

Editorial

# Microstructure–Mechanical Properties and Application of Magnesium Alloys

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## 1. Introduction and Scope

Transport is a major contributor to CO<sub>2</sub> emissions and is considered the most urgent global climate problem. Considering the fact that light-weighting is a crucial factor for efficiency improvements for conventional combustion engines as well as electric vehicles, the increased use of low-density metals, such as aluminum and magnesium, is an effective tool to tackle CO<sub>2</sub> emissions and produce an efficient low—or even—zero-carbon vehicle technology. As a result, the global trend towards light-weighting has triggered major international efforts to develop innovative and cost-effective magnesium alloys and processing for lightweight structural components. Extensive use of magnesium alloys in transportation is nevertheless hindered because we still lack a full understanding of their mechanical and electrochemical behavior that results from the complex interplay between the microstructure and alloy chemistry. Compared to steel or even aluminum, magnesium alloy research is relatively young, being mostly published during the past 20 years, where significant advancements in high-resolution characterization and sophisticated atomistic modeling techniques have emerged. This has introduced many new, exciting possibilities to shed light on still pending questions and stimulate new research areas.

The aim of this Special Issue is to cover a broad scope of contributions that highlight current accomplishments, and to provide the readers with some perspectives on the direction of research on magnesium alloys in the near future with respect to global challenges. This Special Issue contains papers presenting the state of the art and the research trends in the relationship among the microstructure, properties and the industrial application of magnesium alloys. The contributions in the Special Issue cover a wide range of research topics, from alloy fabrication to wrought processing, encompassing advanced material characterization at different length scales, microstructure manipulation using alloying, thermo-mechanical treatments, as well as modern material modeling to establish the best composition/processing/microstructure combinations for targeted applications. The contributions included in the Special Issue will provide guidance towards synergetic development in new alloys and in the processing of the newly developed alloys.

## 2. Contributions

The great success of the Special Issue is evidenced by the 12 high-quality papers covering a wide range of topics, from casting to property optimization to service conditions.

The first article in this Special Issue [1] investigates the interfacial reactions between Mg-40Al and Mg-30Y master alloys at temperatures of 350–400 °C using the diffusion couple method. With a lack of systematic studies of the diffusion kinetics in Mg-Al-Y systems, this study addresses a major gap in determining the interdiffusion interactions of alloying elements and the formation of intermetallic phases at the interface of the diffusion couple. From the interface microstructures, the authors reported that noticeable reaction



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layers with sub-layers of different contrast were formed at the interface of the diffusion couple. The thickness of these layers was found to increase with the annealing temperature and duration (from 24 h to 72 h). Interestingly, Y in the Mg-30Y matrix did not diffuse into the opposite Mg-40Al matrix, which was attributed to its relatively large atomic radius. The Al<sub>2</sub>Y intermetallic phase, which is considered a promising grain refiner, was formed only after Al diffused into the Mg-30Y. Thermodynamic calculations showed that this phase is the most stable among other phase compositions in the Al-Y system. Diffusion path analysis along with XRD measurements also confirmed the formation of the Al<sub>2</sub>Y phase by the interaction between Al and Y. By investigating the growth kinetics of the reaction layers, the authors estimated the diffusion activation energy to be around 90 kJ/mol. They also provided useful diffusion parameters of Al and Y, which were found to diffuse preferably in the layer located in the Mg-30Y matrix. The interdiffusion coefficients of Al at the different temperatures were higher than those of Y. Analogously, the activation energy for the diffusion of Y was higher than that of Al. The results obtained in this study have contributed towards a better understanding of the precipitation behavior in Mg-Al-Y alloys, which are known for their fine microstructure and improved strength properties.

The second contribution, by Z. Yan et al. [2], features a novel severe plastic deformation technique (CEE-AEC) that was used to process an as-cast AZ31B magnesium alloy into extruded plates with a fine grain size and enhanced ductility. This was conducted at 350 °C, with an extrusion rate of 1 mm/s. In the proposed method, cyclic expansion extrusion utilizes an asymmetrical extrusion cavity that introduces shear deformation and thereby modifies the basal texture and increases the Schmid factor for basal slip. The authors used FEM to design the die and billet geometries based on the calculated distribution of effective strain in different deformation zones and the required forces as a function of processing time. Due to a combination of continuous and discontinuous dynamic recrystallization, the resultant microstructure after three passes was fully recrystallized, with a fine grain size of ~10 µm (refinement degree of ~96% relative to the initial as-cast state). The corresponding texture was sharp but showed off-basal peak intensities at 45° from the extrusion direction. With this, the tensile properties of the extruded samples revealed an attractive combination of yield strength (~115 MPa), ultimate tensile strength (~209 MPa) and maximum elongation (~30%). The obtained results clearly demonstrate the feasibility of using this intelligent method to obtain excellent mechanical properties from processed as-cast magnesium alloys readily after three deformation cycles. An additional major advantage is the capability of fabricating large workparts, which enables this severe plastic deformation method to be used on an industrial scale.

