to the higher amount of LPSO phase. Although kinking is one of the most significant deformation mechanisms controlling the plastic deformation, its effect is partially suppressed during deformation at 300 °C. The diffraction data indicated the stress localization at kinks. Unlike twinning, kink formation does not induce a significant drop of residual stress in newly generated kinks. This explains the increase in lattice strains with plastic deformation. Moreover, for the first time, a rough estimation of kink volume fraction was done using the diffraction data. The analysis was done using the principles for the calculations of the twin volume fraction [4, 79, 87]. Due to the limitation of diffraction pattern analysis for Mg-LPSO alloys (given by superposition of the LPSO and α-Mg phase diffraction peaks) and no defined orientation between the kinked and original lattice (like in the case of twinning), the calculated data should be rather taken as an approximate lower limit for the kinked volume. Besides slightly delayed onset of kinking at 300 °C, the obtained results show comparable values for kinking at all tested temperatures (up to 300 ◦C) and similar evolution of kink volume as a function of strain.

The investigation of the kinking process was also extended in our other works dedicated to the description of uncommon <0001>-rotation-type kink [88]. In addition, a large-strain mathematical model for the formation of kink bands in layered systems was proposed in [89], where the numerical simulation was corroborated by experiments performed on a stack of papers.

[B3] K.H. Fekete, **D. Drozdenko**, J. Čapek, K. Máthis, D. Tolnai, A. Stark, G. Garcés, P. Dobroň, *Hot deformation of Mg-Y-Zn alloy with a low content of the LPSO phase studied by in-situ synchrotron radiation diffraction,* ***J Magnes Alloy*** 8(1) (2020), pp. 199–209. IF: 10.088.

In contrast to **[B2]**, present paper **[B3]** addresses the temperature dependence of the compressive deformation behavior of the Mg-LPSO alloy with a low (10%) fraction of the LPSO phase. Similar to **[B2]**, the deformation mechanisms were uncovered using *in-situ* synchrotron radiation diffraction technique at temperatures between room temperature and 350 °C. The microstructure study was completed by detailed investigations using SEM, particularly the backscattered electron imaging and EBSD mapping.

The results show that yielding is controlled by the activation of extension twinning in elongated non-DRX grains and by basal slip in the DRX grains up to 300 °C. With increasing deformation temperature, twinning lost its dominance and kinking of the LPSO phase became more pronounced. However, twinning is still active at 300 °C. The LPSO phase loses its strengthening effect at 350 °C. At the same time, the dynamic recrystallization of the α-Mg matrix became evident already at 300 °C, what significantly affects the deformation behavior of the material. The coherent interface between the Mg matrix and the LPSO phase also reduces the chance of micro-cracks or debonding of the LPSO phase above 200 °C. Most importantly, present work shows that kinking becomes more significant at elevated temperatures even in an alloy with a low volume fraction of the LPSO phase.

Summary remarks on the publications [B1-B3]

The research dedicated to novel Mg-LPSO alloys, having a layered microstructure on the macroscale, is introduced in **[B1-B3]**. Particularly, the effect of orientation of LPSO phase **[B1]**, volume fraction and deformation temperature **[B2-B3]** on plastic deformation and thus performance of this class of Mg alloys have been addressed. The combination of *in-situ* synchrotron diffraction, AE, SEM imaging as well as *post-mortem* EBSD analysis has been used to elucidate main deformation mechanisms, especially kinking process and the reinforcing capacity of the LPSO phase. The evolution of elastic strains estimated from diffraction data together with statistical analysis of AE helped to accurately determine the time of activation and subsequent development of deformation mechanisms. Particularly, it was shown that the kinking plays a key role even in an alloy with a low volume fraction of the LPSO phase, while its effectiveness increases at elevated temperatures. Moreover, for the first time based on obtained diffraction data, a rough estimation of the kink volume fraction during compression was made using similar strategy as for the calculations of the twin volume fraction in conventional Mg alloys.

