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HABILITATION THESIS

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Development of ultra-fine-grained magnesium materials

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Preface

This habilitation thesis encompasses nine scientific papers that have been published in respected journals over the past eleven years. Each of these selected papers has significantly contributed to advancing scientific knowledge in the field of ultra-fine-grained (UFG) magnesium materials. The overarching goal of this ongoing research is to develop magnesium materials that are resistant to ignition and possess high ignition temperatures.

The thesis is structured into two thematic sections. The first chapter provides a comprehensive summary of the results achieved with ultra-fine-grained commercial magnesium alloys that include aluminium and zinc as alloying elements. This chapter primarily focuses on the mechanisms responsible for grain refinement in these well-known materials. The second chapter delves into the subsequent development of magnesium-based alloys containing rare earth elements and calcium. These novel alloys are characterized by high ignition temperatures, making them particularly promising for applications in the aviation industry.

To provide a thorough and cohesive understanding, the thesis is concluded with the original full-length papers. These papers are meticulously discussed within the text, highlighting their contributions to the field and their implications for future research and commercial applications.

1. Introduction and theoretical background

Magnesium alloys are increasingly being considered for use in the aerospace, automotive and other transportation industries due to their exceptionally low density, making them highly attractive for structural components. The lightweight nature of magnesium alloys offers the potential to replace heavier materials such as steel and even aluminium alloys, which are also heavier than magnesium, in various applications, thereby contributing to overall weight reduction, improved fuel efficiency and sustainability [1].

However, despite their advantages, the application of magnesium alloys in more demanding applications is currently limited by several drawbacks such as limited strength and low ductility. These challenges can be overcome with innovative solutions and advancements in research and development. However, primary concern about magnesium materials is their poor corrosion resistance, which makes them susceptible to degradation in harsh environments. However, with the right design of alloys and corrosion prevention techniques (e.g. organic/polymer coatings, multiple surface coatings, laser or other methods [2]), magnesium materials can be effectively shielded from moisture, salts, and other corrosive elements. This opens up exciting possibilities for their use in the automotive and aerospace sectors, where lightweight and durable materials are in high demand.

Inherent flammability of magnesium gives rise to stringent safety measures, particularly in industries such as aerospace and automotive, where the advantages of reduced weight must be carefully balanced against the risks of fire hazards. Until 2015, the use of magnesium alloys as a structural element in aircraft interiors was prohibited by the SAE Aerospace Standard (AS) 8049 C. However, this regulation has been revised, and the current version permits the use of magnesium alloys in aircraft seat construction, provided they meet the flammability performance requirements specified in DOT/FAA/AR-00/12 [3]. Unlike many other metals, pure magnesium has a low ignition temperature of approximately 630°C [4] making its unsuitable for applications in which accident scenarios must be considered. However, the addition of alloying elements can affect the ignition and combustion behaviour of magnesium and improve its resilience to ignition.

While magnesium alloys have limited strength and ductility compared to other metals, this characteristic can be seen as an opportunity for innovation. By understanding and harnessing the unique properties of the hexagonal close-packed (HCP) crystal structure, we can develop novel manufacturing techniques and alloy compositions that enhance the ductility of magnesium materials. This would enable them to undergo significant plastic deformation before rupture, making them suitable for a wider range of structural applications.

Furthermore, the anisotropy of critical resolved shear stress (CRSS) in different slip systems in magnesium alloys can be seen as a challenge that can be overcome through microstructure and texture control and tailoring. Namely, by careful control of the processing conditions during forming, it is possible to minimize the development of strong deformation textures and reduce stress anisotropy.

In conclusion, while there are currently limitations to the broader use of magnesium alloys, these challenges can be addressed through continued research and development efforts. With advancements in corrosion resistance, flammability control, and the enhancement of ductility, magnesium materials have the potential to revolutionize the automotive, aerospace or other industries. This thesis aims on contributing to unlock full potential of magnesium alloys and to expand their applicability in advanced engineering applications, namely by development of ultra-fine-grain magnesium alloys with enhanced strength and by advanced alloying to improve their ignition resistance.

1.1. Ultra-fine-grained structure in magnesium materials

The mechanical properties and other essential characteristics that determine the application of magnesium alloys can be significantly enhanced by refining the grain size to sub-microcrystalline or even nano-crystalline levels. Achieving such ultra-fine-grained (UFG) structures can lead to notable improvements in strength, ductility, and overall performance of the material, making magnesium alloys more viable for a broader range of applications.

Over the past four decades, researchers have developed and proposed a variety of innovative techniques to produce these ultra-fine-grained structures in materials. These techniques aim to drastically reduce the grain size, thereby enhance mechanical properties of material through grain boundary strengthening. Grain refinement can be achieved by so-called top-down approach, during which the originally coarse-grained structure is radically refined by plastic deformation. The most successful techniques employing such approach are referred to as severe plastic deformation (SPD) techniques. During SPD, large plastic strain is repetitively imposed, causing introduction of high densities of lattice defects in the material, namely dislocations, leading consequently to exceptional grain refinement and the formation of ultra-fine-grained structures [5].

Several specific SPD techniques have been developed and refined over the years. One widely used method is equal-channel angular pressing (ECAP), where a material is pressed through a die containing two channels of equal cross-section that intersect at an angle. During this process severe shear deformation is imposed to the material, resulting in continuous fragmentation of the structure and substantial reduction of the grain structure without significantly altering the external shape of the sample [6].

Another prominent SPD technique is high-pressure torsion (HPT). In HPT, a disc-shaped sample is subjected to a high pressure while being simultaneously twisted. The combination of high pressure and torsional strain leads to extreme grain refinement, producing nano-crystalline structures with superior mechanical properties [7], [8].

Additional SPD techniques include accumulative roll bonding (ARB) which was invented by a Japanese group of researchers in 1990s [9], where multiple layers of material are stacked, rolled, and bonded together. Repetitive corrugation and straightening (RCS) is another remarkable SPD method, where the material is repeatedly bent and straightened to induce severe plastic deformation. This technique was developed in the Los Alamos National Laboratory [10].

Each of these techniques has been shown to produce significantly refined microstructure resulting in improved mechanical properties of magnesium alloys. The ultra-fine-grained magnesium base materials exhibit enhanced strength and ductility due to the increased grain boundary area, which impedes dislocation movement and thus strengthens the material. Moreover, the refined grain size can also contribute to improved corrosion resistance and better performance at elevated temperatures, addressing some of the inherent weaknesses of magnesium alloys.

1.1.1. Equal-channel angular pressing (ECAP)

Equal-channel angular pressing (ECAP) is one of the most developed and commercially utilized severe plastic deformation (SPD) technique. ECAP was invented in former Soviet Union by Segal et al. in the 1980s [6]. Since its inception, ECAP has become a well-known and widely used method for grain refinement globally.

The primary advantage of the ECAP technique is that the cross-sectional dimensions of the specimens remain unchanged before and after pressing. This allows repetitive pressing and the accumulation of strain within the specimen. However, multiple passes through ECAP can lead to cracking or other material damage. Therefore, all processing parameters, such as the number of passes, deformation temperature, speed of pressing, applied force, etc. must be optimized to achieve the best results.