The production of highly competitive, thin wires of Mg-Al-Zn (AZ) alloys by means of direct extrusion is discussed in the third contribution, by Nienaber et al. [3]. Wires of magnesium alloys have high specific strength; hence, they are attractive for use as filler materials in joining applications or as biodegradable suture materials in the medical field. Commonly, very thin wires (thicknesses of ~100 µm) are produced through drawing processes of extruded bars employing several passes and intermediate annealing. Alternative production from cast billets via direct extrusion is extremely challenging because of the overwhelmingly high degree of deformation involved. Another challenging aspect is the urgent demand for a high surface quality that has a strong impact on the corrosion behavior of the produced wires. In this study, the authors demonstrated that it is possible to produce 1 mm thin wires of different AZ alloys with various Al content (i.e., AZ31, AZ80 and AZ91) by means of direct extrusion at 325 °C and 0.1 mm/s extrusion speed. The limitation towards reducing the thickness below 1 mm was due to the capacity limit of the extrusion press of 2.5 MN peak force. All Mg alloy wires revealed a homogenous, fine-grained microstructure with an average grain size below 10 µm. The tensile yield strength was between ~180 and ~195 MPa and the ultimate tensile strength was between ~270 and ~300 MPa. With increasing Al content, the maximum texture intensity showed a decrease, accompanied by a more random orientation distribution between the  $\langle 10\bar{1}0 \rangle$  and  $\langle 11\bar{2}0 \rangle$  poles. This had a positive impact on the ductility, which ranged from ~16%

(fracture strain) for AZ31 to ~21% for AZ91. The surface quality of the produced wires was tested before and after a wrapping test. AZ31 showed the smoothest surface among the other alloys prior to the wrapping test. After the test, the roughness of the wires increased considerably, which depended on the wrapping diameter. Interestingly, the AZ91 alloy with the highest Al content revealed the smallest increase in surface roughness, which highlights the success of the reported process in producing high-performance wires with an excellent profile of mechanical and surface properties.

Damage mechanisms in conventional magnesium alloys exhibiting mechanical twinning are the topic of the fourth paper in this Special Issue [4]. In general, twinning is known to induce strain incompatibility in the microstructure, which needs to be accommodated by slip or interaction twins in adjacent grains; otherwise, it would lead to the nucleation of cracks. Investigations of twin accommodation effects at grain and twin boundaries are therefore highly important for understanding damage initiation in magnesium and other HCP metals. In an effort to achieve this, the authors performed thorough microstructural and fractographical analyses on the interactions between twins and other deformation modes and microstructural defects. They employed electron microscopy combined with in-situ electron backscatter diffraction characterization on plane-strain extruded AM30 magnesium alloy with a double fiber texture. This offered the possibility of tracking deformation through various neighboring grain orientations with favorable and unfavorable activation of  $\{10\bar{1}2\}$  and  $\{10\bar{1}1\}$  twinning. The results revealed various types of twin interactions with slip dislocations, grain boundaries and other twins, which led to local cracking. Nucleated twins of a very low macroscopic Schmid factor were less tolerant to damage initiation through these interactions. An interesting observation was made in the case of  $\{10\bar{1}2\}$  twins in favorably oriented grains, where twin variants that were able to nucleate easily at grain boundaries showed favorable growth compared to other variants. It was suggested that this could have been related to the enhanced nucleation of glissile (mobile) disconnections at the intersection between grain and twin boundaries rather than between two twin boundaries. Another interesting finding was that  $\{10\bar{1}1\}$  twins were found to nucleate easier under  $\langle c \rangle$ -axis contraction than what is reported in the literature under  $\langle c \rangle$ -axis compression. Although this aspect has not yet been investigated in detail, the authors propose that it is related to the complex stress state in the case of  $\langle c \rangle$ -axis contraction that could facilitate the atomic shuffle associated with  $\{10\bar{1}1\}$  twinning. With these important findings, the study provides valuable insights towards a better understanding of twin–microstructure interactions and their effect on twin formation and damage initiation in magnesium alloys. It also offers beneficial input for advancing our present crystal plasticity simulation models.