Statement of the author's contribution to the publications [B1-B3]

Obtained knowledge of deformation mechanisms in Mg alloys with pronounced texture formed during processing I used for research on high-strength Mg alloys with LPSO phase summarized in **[B1-B3].** I took part in the design of all experiments and physical interpretations of the results **[B1-B3]**. My expertise helped me to perform myself or/and to lead students in complex microstructure characterization using electron microscopy **[B1-B3]** as well as using the *in-situ* SEM imaging **[B1]** for analysis of the interplay of dislocation slip, twinning, and kinking mechanisms in Mg alloys with multimodal microstructure. Besides microstructure analyses, I participated in realization of deformation testing with AE measurements and following AE data processing (including ASK cluster analysis) **[B1]**. I also contributed to interpretation of the diffraction data **[B2, B3]** and their correlation with the microstructure development. Moreover, I took part in the manuscripts preparation **[B1-B3]**.

2.3. Low-alloyed Mg-LPSO alloys prepared by rapidly solidified ribbon consolidation technique

Development of low-alloyed Mg alloys is desirable from an economic point of view and biocompatibility. In order to maintain the superior mechanical properties of Mg-LPSO alloys with reduced amount of alloying elements, the application of modern processing techniques can be used, as mentioned in the introduction. The RSRC processing of low-alloyed Mg-TM-RE alloys results in the formation of a bimodal microstructure consisting of fine DRX and coarse non-DRX grains, see illustration in **Fig. 6**. Moreover, in low-alloyed alloys, the alloying elements are not sufficiently abundant to form completed LPSO phases. Therefore, the individual solute-rich SFs or their arrangement in cluster arranged layers (CALs) and nanoplates (CANaPs) or fine LPSO phase fractions are formed in basal planes of hcp α-Mg lattice and randomly distributed in the grain interior (**Fig. 6a**). As illustrated in **Fig. 6b** (taken from bachelor thesis of my student [90]), SFs are defined as single-layer planar defects enriched with solute atoms, while CALs are two-layer discontinuities in the stacking sequence. The LPSO phase can thus be considered as an arrangement of CALs with a long periodicity. When the stacking arrangement is not periodic, more specifically the CALs are separated by 1 to 4 atomic layers of Mg, the structure is referred to as CANaPs [23, 91]. Such a microstructure significantly contributes to the improvement of the alloy properties.

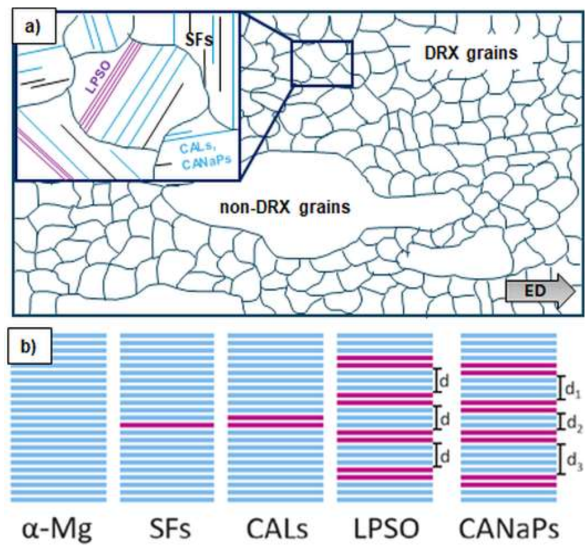

Fig. 6. *Schematic of the microstructure in low-alloyed RSRC Mg-LPSO alloys (a) with illustration of SFs, CALs, LPSO, and CANaPs (b), where lines represent basal planes of hcp lattice [90].*

This subchapter is dedicated to research and development of the low-alloyed high-strength Mg alloys prepared by the RSRC technique. Besides characterization of microstructure and mechanical properties, the general aspects of deformation mechanisms with respect to solute segregated SFs, CALs, CANAPs distributed in Mg matrix (instead of fully formed LPSO phase) are revealed **[C1, C2]**. Specifically, in **[C1]** we addressed the relationship between microstructure and tensile mechanical properties of the set of Mg-Zn-Y alloys with variation in alloying content. In **[C2]** the explanation and possible reduction of tensile-compression asymmetry at the macroscopic yield strength in developed Mg-Zn-Y/Gd/Nd alloys was present. Our attention has been also paid to the nature of yield point phenomena in UFG alloys **[C3]**. Furthermore, the effect of microstructure on active deformation mechanisms during tension of low-alloyed RSRC Mg-LPSO alloy was studied using *in-situ* synchrotron X-ray diffraction measurements, including line profile analysis for a detailed study of the dislocation arrangement **[C4]**. In addition, the microstructural stability in developed Mg alloys with respect to post annealing treatment were studied in detail in **[C5]**.