One of the main disadvantages of ECAP is the limitation on the dimensions of the specimens. Typically, the length of the billets ranges between 5 and 15 cm. This drawback has been subsequently overcome by introducing the technique of continuous ECAP referred to also as ECAP conform (ECAP-C), which is able to produce wires and rods of almost unlimited length [11], [12].

The ECAP die consists of two channels with the same cross-section. It is characterized by two angles: Φ, which is the angle formed by the intersection of the two channels, and Ψ, which indicates the outer arc of curvature at this intersection. The conventional and most commonly used ECAP die has an angle Φ of 90°, as shown schematically in Fig. 1.

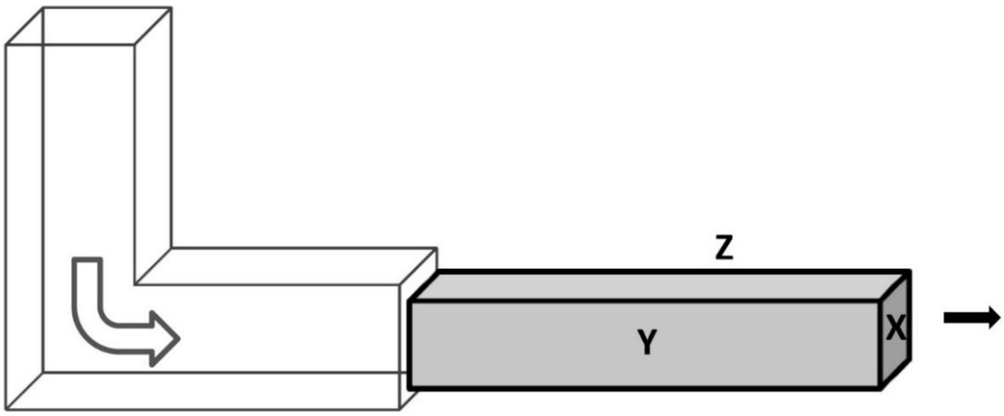

Figure 1: Schematic illustration of a conventional ECAP facility, *X* denotes the transverse plane, *Y* flow plane and *Z* the longitudinal plane [13].

The specimen pressed through the ECAP die is deformed by a simple shear at the intersection of both channels and the imposed strain after *N* passes could be expressed as [14]:

$$\epsilon_N = \frac{N}{\sqrt{3}}\left[2cotg\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right) + \Psi cosec\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right]. \quad (1)$$

The equivalent strain imposed in the material during ECAP depends on both angles Φ and Ψ. However, the angle Ψ plays only a minor role in determining the strain imposed on the sample in comparison with the angle Φ [15]. The equivalent strain ε imposed by a single pass in the commonly used conventional ECAP die with Φ = 90° is close to 1 (100 %). Thus, the equivalent strain after e.g. 4 and 8 ECAP passes is approximately equal to 400 and 800 %, respectively. More details about this SPD method are summarized for example in my dissertation thesis [13] or in well-known review papers [14] and [15].

1.1.2. High pressure torsion

High pressure torsion (HPT) is another well-known severe plastic deformation technique. In this method, the mechanical properties of the material are improved by a high pressure and concurrent torsional straining [16]. The experimental setup of HPT is schematically illustrated in Fig. 2.

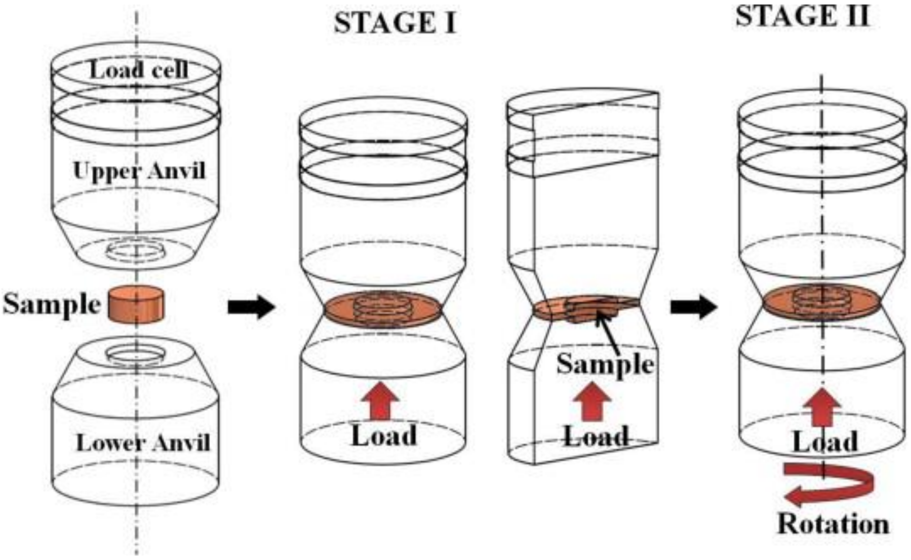

Figure 2: Schematic of the HPT device showing the set-up, compression stage (stage I), and compression-torsion stage (stage II) [17].

The typical size of the disc-shaped sample varies from 10 to 20 mm in diameter and about 1 mm in thickness. A disc sample is placed between two anvils where it is subjected to a compressive pressure of several GPa. Simultaneously, the one of the anvils rotates and the torsional strain is imposed to the sample.

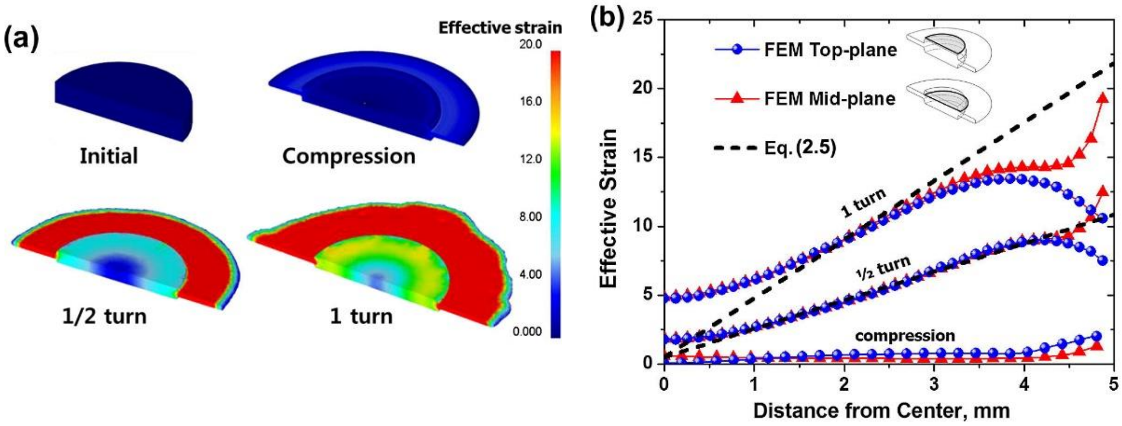

Figure 3: Finite element method (FEM) simulation of effective strain imposed by HPT: a) Deformed geometries at the initial stage, after compression, after 1/2 turn and after 1 turn with effective strain distributions; b) Path plots of the effective strain on the top plane and mid-plane

after compression, 1/2 turn and 1 turn, comparing the theoretical von Mises strain (Eq. 2) and the results from FEM simulations [18].