The paper written by Bian, M., Huang, X. and Chino, Y. [5] describes the development of a new type of precipitation-hardenable magnesium sheet alloy. The authors chose the magnesium silver system with the addition of calcium and explored the feasibility of developing precipitation-hardenable Mg sheet alloys. Based on the Pandat software, they calculated the Mg–Ag phase diagram containing Ca content fixed at 0.1 wt%. They prepared three alloys with different Ag contents (1.6 wt%, 6 wt% and 12 wt%), extruded them to sheets and rolled them to a final gauge. They investigated the age-hardening response at 170 °C by measuring the Vickers hardness and tensile properties and analyzed the microstructure using various electron microscopy techniques.

In a T4-treated condition, the TYS of Mg–1.5Ag–0.1Ca alloy sheet is only 85 MPa, 57 MPa and 47 MPa along the RD, 45° and TD, respectively. With an Ag content of 12 wt%, the tensile yield strength (TYS) is increased in a T4-treated condition to 193 MPa, 130 MPa and 117 MPa along the RD, 45° and TD direction. Artificial aging at 170 °C for 336 h (T6) further increases the TYS of Mg–12Ag–0.1Ca alloy sheet to 236 MPa, 163 MPa and 143 MPa along the RD, 45° and TD direction. The microstructure characterization reveals that  $\text{AgMg}_4$  are responsible for the strength improvement, and, in a high-resolution HAADF-STEM image analyzing the FFT patterns, the orientation relationship between the  $\alpha$ -Mg matrix and  $\text{AgMg}_4$  is confirmed by the authors to be  $(0001)_\alpha \parallel (0001)\text{AgMg}_4$ ,  $[-2110]_\alpha$

|| [10-10]AgMg<sub>4</sub>. These findings show that it is possible to improve the age-hardening response and accelerate the aging kinetics of Mg-Ag-Ca alloys by microstructural design.

The contribution, written by Ostapovets, A., Kushnir, K., Máthis, K. and Šiška, F. [6] is focused on the numerical analysis of the interaction between the growing tensile twin and obstacles in magnesium. The study is based on a multiscale approach as the twin growth is simulated using an atomistic model, while the overall stress state is evaluated using a finite element method. As is known, abundant twinning can lead to the formation of structure inhomogeneity during the plastic deformation of magnesium and its alloys. Such inhomogeneity can be a serious restriction for engineering applications. Suppression of the twinning by twin interactions with obstacles can also be considered as a means of decreasing the plastic anisotropy and improving the mechanical properties of the material. The obstacle for twin boundary migration was inserted into the simulation block. Two types of obstacles were considered. One type was a void, and the other type of obstacle was obtained by freezing atoms inside the selected volume. The size of the obstacle was the same as the size of the void. The external tensile stress was applied in the X-direction on the vertical sides, which represents tension in the crystallographic c-direction. The results revealed an increase in critical resolved shear stress, which was higher for the passage of the twin boundary through a row of voids than for the interaction with non-shearable obstacles. Two basal stacking faults were nucleated during the detachment of the boundary from the obstacle. These stacking faults were trailed by the migrating boundary and grew together with the twin. It could be seen that this stress is dependent on the type of obstacle as well as on the initial density of disconnections. It is interesting that voids serve as a stronger barrier for twin boundary migration than non-shearable obstacles.