[C1] **D. Drozdenko**, M. Yamasaki, K. Máthis, P. Dobroň, P. Lukáč, N. Kizu, S-I Inoue, Y. Kawamura *Optimization of mechanical properties of dilute Mg-Zn-Y alloys prepared by rapid solidification*, ***Mater & Design*** 181 (2019) 107984. IF: 6.289. doi: 10.1016/j.matdes.2019.107984.

This work summarizes optimization of mechanical properties of low-alloyed Mg-Zn-Y alloys prepared by RSRC techniques, where consolidation of rapidly solidified ribbons were realized by hot extrusion. Previous research activities [42] showed that even small amount of Zn and Y alloying (up to 2 at.%) can lead to exceptional mechanical properties. However, owing to better biocompatibility and from the economical point of view further reduction of rare earth (RE) alloying elements motivated the design of low-alloyed RSRC alloys. To preserve good mechanical properties of Mg-Zn-RE alloys with reduced alloying elements, optimization of processing parameters (such as temperature or extrusion speed) must be considered, as well. Therefore, we addressed the influence of both the concentration of alloying elements and the metal flow rate during extrusion on the microstructure and resulting mechanical properties. Alloys with Zn and Y concentrations varied between 1 and 2 at.% were investigated. The RSRC processing of low-alloyed material resulted in the formation of ultrafine DRX and worked non-DRX α-Mg grains with Zn- and Y-rich SFs segregated in the basal planes and dispersedly distributed in the grain interior. The average grain size of investigated alloys varied between 700 and 1200 nm. The overall texture is weak due to random orientation of small DRX grains. However, slightly higher texture intensity around $\langle 10\bar{1}0\rangle$ pole originating from non-DRX grains is observed.

In general, for such materials, superior mechanical properties (high yield and ultimate strength in tension) can be explained by a nanoscale grain size and highly dispersive solute segregated SFs inside these grains. In the course of the present study, the pseudo *in-situ* SEM/EBSD imaging was employed, as was successfully used for conventional Mg alloys or alloys with completed LPSO phase. However, the UFG microstructure with segregation discontinuities in case of low-alloyed Mg-LPSO alloys brings strong challenges. To elucidate the behavior of alloys extruded at a different metal flow rate, tensile properties were correlated to inhomogeneous distribution of KAM angle values. It was concluded that a bimodal microstructure, which exhibits a variability not only in grain size but also in internal strain distribution, can lead to significant improvement in the mechanical properties, including moderate elongation. High yield strength can be explained by deformation in elongated non-DRX grains with a higher internal strain (i.e., high dislocation density). Further deformation transits to recovered fine DRX grains with low KAM, where basal and non-basal dislocation can be easily activated resulting in a high elongation. Our following research on corrosion performance of these materials [92] showed that inhomogeneous internal strain distribution in the bimodal microstructure is of a key importance not only for mechanical properties but also for the corrosion behavior.

We have shown that even a small change in processing parameters can be effective for improving the strength and ductility of low-alloyed Mg-Zn-Y alloys. Within all investigated alloys the optimal tensile properties (yield strength and elongation to failure of 362 MPa and 18.2%, respectively) were achieved for Mg-0.56Zn-1.5Y (at.%) alloy extruded at a metal flow rate of 1.9 s^{-1} and having an average grain size of 912 nm. The selected alloy has comparable tensile properties as those for the record holders Mg-1Zn-2Y alloy and Mg-0.5Zn-1Y (at.%) alloy prepared by powder metallurgy [42, 44]. Thus, the Mg alloys processed by RSRC techniques are economically favorable alternative to the materials produced by powder metallurgy. Exceptional mechanical performance of developed materials indicates promising application potential.

[C2] **D. Drozdenko**, K. Fekete, P. Dobroň, G. Németh, J. Veselý, S. Nishimoto, M. Yamasaki, Y. Kawamura *The microstructure and anisotropic deformation behavior of rapidly solidified ribbon consolidated Mg-Zn-X (X=Y, Gd, Nd) alloys,* ***J Alloy Compd*** 944 (2023) 169175. IF: 6.371 doi: 10.1016/j.jallcom.2023.169175.