The effective strain (obtained from the FEM results) imposed by HPT increase at the centre after half a turn, while the torsion (shear) strain given by Eq. (2) is zero. The strain obtained by the FEM simulations at the centre, which is higher than the theoretical value (Eq. (2)), can be explained by the compressive strain in HPT and the finite mesh size in the FEM [17], [18]. The FEM simulation and torsion theory results are in a good agreement in the regions near the middle of the disc (r = 4–6 mm for total disc radius equal to 10 mm) after a single turn. However, at the periphery region (r = 8–10 mm), the results of the FEM simulations deviate from the theoretical values because of "dead metal zone" phenomenon [18]. Therefore, the strain of middle region of the disc after HPT is well fitted according to the theoretical equation

$$\varepsilon = \frac{\gamma}{\sqrt{3}} = \frac{r\theta}{\sqrt{3}h}, \quad (2)$$

where γ is the shear strain, r is the radial distance from the centre of the disc-shaped workpiece, θ is the rotation angle and h is the thickness of the workpiece.

Nevertheless, HPT has similarly as ECAP a significant limitation: the small size of the processed specimens. Materials produced by classic HPT are primarily used in modern, micro-sized specialized industrial fields, such as micro-electro-mechanical systems [16]. Unlike ECAP, which can be relatively easily adapted for larger samples [19], upscaling of HPT is more demanding. Several efforts in upscaling of HPT have been undertaken [20]. There has been some progress with the extension of HPT to cylindrical samples. However, this approach has been found to cause strong microstructural inhomogeneities in the vertical sections of the cylinders [21]. A recent variation of the method is the hollow cone HPT, which processes hollow cone-shaped samples. This technique is useful for manufacturing projectiles, valves, nozzles, drill heads, and other engineering parts that require superior mechanical properties [22]. Another technique of HPT upscaling has recently been reported by the Japanese group led by prof. Z. Horita who introduced the technique of High Pressure Sliding (HPS) which allows to prepare sheets of the macroscopic size (up to 100 mm in length) while maintaining excellent properties reported in materials processed by classic HPT [20], [23].

1.1.3. Spark plasma sintering

The production of ultra-fine-grained materials is not limited to severe plastic deformation techniques, known as the “top-down” approach. An alternative method via powder compactization, known as “bottom-up” approach, offers a promising alternative for the production of such materials.

It has been widely recognized that the advanced technique of powder metallurgy plays a crucial role in enhancing mechanical and corrosion properties of materials [24]. This improvement can be attributed to the small grain size and the high concentration of alloying elements present in the solid solution, after rapid cooling, and possible presence of very fine intermetallic phases. It should be noted that the properties of the material are significantly influenced by the specific selected powder processing technique and the method employed for compaction [25].

Spark plasma sintering (SPS) represents an advanced sintering method that enables a rapid production. This technique involves powder pressing, and the application of a high current through the powder, where the high resistance between particles generates Joule heating. This process is completed in few minutes, allowing the microstructure of originally powder particles to remain largely unchanged [26]. As a result, the final material retains a fine-grained structure with a high concentration of alloying elements in the solid solution [27]. The sintering by direct Joule heating is associated with the electrical resistance of the materials and influenced by the size, the geometry and surface condition of powder particles [28], [29], [30]. Increasing the number of interfaces with high resistivity can therefore enhance the efficiency of sintering, which can be achieved by sieving the powder to separate particles by size or modifying the particle surfaces.

1.1.4. Superplasticity in UFG materials

The superplastic behaviour exhibited by some magnesium materials is an alternative plastic deformation behaviour allowing to bypass their limited ductility. It represents therefore a promise for their wider application in various industries. Superplasticity allows the material to undergo significant deformation without fracturing, enabling complex shaping processes and the production of intricate components with high precision. There are two common definitions of superplastic behaviour. The first one relates the superplasticity to the minimum elongation to fracture (elongation > 400 %, true strain > 160 %) [31] while the other one to the dimensionless strain rate sensitivity m-parameter (0.3 < m < 0.8) [32], [33], [34]. This duality in definitions brings some ambiguity. However, both conditions are usually fulfilled at the same time. The reason is that strain rate sensitivity effectively suppresses necking and extends the region of plastic stability. The localization of plastic deformation leads in fact to the local increase of strain rate, and due to strain rate sensitivity to consequent increase of stress, which avoids further localization of plastic deformation and premature necking. However, the exact effect of strain rate sensitivity on stability of plastic deformation is not quantitatively agreed.

The plastic stability limit is determined by Considère criterion [35] as the lowest ε, for that holds:

$$\frac{\theta(\varepsilon)}{\sigma(\varepsilon)} < 1, \qquad (3)$$

where ε is the true strain, σ is the true stress and θ is the strain hardening computed at constant strain rate: $\theta(\varepsilon) = \frac{d\sigma(\varepsilon)}{d\varepsilon}$.

As pointed out by Hart [36], if the material presents non-zero strain rate sensitivity, the plastic stability is extended due to the fact that the actual strain rate at the smallest cross-section (potential neck) is increased. The extended plastic stability limit can be expressed as:

$$\frac{\theta(\varepsilon)}{\sigma(\varepsilon)} < 1 - m(\varepsilon), \qquad (4)$$

where $m(\varepsilon)$ is well-known strain rate sensitivity parameter. Note that the actual value of m-parameter for a given ε is required. Equation (4) clearly extends the plastic stability range since $m(\varepsilon)$ is positive. Considering that $m(\varepsilon) < 1$, stable plastic deformation is possible only when the strain hardening $\theta(\varepsilon)$ is positive. However, several experiments show very high elongations even for negative strain hardening (i.e. softening) [37], [38], [39].

More general criterion of plastic stability is provided by Hähner [33]. It is based on the consideration of the microstructure evolution on characteristic intrinsic or extrinsic length scales. These length scales depend on the specific mechanism of plastic deformation and determine the extent of strain rate sensitivity effect on stability of plastic deformation. In particular, for grain boundary sliding (GBS), the Hähner criterion of plastic stability can be written as [33]:

$$\frac{\theta(\varepsilon)}{\sigma(\varepsilon)} < 1 - \left(1 + \frac{\eta}{\varepsilon_0}\right). m(\varepsilon), \tag{5}$$

where $0 < \eta < 1$ is the fraction of plastic deformation realized by GBS and $\varepsilon_0 \approx 0.5$ is elementary deformation step assumed to occur during superplastic flow according to model proposed by Ashby and Verrall [40]. Considering that all plastic deformation is realized by GBS $(\eta = 1)$ the criterion of plastic stability may be expressed in the following form:

$$\frac{\theta(\varepsilon)}{\sigma(\varepsilon)} < 1 - 3\, m(\varepsilon). \tag{6}$$

It is concluded in [33] that if $0 < \theta(\varepsilon) \ll \sigma(\varepsilon)$, then the plastic deformation is stable for $m(\varepsilon) > 1/3$. However, the range of plastic stability according to equation (6) can be obviously extended to a region of work softening $\theta(\varepsilon) < 0$, as long as the $m(\varepsilon)$ is sufficiently large.

It is well-known that a possible superplastic behaviour depends on the microstructure, namely on the grain size and other factors affecting the diffusivity [41]. Ultra-fine-grained materials (UFG) are therefore promising candidates and their enhanced superplastic behaviour was reported in numerous studies including those on hexagonal materials [37], [42]. The disadvantage of UFG materials is that their microstructure is often not stable at increased temperatures, and grain growth occurs.