In the next fundamental study, the authors Mouhib, F., Sheng, F., Mandia, R., Pei, R., Korte-Kerzel, S. and Al-Samman, T. [7] aimed to further the understanding of the texture development in a binary Mg-1 wt%Er and in a ternary Mg-1wt %Er-1wt%Zn alloy during recrystallization and grain growth under the influence of solutes and second-phase precipitates. The rolling texture of the binary alloy showed a typical basal component with a pole spread towards the rolling direction (RD) and moderate intensity. In contrast, the ternary alloy exhibited a weaker and much softer rolling texture characterized by two off-basal components at  $\pm 20^\circ$  RD that bear a significant pole spread in the transverse direction  $\pm 40^\circ$  TD. The discussion addresses the microstructure and texture development in both binary and ternary versions of the alloy during recrystallization and grain growth under the influence of solutes and second-phase precipitates and the different recrystallization kinetics. Therefore, both alloys underwent characterization of the microstructure and their evolution, their texture development, as well as EBSD-assisted slip trace analysis in order to understand the activation of deformation modes during tension to 5% strain. The authors report that the addition of Zn alters the substitutional solute chemistry of the Mg-1%Er alloy, which would obviously lead to the complex interaction of solute species within the matrix. From an energetic perspective, Zn and Er can cluster in the lattice to relieve the misfit strains arising from the solute size mismatch. They would also cosegregate to defects in the microstructure and are therefore likely to have a stronger interaction with grain boundaries and dislocations, leading to strikingly different slip system activation during deformation and boundary migration characteristics during annealing. The authors showed a remarkable enhancement in yield strength, strain hardening capability and failure ductility due to precipitates, solute strengthening effects and a favorable soft texture, and they also identified the main mechanism for the  $\pm 20^\circ$  RD  $\rightarrow$   $\pm 40^\circ$  TD of the deformed Mg-1%Er-1%Zn alloy.

The modification of the microstructure to adjust the mechanical properties and the reduction of the asymmetric yield behavior and anisotropy of the extruded magnesium alloys Mg<sub>2</sub>Nd and Mg<sub>2</sub>Yb is reported in the article of Schmidt, J., Beyerlein, I., Knezevic, M. and Reimers, W. [8]. For their study, they extruded the binary magnesium alloys Mg<sub>2</sub> wt%Nd and Mg<sub>2</sub> wt%Yb and investigated their deformation behavior at room temperature. To modify the microstructure and thus the mechanical properties, they used variations in

extrusion parameters. In addition due to the low solubility of Nd and ytterbium (Yb) in Mg, subsequent heat treatments were used to further increase the strength. The extruded bars were investigated by electron microscopy (SEM, TEM), laboratory X-ray texture measurements and mechanical testing (compression, tension) to determine the mechanical properties, the microstructure and their changes during deformation. To gain further insight into the different deformation behavior, the authors used a combination of in situ energy-dispersive X-ray synchrotron diffraction and applied simulations with the elastoplastic self-consistent (EPSC) model. In the Mg<sub>2</sub>Nd alloy, subsequent heat treatments lead to the formation of fine precipitates, generating a significant hardening effect. In the case of the Mg<sub>2</sub>Yb alloy, heat treatments for precipitation hardening were less effective. The authors report that, by reducing the extrusion temperature, a decrease in the grain size of the Mg<sub>2</sub>Yb and an advantageous texture could be achieved. This was accompanied by a significant increase in YS. Since critical resolved shear stresses for tension twinning (CRSS<sub>ttw</sub>) in particular are very sensitive to grain size, the compressive yield strength (CYS) increases more than the tensile yield strength (TYS). By adjusting the process parameters, including heat treatments, the grain size can be reduced and the texture improved as well, so that, despite differences in plastic deformation, the yield strengths in compression are almost equal to the yield strengths in tension. This resulted in high strengths and low strength differential effects (SDE). As a result, the Mg<sub>2</sub>Nd series, which had high yield strengths and an SDE close to 0, could be extruded. By adjusting the extrusion parameters, the grain size of the Mg<sub>2</sub>Nd alloy can be gradually reduced, thereby significantly increasing the strength. The Mg<sub>2</sub>Yb series tend to have larger grain sizes and less pronounced rare earth textures. This leads to intensive tension twinning (TTW-ing) under compressive stress, resulting in high SDEs. Subsequent heat treatments can increase the YS. Since the effect on the slip systems is stronger than on the formation of TTWs, this leads to further increased SDEs. However, by reducing the extrusion temperature, the grain size of the Mg<sub>2</sub>Yb alloy is also reduced and an advantageous texture can be achieved. This is accompanied by a significant increase in YS. Since CRSS<sub>ttw</sub>, in particular, is very sensitive to grain size, the CYS increases more than the TYS. Therefore, it is also possible to obtain an SDE close to 0 for the Mg<sub>2</sub>Yb alloy.