Previous research **[C1]** motivated the following development of low-alloyed Mg alloys processed by the RSRC technique. Particularly, there was a question about the effect of the alloying elements (Y, Gd, and Nd) on the microstructure and resulting anisotropy of mechanical properties of RSRC Mg-Zn-based alloys. Thus, the RSRC processing has been applied on low-alloyed Mg-Zn-Y, Mg-Zn-Gd, and Mg-Zn-Nd alloys with the content of alloying elements up to 1.5 at%. The processing technique leads to a significant grain refinement together with formation of solute-enriched SFs and cluster-arranged layers

CALs and substantial precipitation with respect to alloying elements. In general, the small grain size and overall weak texture, caused by the random orientation of small DRX grains, resulted in relatively high yield strength and moderate elongation. Moreover, a reverse tension-compression yield asymmetry was achieved owing to the microstructure features, namely the presence of different types of precipitates and/or CALs of solute atoms-rich SFs in the α-Mg matrix. For instance, in the Mg-0.54Zn-1.48Y (at%) alloy characterized by the average grain size of DRX grains of 540 nm, the formation of CALs and precipitates led to enhanced mechanical properties both in tension and compression. However, the tension-compression yield point asymmetry in this alloy is relatively high - about 81 MPa. It was shown that replacing Y by Gd or Nd leads to a reduction in yield point asymmetry with minimal loss in the yield strength values. The Mg-0.56Zn-1.43Gd (at%) alloy is characterized by a slightly higher grain size of the DRX grains (650 nm), however, reduced tension-compression asymmetry down to 37 MPa with keeping TYS of 381 MPa and tensile elongation to fracture of 17 % can be achieved. The use of Nd as an alloying element has a significant influence on the microstructure evolution during processing, and the high density of Mg_3Nd precipitates of different sizes (at the expense of solution-enriched SFs) leads to the suppression of the recrystallization process. Despite a slight increase in the yield strength value, the elongation to fracture of Mg-Zn-Nd alloy decreases dramatically due to the presence of brittle intermetallic Mg_3Nd precipitates. Thus, the use of Gd, as an alloying element in low-alloyed Mg-Zn-based alloys prepared by the RSRC technique, proves to be the most suitable for reducing the tensile-compression asymmetry with minimized loss in mechanical properties.

[C3] **D. Drozdenko**, K. Fekete, P. Dobroň, M. Knapek, K. Máthis, P. Minárik, M. Yamasaki, Y. Kawamura, *The yield point phenomenon in ultrafine-grained dilute Mg-Zn-Y alloys,* ***Materials Letters*** 330 (2023) 133315. IF: 2.7 doi: 10.1016/j.matlet.2022.133315.

Despite the high yield strength and moderate elongation of developed low-alloyed RSRC Mg alloys described in **[C1-C2]**, these alloys are characterized by pronounced yield point phenomenon during tensile loading at room temperature. Thus, in scope of the study **[C3]** the global and local microstructural changes leading to the appearance of the upper and lower yield points in low-alloyed RSRC Mg-Zn-Y alloy are clarified by advanced *in-situ* image techniques and post-processing data analysis. It was shown that the tensile deformation of the investigated alloy is solely dislocation-slip-mediated. The yield point phenomenon indicates a lack of mobile dislocations, as dislocations are pinned on SFs and solute atoms. Sudden release of un-pinned non-basal dislocations leads to stress drop. Moreover, constrained propagation of the deformation band was visualized using digital image correlation technique. It was proposed that microstructure features, incl. the solute segregated SFs dispersedly distributed in the randomly oriented grains and the submicrometer grain size, cause constrained propagation of the deformation band.

Further investigation of deformation mechanisms activated during tensile loading of low-alloyed RSRC Mg-Zn-Y alloys were conducted using *in-situ* synchrotron diffraction measurements and have been summarized in the bachelor thesis of my student Zsolt Beke [93]. Particularly, the significant contribution of non-basal slip systems to plastic deformation was revealed using diffraction data.