2. Development of the ultra-fine-grained magnesium materials

2.1. Mg-Al-Zn ultra-fine-grained alloys

Magnesium alloys with aluminium (Al) and zinc (Zn) as alloying elements have been extensively studied over the past decades. These commercial alloys have found applications in various industrial sectors due to their favourable properties. Moreover, mechanical characteristics and performance of these materials can be significantly improved by processing using techniques such as equal-channel angular pressing (ECAP) and high pressure torsion (HPT). These processes result in ultra-fine grained microstructures, which markedly enhance the mechanical strength compared to their coarse-grained counterparts.

This enhancement is attributed to the refined microstructure and undergoing alteration of plastic deformation mechanisms. Detailed investigations of these materials are documented in the following selected papers, providing comprehensive insights into their microstructural evolution and mechanical properties. These studies underscore the potential of advanced processing techniques in significantly improving the performance of magnesium alloys in industrial applications.

[JS1] J. Vrátná, M. Janeček, J. Čížek, D.J. Lee, E.Y. Yoon, H.S. Kim: Mechanical properties and microstructure evolution in ultrafine-grained AZ31 alloy processed by severe plastic deformation, Journal of Materials Science, 2013, Vol. 48, pp. 4705–4712.

The primary aim of this paper was to contribute to the long-lasting discussion in the UFG research community about the efficiency of grain refinement and enhancement of mechanical properties of magnesium alloys prepared by two different techniques of SPD: ECAP and HPT. For this purpose, we prepared two microstructure conditions of commercial alloy AZ31. The first one was achieved by extrusion followed by equal channel angular pressing (hereinafter referred to as EX-ECAP) while the second one by high pressure torsion (HPT) at room temperature.

Mechanical properties, dislocation density, microstructure evolution and grain fragmentation of the alloy in these microstructure conditions were investigated and compared in detail.

EX-ECAP after four passes of ECAP (EX-ECAP 4) produced a homogeneous microstructure across the specimen cross-section corresponding to the grain size reduction of the original coarse-grained structure by a factor of 100, whereas HPT resulted, as expected, in a laterally inhomogeneous microstructure, with grain sizes decreasing from the centre towards the periphery. However, after 15 HPT turns, the microstructure and microhardness inhomogeneities were minimized, yielding an almost homogeneous ultra-fine-grained structure, corresponding to the grain size reduction by a factor of about 1000.

HPT proved to be more effective in grain refinement, leading also to significantly higher microhardness, both due to the achieved extreme equivalent strain. Transmission electron microscopy (TEM) observations confirmed that the peripheral parts of HPT-processed samples exhibited extremely ultra-fine grains, around 100 nm, contributing obviously to the increased microhardness.

The dislocation densities, measured in the same areas as the microhardness, were higher in HPT specimens, due to higher equivalent strain and the different processing temperatures. Processing by EX-ECAP required using elevated temperatures (180 °C) to avoid cracking of the hexagonal structure of magnesium which lacks five independent slip systems for uniform deformation at room temperature [43]. Conversely, HPT can be performed at room temperature due to a high hydrostatic pressure (2.5 GPa in our case), which enabled uniform deformation by activating additional slip systems [44]. Elevated temperature of EX-ECAP processing causes imminent partial recovery of dislocations which limits achievable grain refinement. For instance, processing by additional four ECAP passes (EX-ECAP 8) did not provide additional grain refinement.

I prepared all the samples for experiment and conducted the microhardness measurements. I summarized the obtained results and compared the properties of regions subjected to the same equivalent strain by both SPD techniques. Furthermore, I analysed the findings and contributed to the writing of the manuscript.

[JS2] J. Vrátná, B. Hadzima, M. Bukovina, M. Janeček: Room temperature corrosion properties of AZ31 magnesium alloy processed by extrusion and equal channel angular pressing, Journal of Materials Science, 2013, Vol. 48, pp. 4510–4516.

In this study, UFG magnesium alloy AZ31 prepared by a combined two-step process (hot extrusion with subsequent 8 passes of ECAP - EX-ECAP 8) was investigated and compared to the corrosion behaviour of the extruded coarse-gained material. Classical potenciodynamic tests in corrosion solution of 0.1 M sodium chloride combined with the advanced electrochemical impedance spectroscopy tests were employed to access the corrosion properties of the alloy in both microstructural conditions. Electrochemical properties on the specimen surface were correlated with microstructure evolution. This paper was one of the first pioneering works unveiling the corrosion properties of an UFG magnesium alloy.

The main finding, subsequently well-accepted in the UFG community, was that the corrosion properties of the UFG material depend strongly on the immersion time. For shorter times no improvement of corrosion properties in the UFG material was observed. However, for longer times (>168 hours) the material in UFG condition exhibits superior corrosion resistance compared to the coarse-grained material. This peculiar enhancement of corrosion properties in UFG condition was attributed to the different mechanism of detachment of protective corrosion products. The microstructure after extrusion contains relatively large grains, whereas the material after EX-ECAP exhibits a homogeneous UFG microstructure. Consequently, the material after EX-ECAP offers more corrosion nucleation sites and only small clusters of corrosion products are formed on the UFG microstructure. As the result, UFG microstructure prevents detaching large clusters (flakes) of corrosion products from the surface leading to easier and faster restoration of the corrosion protective layer on the surface of UFG material eventually enhancing long-term corrosion resistance.

I conducted all experiments, including measurements of corrosion properties and scanning electron microscopy (SEM) observations. I summarized the obtained results, engaged in discussions, and wrote the manuscript. I have conducted most of the corrosion experiments during research stays at University of Žilina, Slovakia, at the research group of Prof. Hadzima.

[JS3] J. Stráská, M. Janeček, J. Čížek, J. Stráský, B. Hadzima: Microstructure stability of ultra-fine grained magnesium alloy AZ31 processed by extrusion and equal-channel angular pressing (EX-ECAP), Materials Characterization, 2014, Vol. 94, pp. 69-79.

This highly-cited paper focuses on the microstructure stability of AZ31 magnesium alloy processed by EX-ECAP (4 passes) at elevated temperatures. Microstructure stability is of primary interest for subsequent material processing and its potential use under elevated temperatures. To assess the microstructure stability of the UFG material, it was annealed isochronally at temperatures ranging from 150 °C to 500 °C for 1 hour. Several complementary experimental techniques, including microhardness measurements, electron backscatter diffraction (EBSD), and positron annihilation spectroscopy (PAS), were employed to identify the softening processes occurring in this temperature range.

At lower annealing temperatures (approximately 190 °C), softening, indicated by a drop in microhardness, is primarily controlled by dislocation annihilation, as confirmed by PAS. On the other hand, at higher annealing temperatures (around 300 °C and above), grain growth becomes the principal softening mechanism, as evidenced by EBSD.

The experimental observations were complemented by theoretical analysis of thermally activated processes controlling the evolution of the UFG structure. The kinetics of grain growth was analysed using the kinetic equations, and the activation energies for grain growth in different temperature ranges were calculated. The evolution of microhardness in the higher temperature range (250 °C–500 °C) can be successfully explained by the Hall–Petch relation [45], [46]. In the lower temperature range, dislocation density plays a significant role, resulting in microhardness values significantly higher than those predicted by the Hall–Petch extrapolation. To obtain reliable estimates of the Hall–Petch relation parameters, the effect of dislocations must be considered and quantified.