The active deformation mechanisms during the hot rolling of magnesium single crystals, with different initial orientations, are reported in the contribution of Estrada-Martinez, J., Hernandez-Silva, D. and Al-Samman, T. [9]. In this study, the texture evolution during the rolling of the single crystals with two different initial orientations is evaluated, in terms of the prismatic axis aligned in the compression direction (sheet normal direction, ND), while the extension in the c-axis is allowed by the c-axis laid in the rolling direction (RD) in both cases. The systematic investigations using XRD and EBSD revealed the high activities of extension twinning and the concomitant reorientation of original orientations. The orientations formed by the extension twinning are determined by the active twin variants. When the  $[10\bar{1}0]$  direction is parallel to the ND, the reorientation by extension twinning results in the c-axis being parallel to the ND. In contrast, the active twin variants by compression in the  $[11\bar{2}0]$  axis lead the twin orientations with the c-axis to be aligned at an angle of 30° from the ND. Further deformation of the fully reoriented matrix induces the contraction twins, in which the basal slip occurs relatively easily. The recrystallization triggered at the contraction twins brings about continuously recrystallized grains with non-basal orientations. By using the single crystal and the deformation in a specific orientation, the active deformation mechanism and its contribution to the final texture could be successfully described with respect to the initial orientation.

The tenth contribution, by Jo, S., Letzig, D. and Yi, S. [10], deals with the microstructure and texture control of a non-flammable Mg alloy containing Y and Ca simultaneously. The results regarding the interrelationship between the Al content, texture weakening and mechanical properties provide an efficient guideline towards the improvement of sheet formability, which is important to widen the industrial application of magnesium alloys. By decreasing the Al content, the amount of secondary phases decreases, while the amount

of solute atoms, especially Ca, dissolved in the matrix concomitantly increases. A higher amount of solute atoms enhances the activity of non-basal deformation modes and retards the recrystallization such that the texture weakening and the accompanying formation of the non-basal type components are observed in the alloy sheets with smaller Al amounts. The thermodynamic calculation complementarily supports the experimental observations regarding the formation of secondary phases and the solute amount. This study contributes to establishing an alloy design strategy facilitating the development of high-performance magnesium alloy sheets.

The superplastic behavior of Al-free ZK60 alloy having a fine and homogeneous grain structure, which was processed by indirect extrusion, was investigated in the contribution of Palacios-Trujillo, C., Victoria-Hernandez, J., Hernandez-Silva, D., Letzig, D. and Garcia-Bernal, M. [11]. From the tensile tests at various testing conditions, the superplastic behavior was found at 250 °C and  $10^{-4}$ /s, resulting in an elongation to failure of 464%. The tensile sample deformed under a superplastic regime showed a dynamic recrystallized grain structure, while significant texture weakening was observed in the deformed sample. These results imply that the superplastic behavior of the ZK60 alloy at intermediate temperature is brought about by the combined effect of dynamic recrystallization and the grain boundary sliding, while the increase in the strain rate or temperature reduction causes the change in the deformation mechanism to dislocation glide. The activation energy, based on the Zener–Hollomon parameter, of 446 kJ/mol through the range of strain rates and temperature indicates that the grain boundary sliding is responsible for the superplasticity of the examined ZK60 alloy. The most important findings of this study indicate that the superplastic behavior can be obtained at an intermediate temperature even in commercial magnesium alloys processed without employing severe plastic deformation.

The contribution of R. Yamada, S. Yoshihara and Y. Ito examined the effect of equal channel angular pressing (ECAP) on the fatigue properties of AZ31B alloy [12]. The experimental results comprising the tensile properties, fatigue behavior and residual stress indicate that the strengthening brought about by the grain refinement after one ECAP pass effectively improves the fatigue property, while the sample after eight ECAP passes shows a deteriorated fatigue property despite its improved ductility. Moreover, a lower compressive residual stress in the ECAP samples after the fatigue test was measured in comparison to that in the annealed sample. The authors inferred from the different residual stresses after the fatigue tests that the ECAP-treated samples should have a higher tensile residual stress before the fatigue test, which is harmful for the fatigue properties. The experimental results of this study clearly show that strength improvement, especially yield strength, is a more effective means of enhancing the fatigue properties than a ductility increase.

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### 3. Conclusions and Outlook

We hope that this Special Issue will serve as a useful reference and will help to incite new ideas in magnesium research, not only for those researchers, theoreticians and experimentalists who have been working in this field but also for those who are new to the field.

**Conflicts of Interest:** The authors declare no conflict of interest.

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