[C4] J. Gubicza, K. Máthis, P. Nagy, P. Jenei, Z. Hegedűs, A. Farkas, J. Veselý, S. Inoue, **D. Drozdenko**, Y. Kawamura, *In-situ study of the microstructure evolution during tension of a Mg-Y-Zn-Al alloy processed by rapidly solidified ribbon consolidation technique,* ***J Magnes Alloy*** 12 (2024), 2024-2040. IF: 17.6. doi.org/10.1016/j.jma.2024.05.008

To identify deformation mechanisms leading to exceptional mechanical performance of RSRC Mg-Zn-Y alloy with additional microalloying by Al, *in-situ* synchrotron X-ray diffraction line profile analysis was employed for a detailed study of the dislocation arrangement created during tensile loading of the Mg-0.9 Zn-2.05 Y-0.15 Al (at%) alloy. Moreover, to find the effect of the initial microstructure on the mechanical performance, additional samples were obtained by post processing heat treatment (for 2 h) of the as-consolidated alloys. Besides solute-rich SFs, CALs and CANaPs or complete LPSO plates, a secondary $Mg_{12}YZn$ phase with 18R structure in the primary α-Mg matrix were observed in the as-received and the heat-treated samples. The heat treatment at 300 °C had no significant effect on the initial microstructure, its development during tensile loading and consequently on the overall deformation behavior under loading. In contrast, annealing at 400 °C led to a significant increase in the fraction of recrystallized grains and a decrease of the dislocation density, resulting in only minor degradation of the mechanical strength.

Synchrotron data indicated that the dislocation density increased with increasing the plastic strain during tension achieving about 16–20 × 10^{14} m^{-2} at the plastic strains of 10–15% (in case of sample in the as-received state and after heat treatment at 300 °C). For the specimen heat treated at 400 °C, the dislocation density at the same plastic strain was lower (with the values between 2 and 4 ×10^{14} m^{-2}). The diffraction profile analysis indicated that most dislocations formed during tension were of non-basal $< a >$ and pyramidal $< c + a >$

types, which were congruent with the Schmid factor values derived independently from the orientation maps obtained by the EBSD technique.

Experimental data from deformation tests with *in-situ* synchrotron data were used for developing the strain hardening model based on the evolution of dislocation density. It was shown that dislocation-induced Taylor hardening was much less pronounced below the plastic strain of 3% than above this value. This was particularly explained by a model of the interaction between prismatic dislocations and CANaPs/LPSO plates. At low strains, the dislocation segments sliding close to the plates had no additional strain hardening beside the secondary phase hardening (given by dislocations sliding on another plane crossing this configuration). As the deformation progressed, more dislocations formed/penetrated between the CANaPs and LPSO plates, and these dislocation segments could not move close to the plates, effectively hindering the movement of the intersecting dislocations.

Moreover, *in-situ* synchrotron diffraction was also used for detailed investigation of the microstructure development during annealing of same low-alloyed Mg-Y-Zn-Al alloy and results were reported in [94]. Particularly, the temperature dependence of the ratio of the matrix lattice constants (*c/a*) and thermal expansion coefficients were clarified. The investigations were supported by SEM and TEM analysis.

[C5] A. Farkas, G. Farkas, P. Dobroň, J. Veselý, S. Inoue, Y. Kawamura, K. Máthis, **D. Drozdenko**, *Microstructure and thermal stability of MgZnYAl alloy containing cluster-arranged nanoplates (CANaPs), **Mater Charact*** 218 (2024) 114492. IF: 4.8. doi: 10.1016/j.matchar.2024.114492.