I performed all the experiments (except PAS), which included annealing the samples, microhardness measurements, both light and scanning electron microscopy observations and EBSD measurements. I summarized the experimental results, determine the evolution of grain size from EBSD observations, applied the kinetics equations and calculated the activation energies for grain growth. Finally, I discussed the achieved results and prepared the manuscript for publication.

[JS4] J. Stráská, M. Janeček, J. Gubicza, T. Krajňák, E.Y. Yoon, H.S. Kim: Evolution of microstructure and hardness in AZ31 alloy processed by high pressure torsion, Materials Science & Engineering A, 2015, Vol. 625, pp. 98–106.

This influential paper investigates the microstructure evolution and strengthening mechanisms in AZ31 magnesium alloy subjected to high pressure torsion (HPT). HPT deformation at room temperature induces even more grain refinement than previously studied EX-ECAP process. However, HPT introduces lateral inhomogeneity in both microhardness and microstructure which was the main subject of the performed experimental study. Notably, lower microhardness values are observed near the centre compared to the periphery of the specimen. This microhardness variation gradually diminishes as the zone of enhanced microhardness expands from the periphery towards the centre, indicating a smearing out of lateral strain inhomogeneity imposed by HPT.

The lateral strain distribution influences the rate of grain fragmentation, resulting in much finer grains at the periphery than in the central region of the specimen. Most importantly, with increasing strain, both microhardness and microstructure homogenize. Microhardness examinations are closely correlated with microstructural findings throughout the study.

Even in the initial stages of HPT without rotation, a high density of dislocations ($\rho \approx 3 \times 10^{14}$ m^{-2}) is observed. As HPT torsion progresses, crystallite size decreases while dislocation density further increases. These parameters reach saturation at the periphery of the specimen early in the deformation process, with the maximum dislocation density reaching about 6×10^{14} m^{-2} and the minimum crystallite size stabilizing around 70 nm after just ¼ turn at room temperature. Further increases in the number of HPT revolutions up to 15 turns do not lead to significant changes in the dislocation structure at the periphery, indicating saturation with imposed strain. However, such refined zone expands gradually towards the sample centre.

Interestingly, the dislocation density saturates earlier than the grain refinement process, implying that grain size reduction continues even after dislocation density reaches its peak. The strengthening effect due to dislocations outweighs the contribution from high-angle grain boundaries (HAGBs) disregarding the number of HPT revolutions or radial positions in the disc samples. Earlier grain refinement at the periphery of HPT-processed discs was also documented by transmission electron microscopy (TEM).

In this research, I played a central role in designing the study; I performed majority of the experiments, including sample preparation, microhardness measurements, scanning electron microscopy (SEM) observations, and electron backscatter diffraction (EBSD) measurements. I synthesized and summarized the experimental findings. Collaborating closely with Prof. Gubicza from Eötvös Loránd University in Budapest, Hungary, we extensively discussed the results and jointly authored the manuscript. This paper also represents the culmination of our long-lasting collaboration with Prof. Hyoung Seop Kim's research team at POSTECH University in Pohang, South Korea, highlighting our collective efforts in advancing the understanding of materials science through rigorous experimentation and analysis.

[JS5] J. Stráská, J. Stráský, P. Minárik, M. Janeček, B. Hadzima: Continuous measurement of m-parameter for analyzing plastic instability in a superplastic ultra-fine grained magnesium alloy, Materials Science & Engineering A, 2017, Vol. 684, pp. 110-114.

The UFG structure in magnesium alloys enables superplastic behaviour, offering unique mechanical properties ideal for high-temperature applications. However, at elevated temperatures, the UFG microstructure tends to coarsen, impacting the superplastic characteristics of the material. Conventional testing methodologies face challenges related to the accuracy in assessing superplastic behaviour. As mentioned in the Introduction, the m-parameter, a critical indicator of superplasticity, is uneasy to be determined in real-time during testing. However, m-parameter indeed evolves during the mechanical test at elevated temperatures due to ongoing microstructural changes.

To address this issue, our study introduced an innovative methodology aimed at measuring the evolution of the m-parameter throughout the entire tensile test using a computer-controlled tensile deformation system. An advanced use of this system involved regular small variations in strain rate during testing. Specifically, our approach involved regular alternating of strain rates to capture the dynamic changes in the m-parameter during deformation.

The developed methodology was applied to investigate the superplastic behaviour of an UFG magnesium alloy, providing valuable insights into its mechanical response and validating existing physical models regarding the prediction of plastic instability under superplastic conditions. By systematically measuring and analysing the evolution of the m-parameter, we were able to assess comprehensively the superplastic characteristics of the alloy in UFG condition.

In this research, I played an important role in devising the methodology for measuring the evolution of the m-parameter. I conducted all deformation tests and experimental measurements, ensuring accurate data collection and analysis. Furthermore, I synthesized the obtained results, facilitated discussions, consolidated findings and contributed to the advancement of knowledge in superplasticity and materials science. This effort underscores the importance of innovative testing methodologies in expanding our understanding of UFG magnesium alloys and their potential applications in engineering and technology.

[JS6] P. Minárik, M. Zimina, J. Čížek, J. Stráská, T. Krajňák, M. Cieslar, T. Vlasák, J. Bohlen, G. Kurz, D. Letzig: Increased structural stability in twin-roll cast AZ31 magnesium alloy processed by equal channel angular pressing, Materials Characterization, 2019, Vol. 153, pp. 199-207.

This study investigates the impact of ECAP on the microstructure of AZ31 magnesium alloy in two initial conditions before ECAP processing: conventionally cast (CC) and twin-roll cast (TRC). Twin-roll casting represents an advanced continuous casting technique, where molten metal is fed through a nozzle into the gap between two rotating water-cooled rolls, rapidly cooling and solidifying to form thin sheets (4–6 mm) with a high-quality surface due to the high cooling rate and non-intermittent withdrawal regime [47]. TRC strips typically exhibit a heterogeneous structure with finer grains ranging from 50 to 100 μm in diameter [48], [49]. On the other hand, conventionally cast alloys have a more homogeneous structure but coarser grains.

The fine-grained conditions of both TRC and conventionally cast AZ31 alloys were prepared by ECAP, and material characterization focused on microhardness, grain structure, and dislocation density changes. Despite differences in initial microstructure, intensive plastic deformation by ECAP resulted in achieving comparable average grain sizes in both types of initial conditions. The primary difference observed, was the different distribution of nano-scale β $Al_{12}Mg_{17}$ secondary phase particles. These β-phase particles were distributed relatively non-homogeneously in the CC-8P, while a homogenous distribution was observed in the TRC-8P sample, where 8P indicates the number of subsequent ECAP passes in both conditions.

To assess the thermal stability, isochronal annealing between 160–500 °C was conducted, revealing distinctly different responses to annealing of the two studied conditions. Variations in the distribution of secondary phase particles significantly influenced static recovery and grain growth during annealing. Notably, TRC samples exhibited superior thermal stability between 220–340 °C, attributed to a more favourable distribution of secondary phase particles. Above 340 °C, accelerated dissolution of the β $Al_{12}Mg_{17}$ phase smeared the differences and has led to similar microstructural evolutions in both sets of samples. The paper therefore unveils the critical role of secondary phase particle distribution in the thermal stability of fine grained AZ31 alloy.