Complex microstructure containing individual SFs, CALs, CANaPs and complete LPSO phase dispersedly distributed in the grain interior because of RSRC processing revokes a question of its thermal stability. Therefore, several studies on the thermal stability of the microstructure were performed on the series of the RSRC Mg alloys. Particularly, **[C5]** addresses the thermal stability of the microstructures of Mg-0.9 Zn-2.05 Y-0.15 Al (at.%) alloy prepared by (i) conventional extrusion from cast ingot (hereafter, cast-extruded) and (ii) RSRC technique. The cast-extruded alloy was involved to uncover the effect of processing technique on developed microstructure and its further development with respect to post processing heat treatment. In order to study the thermal stability of the microstructure, isothermal annealing in a temperature range of 300–500 °C was applied. Development of microstructure was systematically analyzed using SEM and TEM. It is shown that in the initial state, although both microstructures consist of small (below 1 μm) DRX and large non-DRX grains, the distribution and morphology of the solute-enriched phases are strongly influenced by the processing route. Particularly, the RSRC alloy contains mainly fine homogeneously distributed CANaPs, while the cast-extruded alloy is characterized by larger solute-enriched phases (incl. CANaPs and LPSO fractions) together with individual CALs and/or thin CANaPs. Elongated lamellar LPSO fractions in the non-DRX grains of the cast-extruded alloy restrict the DRX process during post processing annealing, resulting in the growth of DRX grains formed during material production. In the RSRC alloy, fine and homogeneously distributed solute-enriched phases (present in the non-DRX grains) promoted the DRX process through the PSN mechanism. As a result, in the RSRC alloy, recrystallization occurs simultaneously in both the non-DRX and DRX grains. Thus, the differences in the size and distribution of solute-enriched phases affect the recrystallization dynamics during post-processing heat treatment. Nevertheless, independent on the processing route, both types of material exhibited exceptionally high thermal stability up to 300 °C, which significantly exceeds the values reported for conventional Mg alloys. After annealing at 400 °C, the CANaPs became thicker and more clustered, especially in the RSRC alloy. The results are in agreement with our previous work on the RSRC Mg-0.56Zn-1.5Y (at.%) alloy reported in [95]. This alloy showed stability up to 400 °C and above this temperature the recrystallization process started leading to randomization of the texture.

The study of thermal stability of low-alloyed RSRC Mg-Zn-Gd alloy, which is supposed to be better solution for reducing tensile-compression asymmetry as reported in **[C2]**, has been recently summarized in bachelor thesis of my student Adela Kolembusová [90]. Investigations showed that microstructure of RSRC Mg-Zn-Gd alloy is stable up to 350 °C.

Results of **[C5]** and [90] indicate that, as in case of Mg-Zn-Gd/Y alloys, the order of dispersion of SFs is independent of the annealing temperatures. However, the thickness of the stacking faults blocks increases with increasing temperature, i.e., the stacking faults become more agglomerated. Nevertheless, even after annealing at 500 °C, there is still a mixture of several polytypes of the LPSO phase rather than a single-ordered LPSO phase.

In general, compared to the commercial UFG Mg alloys, the thermal stability of the microstructure of the developed low-alloyed Mg-LPSO alloys is exceptionally high, what indicates their application potential at high temperatures.

Summary remarks on the publications [C1-C5]

Publications **[C1-C5]** are dedicated to knowledge-based research and development of low-alloyed Mg alloys prepared by RSRC technique. The application of an advanced processing technique (consolidation of rapidly solidified ribbons) on low-alloyed alloys was intended to maintain enhanced properties of Mg alloys with layered structure, as motivated by performance of Mg-LPSO alloys (chapter 2.2.). Processing technique together with reduced amount of alloying elements leads to the formation of UFG microstructure with the individual solute-rich SFs or their arrangement in CALs/CANaPs or complete LPSO phase formed in basal planes of *hcp* α-Mg lattice, and thus organizing a layered microstructure on the microscale. Moreover, we reported for the first time that the bimodal microstructure, which has a variety not only in grain size but also in internal strain distribution, played a key role in improving mechanical properties. The developed alloys demonstrated tensile yield strength and elongation comparable to leading Mg alloys prepared by powder metallurgy. Thus, the RSRC processing offers a cost-effective alternative with promising mechanical performance, paving the way for further development of low-alloyed Mg alloys for biomedical or engineering application **[C1]**. Further development of the RSRC Mg-Zn-Y alloys indicated that replacing Y by Gd or Nd leads to a reduction in asymmetry with minimal loss in the yield strength values **[C2]**. It should be noted that these alloys are characterized by pronounced yield point phenomenon during tensile loading at room temperature, which is rather unusual in common Mg alloys. Using advanced *in-situ* image techniques and post-processing data analysis it was demonstrated that tensile deformation is solely dislocation-slip-mediated and sudden release of un-pinned non-basal dislocations leads to stress drop **[C3]**. In addition, the *in-situ* synchrotron X-ray diffraction line profile analysis was used for analysis of plastic deformation during tensile loading of the Mg-Zn-Y alloy with microalloying by Al **[C4]**. The experimental data provided information on the evolution of dislocation density and their arrangement (defining types), which have been used for developing the strain hardening model. Besides improved mechanical properties, the developed RSRC Mg-Zn-Y-based alloys with solute segregated SFs, CALs and CANaPs (dispersedly distributed in the grains interior) are characterized by enhanced corrosion and thermal properties, which indicate their high application potential in various environments. These alloys are in particular characterized by exceptionally high thermal stability up to 400 °C **[C5]**, contrasting with conventional UFG Mg alloys.

Statement of the author’s contribution to the publications [C1-C5]

I proposed the concept of all experiments and led the research reported in **[C1-C5]**. I directly participated in production of alloys for studies in **[C1-C3]** during my post-doc fellowship and following visits within the framework of the cooperation with the research team of MRC, Kumamoto University, Japan. The results in **[C1]** became a motivation and basis for proposal of the junior project funded by Grant Agency of Czech Republic, which was accomplished in 2020-2024 under my supervision. The papers **[C2-C3]** are reporting on the project results.

In the course of research, I directly participated **[C1, C3]** or guided students through investigations using SEM and TEM **[C1-C5]**. I performed all deformation tests presented in **[C1-C3]**, including acquisition data for post processing digital image correlation **[C3]**. My expertise in microstructure investigation and deformation mechanisms of conventional Mg alloys and recently developed Mg-LPSO alloys allowed me to contribute significantly to physical interpretation of the obtained results for low-alloyed Mg alloys with complex layered microstructure **[C1-C5]**. While the papers **[C1-C3]** were prepared by me with a contribution of my colleagues, I also actively participated in the manuscripts preparation of **[C4, C5]**.

3. Conclusions and future perspectives

The present thesis summarizes key findings published by the applicant in selected publications focused on investigation of fundamental deformation mechanisms in conventional Mg alloys, as well as their manifestation in novel Mg alloys with layered microstructures at both the macro- and microscale. The application of advanced methods of (*in-situ* and *post-mortem)* microstructure analysis in combination with the *in-situ* AE technique and diffraction methods has raised the investigation to a new level, allowing us to reveal the minute details of the microstructure evolution and activity of deformation processes. The results on the activity of deformation mechanisms obtained by mechanical loading of specifically oriented Mg single crystals have served as benchmark data for the investigation of more complex Mg alloy systems. In particular, this knowledge has been used to elucidate the physical background of the high strength of a new generation of Mg alloys containing LPSO phases. We demonstrated the role of the mixture of fine and coarse α-Mg grains and elongated LPSO phases in plasticity. Obtained comprehensive information about the beneficial role of the multimodal layered microstructure and the reinforcing effect of the LPSO phase helped us in the development of high-strength Mg alloys with significantly reduced alloying content. To preserve the desirable mechanical properties in low-alloyed Mg-LPSO alloy, the application of the RSRC technique was found to be effective. The processing technique and the reduced alloying results in the formation of individual solute-rich SFs or their arrangement in cluster arranged layers and nanoplates (CALs/CANaPs), thus forming a layered nanostructure on the microscale. As we have shown, the layered microstructure tuned by appropriate fabrication parameters and post-treatment can result in significant enhancement of mechanical performance, which opens new avenues for the development of application-oriented, high-strength Mg alloys.

Obtained results motivate further investigation, supported by current development of advanced experimental and numerical tools. Particularly, the effect of deformation temperature – across a wide range from low to high – is essential for evaluating the practical potential of RSRC-processed Mg alloys. At the same time, the inherent biocompatibility of these alloys makes them promising candidates for biomedical implant applications, necessitating further research into revealing their static and dynamic performance in biological environments. Beyond alloy development, questions regarding fundamental deformation mechanisms — such as the interaction of twin boundaries with dislocations, precipitates, and strain fields — remain unresolved and continue to fuel scientific curiosity.

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[A4] F. Siska, **D. Drozdenko**, K. Máthis, J. Cizek, T. Guo, M. Barnett, *Three-dimensional crystal plasticity and HR-EBSD analysis of the local stress-strain fields induced during twin propagation and thickening in magnesium alloys,* ***J Magnes Alloy*** 11 (2) (2023) 657-670. IF: 17.6. doi: 10.1016/j.jma.2022.11.006.

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Attachment - Reprints of the selected publications