My proficiency in investigating microstructure stability has made substantial contributions to both experimental procedures and the discussion of results presented in this manuscript. In this research, my role encompassed conducting EBSD measurements and analysing the acquired data. This collaborative endeavour highlights the significance of comprehending microstructural evolution in enhancing the performance and application possibilities of AZ31 magnesium alloys in engineering applications.

2.2. Magnesium ultra-fine-grained alloys with RE elements

Ultra-fine-grained magnesium alloys incorporating rare earth (RE) alloying elements offer significant advantages, including high resistance to ignition and excellent mechanical properties. These characteristics make them promising materials for example for aerospace applications, where weight reduction and material performance are critical. The addition of RE elements to magnesium alloys enhances their thermal stability, making them less prone to ignition and more reliable under high-temperature conditions, which is required in aerospace industry for the case of emergency scenarios.

However, the high cost of RE alloying elements presents a considerable limitation and challenge. Cheap and promising alternative is to use calcium as an alloying element at the extent of expensive rare earth elements. Alloying by calcium on one hand has been shown to significantly improve flammability resistance, but on the other hand it adversely affects mechanical properties. This trade-off necessitates a careful balance in alloy design to optimize both safety and performance. Our research group has designed several magnesium alloys that aim to achieve this balance. Through systematic experimentation and analysis, we strived to develop alloys that leverage the benefits of RE elements while mitigating their drawbacks, thereby advancing the practical application of magnesium alloys in the aerospace industry.

[JS7] D. Dvorský, J. Kubásek, M. Roudnická, F. Průša, D. Nečas, P. Minárik, J. Stráská, D. Vojtěch: The effect of powder size on the mechanical and corrosion properties and the ignition temperature of WE43 alloy prepared by spark plasma sintering, Journal of Magnesium and Alloys, 2021, Vol. 9, Issue 4, pp. 1349-1362.

This study in the highly reputed journal delved into the impact of powder size on mechanical, corrosion and ignition properties of WE43 alloy fabricated using the spark plasma sintering (SPS). The investigation also highlighted the influence of surface conditions through HF (hydrogen fluoride) pre-treatment of the powder, demonstrating that specific interfaces, primarily composed of Y_2O_3 or YF_3, formed between particles during sintering. Materials prepared from finer powder fractions exhibited a higher concentration of these interfaces, significantly influencing the resulting properties.

A notable and quite novel finding was the detrimental effect of YF_3 at particle interfaces, resulting in reduced elongation, as well as tensile yield strength (TYS) and ultimate tensile strength (UTS). Moreover, specific crack propagation patterns were observed, and shown to depend on the microstructure, further impacting material integrity.

On the other hand, an increased volume fraction of interfaces substantially enhanced corrosion resistance by slowing down the corrosion front at each interface barrier. However, near these interfaces, the solid solution was depleted in Y, which was consumed during the formation of Y_2O_3 or YF_3, resulting in localized inhomogeneities and accelerated corrosion rates in larger particles.

Particle size also affected the ignition temperature of magnesium alloys powder, with smaller particles exhibiting higher ignition temperatures due to the enhanced protective effect of the Y_2O_3 surface layer. This was attributed to greater Y supersaturation in the solid solution and shorter diffusion distances towards the surface in smaller particles. Furthermore, chemical treatment of the powder further improved ignition temperatures, reaching up to 700 °C, enhancing safety during storage and use, especially in additive manufacturing applications.

In terms of mechanical properties, a powder mixture approach appeared advantageous. However, for improved corrosion resistance, selecting small particles or chemically treated powders was more beneficial. These materials exhibit not only superior corrosion resistance but also offer enhanced safety due to their higher ignition temperatures.

In this research, my primary contributions centred on the discussion and synthesis of findings presented in the manuscript. I conducted a thorough review and provided editorial input to refine the manuscript clarity and coherence. This paper reflects ongoing collaboration with Prof. Vojtěch's research team at the University of Chemistry and Technology in Prague (UCT) during the course of several projects funded by Czech Science Foundation and underscoring our collective efforts to advance understanding in materials science and engineering.

[JS8] J. Kubásek, P. Minárik, K. Hosová, S. Šašek, M. Knapek, J. Veselý, J. Stráská, D. Dvorský, M. Čavojský, D. Vojtěch: Novel magnesium alloy containing Y, Gd and Ca with enhanced ignition temperature and mechanical properties for aviation applications, Journal of Alloys and Compounds, 2021, Vol. 877, 160089.

Our team embarked on the development of a novel Mg-4.5Gd-3.4Y-2.6Ca (wt.%) alloy specifically designed as a lightweight material intended for the fabrication of aircraft components. This alloy was chosen for its promising mechanical properties and notably high ignition temperature, a critical factor in ensuring safety in aerospace applications. The material synthesis involved casting followed by hot extrusion at two distinct temperatures. Our investigation focused on understanding the impact of these extrusion conditions on the microstructure, mechanical properties, and ignition temperature of the alloy.

The results of our study revealed significant findings regarding the influence of extrusion temperature on several key parameters. Specifically, we observed considerable effects on average grain size, recrystallized fraction, and the texture formation in the material. These microstructural variations directly correlated with the mechanical behaviour of the alloy, namely with the average tensile yield strength reaching 300 MPa under the tested conditions.

One of the key characteristics of our alloy was its exceptionally high ignition temperature of approximately 1100 °C, which ranks among the highest ones reported in magnesium alloys. This impressive thermal stability was attributed to a synergistic interaction involving Y, Gd, and Ca oxides, with Y_2O_3 identified as the primary contributor.

The combination of enhanced mechanical properties and high ignition temperature positions our novel alloy as a promising candidate for demanding aircraft applications where both performance and safety are paramount characteristics.

Throughout this research endeavour during continuing cooperation with UCT, I served as the project coordinator, overseeing the research direction and contributing to the development of experimental methodologies. A significant part of the experimental work was carried out by my PhD student, Stanislav Šašek, whose dedication and expertise were invaluable in conducting experiments, analysing data, and contributing to the preparation of the manuscript under my close supervision. Stanislav played an important role in summarizing experimental findings and ensuring that our research outcomes were effectively communicated. This collaborative effort underscores our commitment to advancing materials science and engineering for aerospace applications.

[JS9] P. Minárik, K. Hosová, S. Šašek, J. Kubásek, J. Veselý, R. Král, M. Čavojský, J. Stráská, D. Vojtěch: Ignition-resistant Mg-2Y-2Gd-1Ca alloy for aviation applications, Journal of Alloys and Compounds, 2023, Vol. 948, 169683.

This paper presents a comprehensive study on a novel Mg-2Y-2Gd-1Ca alloy processed by extrusion and equal channel angular pressing (EX-ECAP) aiming to investigate the influence of microstructure on ignition temperature, mechanical properties, and corrosion resistance. The alloy exhibited an impressive ignition temperature of approximately 950 °C, as determined by linear heating experiments, irrespective of the processing method and resulting microstructure. This is a lower value than in the case of previously studied Mg-4.5Gd-3.4Y-2.6Ca (wt.%) alloy, however in terms of potential use it is well-balanced by lower content of expensive rare earth elements Y and Gd.

Thermogravimetry analysis and differential thermal analysis further revealed that the onset of oxidation occurred approximately 50 °C below the ignition temperature. This finding underscores the robust thermal stability of the alloy which is critical for applications in high-temperature environments.

The microstructural evolution due to processing has significant implications to mechanical properties. The extruded alloy, characterized by a partially recrystallized microstructure, demonstrated a favourable balance of strength and ductility. In contrast, EX-ECAP processing resulted in higher strength but reduced ductility due to its finer microstructure.

Corrosion resistance was also evaluated, with initial results showing distinct differences influenced by the processing technique. However, these differences diminished after one day of immersion in a 3.5% NaCl solution, and corrosion rates became comparable after one week. The distribution of corrosion attack, which was more localized in extruded samples due to the presence of larger Mg_2Ca particles compared to those processed by ECAP, made the main distinction between both conditions.

Our work indicates unambiguously that Mg-2Y-2Gd-1Ca alloy exhibits exceptional properties for aircraft manufacturing applications. Its combination of high ignition temperature, superior mechanical properties tailored by processing method, and robust corrosion resistance makes it an attractive candidate for components requiring reliability and performance under demanding operational conditions. This research underscores the potential of the alloy to advance materials science and engineering, particularly in aerospace sectors where stringent requirements for safety, strength, and durability are a paramount issue.

I oversaw and coordinated the research efforts and the co-operation with the UCT group. A substantial part of the experimental work in our team was conducted by my PhD student, Stanislav Šašek, whose dedicated efforts were instrumental in gathering and analysing the experimental data. Under my guidance and supervision, Stanislav meticulously summarized the findings from our experiments and made significant contributions to the preparation of the manuscript. His detailed analysis and interpretations greatly enriched our understanding of the obtained results, ensuring that our findings were accurately presented and effectively communicated. This collaborative effort underscores the importance of mentorship and teamwork in achieving rigorous scientific inquiry and scholarly output.

3. Conclusions and outlook

The research and development of magnesium-based materials remain a broad and significant scientific field. This habilitation thesis addresses two primary areas of focus. The first area concentrates on the study of ultra-fine-grained (UFG) magnesium materials alloyed with aluminium and zinc, with an emphasis on the processes of grain fragmentation, strengthening and thermal stability of microstructure. This investigation was conducted using well-known commercial magnesium alloys; however, these alloys were rarely processed by equal channel angular pressing (ECAP) and high pressure torsion (HPT) at the time of research. The main findings from this area can be summarized as follows:

- Both severe plastic deformation techniques, equal channel angular pressing (ECAP) and high pressure torsion (HPT), induced substantial deformation in the material causing an increase of dislocation density and continuous grain fragmentation and refinement of the microstructure. When comparing the microstructure conditions corresponding to the same equivalent strain introduced by ECAP and HPT, HPT was found to be more effective in grain refinement and dislocation density increase.
- The ultra-fine grain size and high dislocation density result in excellent mechanical properties, particularly microhardness and strength.
- Thermal stability of UFG microstructure was thoroughly studied via dedicated experiments focusing on dislocation density and grain growth. Activation energy for grain growth was determined.
- The UFG microstructure can induce superplastic behaviour, allowing the material to undergo significant deformation without fracture. This property facilitates complex shaping processes and the production of intricate components with high precision. Superplastic behaviour was confirmed and studied in detail in material processed by ECAP.
- In summary, the series of papers (JS1 – JS5) published in years 2013 – 2017 presents a comprehensive study of ultra-fine-grained magnesium alloys, their properties and undergoing physical processes during processing a subsequent treatment. It can be considered as a valuable contribution to the global knowledge about UFG magnesium alloys and in general to processes of grain refinement, microstructure recovery and recrystallization, strengthening, plastic deformation mechanisms including superplasticity and corrosion processes.

The second part of this thesis focuses on newly designed magnesium alloys incorporating rare earth elements and calcium, with the objective to achieve higher ignition temperatures and improved flammability resistance. These alloys were specifically designed to enhance safety and performance in applications where resistance to flammability is crucial, such as accident scenarios in aerospace industry. These materials were studied in both their as-cast and extruded conditions, as well with subsequent processing by ECAP. The most significant findings are as follows:

- One of the most notable and firstly reported characteristics of the newly designed alloys Mg-Gd-Y-Ca are their exceptionally high ignition temperatures, exceeding 1100 °C, which is in the range of the highest reported ignition temperatures in magnesium alloys ever. The remarkable thermal stability of this alloy is attributed to the synergetic interaction among Y, Gd, and Ca oxides, with Y_2O_3 identified as the primary contributor.
- The corrosion resistance of these alloys is acceptable. In extruded materials, corrosion was relatively localized; however, the alloys processed by ECAP exhibited a more uniform corrosion attack and significantly shallower corrosion pits.
- Mg-Y-Gd-Ca alloys demonstrates significant potential for the use in aerospace industry. Their high ignition temperature meeting technical standards, outstanding mechanical properties—refined by specific processing methods—and strong corrosion resistance render them an excellent choice for components that demand reliability and performance under challenging operating conditions.
- This research highlights the capacity of the specially designed magnesium alloys to advance materials science and engineering, especially within the aerospace sector, where stringent requirements for safety, strength, and durability are critical. My research team, and in particular my PhD student S. Šašek, are currently engaged in this research and will continue to work on this intriguing and promising topic. New undergraduate and graduate students are expected to join this challenging research in the future.

4. References

[1] M. Gupta, *Magnesium, magnesium alloys, and magnesium composites*. New York: John Wiley & Sons, 2011.

[2] P. Predko *et al.*, 'Promising Methods for Corrosion Protection of Magnesium Alloys in the Case of Mg-Al, Mg-Mn-Ce and Mg-Zn-Zr: A Recent Progress Review', *Metals*, vol. 11, no. 7, p. 1133, Jul. 2021, doi: 10.3390/met11071133.

[3] Office of Aviation Research Washington, D.C. 20591, *Aircraft Materials Fire Test Handbook: DOT/FAA/AR-00/12*.

[4] N. V. Ravi Kumar, J. J. Blandin, M. Suéry, and E. Grosjean, 'Effect of alloying elements on the ignition resistance of magnesium alloys', *Scr. Mater.*, vol. 49, no. 3, pp. 225–230, Aug. 2003, doi: 10.1016/S1359-6462(03)00263-X.

[5] G. Gottstein, *Physical foundations of materials science*. Berlin ; New York: Springer, 2004.

[6] V. M. Segal, V. I. Reznikov, A. E. Drobyshevskiy, and V. I. Kopylov, 'Plastic working of metals by simple shears', *Russ. Metall.*, vol. 1, pp. 99–105, 1981.

[7] P. W. Bridgman, 'Effects of High Shearing Stress Combined with High Hydrostatic Pressure', *Phys. Rev.*, vol. 48, no. 10, pp. 825–847, Nov. 1935, doi: 10.1103/PhysRev.48.825.

[8] R. Z. Valiev and I. V. Alexandrov, 'Nanostructured materials from severe plastic deformation', *Nanostructured Mater.*, vol. 12, no. 1–4, pp. 35–40, Jan. 1999, doi: 10.1016/S0965-9773(99)00061-6.

[9] Y. Saito, H. Utsunomiya, N. Tsuji, and T. Sakai, 'Novel ultra-high straining process for bulk materials—development of the accumulative roll-bonding (ARB) process', *Acta Mater.*, vol. 47, no. 2, pp. 579–583, Jan. 1999, doi: 10.1016/S1359-6454(98)00365-6.

[10] J. Y. Huang, Y. T. Zhu, H. Jiang, and T. C. Lowe, 'Microstructures and dislocation configurations in nanostructured Cu processed by repetitive corrugation and straightening', *Acta Mater.*, vol. 49, no. 9, pp. 1497–1505, May 2001, doi: 10.1016/S1359-6454(01)00069-6.

[11] C. Xu, S. Schroeder, P. B. Berbon, and T. G. Langdon, 'Principles of ECAP–Conform as a continuous process for achieving grain refinement: Application to an aluminum alloy', *Acta Mater.*, vol. 58, no. 4, pp. 1379–1386, Feb. 2010, doi: 10.1016/j.actamat.2009.10.044.

[12] C. F. Davis, A. J. Griebel, and T. C. Lowe, 'Isothermal Continuous Equal Channel Angular Pressing of Magnesium Alloy AZ31', *JOM*, vol. 72, no. 7, pp. 2603–2611, Jul. 2020, doi: 10.1007/s11837-020-04195-4.

[13] Stráská, Jitka, *Doctoral Thesis: Physical properties of ultrafine-grained magnesium based alloys prepared by various severe plastic deformation techniques*. 2014.

[14] Y. Iwahashi, J. Wang, Z. Horita, M. Nemoto, and T. G. Langdon, 'Principle of equal-channel angular pressing for the processing of ultra-fine grained materials', *Scr. Mater.*, vol. 35, no. 2, pp. 143–146, Jul. 1996, doi: 10.1016/1359-6462(96)00107-8.

[15] R. Z. Valiev and T. G. Langdon, 'Principles of equal-channel angular pressing as a processing tool for grain refinement', *Prog. Mater. Sci.*, vol. 51, no. 7, pp. 881–981, Sep. 2006, doi: 10.1016/j.pmatsci.2006.02.003.

[16] A. P. Zhilyaev and T. G. Langdon, 'Using high-pressure torsion for metal processing: Fundamentals and applications', *Prog. Mater. Sci.*, vol. 53, no. 6, pp. 893–979, Aug. 2008, doi: 10.1016/j.pmatsci.2008.03.002.

[17] Y. Song, E. Y. Yoon, D. J. Lee, J. H. Lee, and H. S. Kim, 'Mechanical properties of copper after compression stage of high-pressure torsion', *Mater. Sci. Eng. A*, vol. 528, no. 13–14, pp. 4840–4844, May 2011, doi: 10.1016/j.msea.2011.02.020.

[18] D. J. Lee, E. Y. Yoon, L. J. Park, and H. S. Kim, 'The dead metal zone in high-pressure torsion', *Scr. Mater.*, vol. 67, no. 4, pp. 384–387, Aug. 2012, doi: 10.1016/j.scriptamat.2012.05.024.

[19] S. Ferrasse, V. M. Segal, F. Alford, J. Kardokus, and S. Strothers, 'Scale up and application of equal-channel angular extrusion for the electronics and aerospace industries', *Mater. Sci. Eng. A*, vol. 493, no. 1–2, pp. 130–140, Oct. 2008, doi: 10.1016/j.msea.2007.04.133.

[20] Z. Horita, Y. Tang, T. Masuda, and Y. Takizawa, 'Severe Plastic Deformation under High Pressure: Upsizing Sample Dimensions', *Mater. Trans.*, vol. 61, no. 7, pp. 1177–1190, Jul. 2020, doi: 10.2320/matertrans.MT-M2020074.

[21] G. Sakai, K. Nakamura, Z. Horita, and T. G. Langdon, 'Developing high-pressure torsion for use with bulk samples', *Mater. Sci. Eng. A*, vol. 406, no. 1–2, pp. 268–273, Oct. 2005, doi: 10.1016/j.msea.2005.06.049.

[22] H. Y. Um *et al.*, 'Hollow cone high-pressure torsion: Microstructure and tensile strength by unique severe plastic deformation', *Scr. Mater.*, vol. 71, pp. 41–44, Jan. 2014, doi: 10.1016/j.scriptamat.2013.09.032.

[23] T. Fujioka and Z. Horita, ‘Development of High-Pressure Sliding Process for Microstructural Refinement of Rectangular Metallic Sheets’, *Mater. Trans.*, vol. 50, no. 4, pp. 930–933, 2009, doi: 10.2320/matertrans.MRP2008445.

[24] J. Kubásek, D. Dvorský, M. Čavojský, D. Vojtěch, N. Beronská, and M. Fousová, ‘Superior Properties of Mg–4Y–3RE–Zr Alloy Prepared by Powder Metallurgy’, *J. Mater. Sci. Technol.*, vol. 33, no. 7, pp. 652–660, Jul. 2017, doi: 10.1016/j.jmst.2016.09.019.

[25] D. Dvorský, J. Kubásek, D. Vojtìch, M. Čavojský, and P. Minárik, ‘Effect of heat pre-treatment and extrusion on the structure and mechanical properties of WZ21 magnesium alloy’, *Mater. Tehnol.*, vol. 52, no. 4, pp. 499–505, Aug. 2018, doi: 10.17222/mit.2017.214.

[26] M. Tokita, ‘Spark Plasma Sintering (SPS) Method, Systems, and Applications’, in *Handbook of Advanced Ceramics*, Elsevier, 2013, pp. 1149–1177. doi: 10.1016/B978-0-12-385469-8.00060-5.

[27] P. Minárik, M. Zemková, F. Lukáč, J. Bohlen, M. Knapek, and R. Král, ‘Microstructure of the novel biomedical Mg–4Y–3Nd alloy prepared by spark plasma sintering’, *J. Alloys Compd.*, vol. 819, p. 153008, Apr. 2020, doi: 10.1016/j.jallcom.2019.153008.

[28] A. Mostafaei, C. Hilla, E. L. Stevens, P. Nandwana, A. M. Elliott, and M. Chmielus, ‘Comparison of characterization methods for differently atomized nickel-based alloy 625 powders’, *Powder Technol.*, vol. 333, pp. 180–192, Jun. 2018, doi: 10.1016/j.powtec.2018.04.014.

[29] J. Gu, S. Gu, L. Xue, S. Wu, and Y. Yan, ‘Microstructure and mechanical properties of in-situ Al13Fe4/Al composites prepared by mechanical alloying and spark plasma sintering’, *Mater. Sci. Eng. A*, vol. 558, pp. 684–691, Dec. 2012, doi: 10.1016/j.msea.2012.08.076.

[30] F. Průša, D. Vojtěch, M. Bláhová, A. Michalcová, T. F. Kubatík, and J. Čížek, ‘Structure and mechanical properties of Al–Si–Fe alloys prepared by short-term mechanical alloying and spark plasma sintering’, *Mater. Des.*, vol. 75, pp. 65–75, Jun. 2015, doi: 10.1016/j.matdes.2015.03.016.

[31] M. Kawasaki, R. B. Figueiredo, and T. G. Langdon, ‘The Requirements for Superplasticity with an Emphasis on Magnesium Alloys’, *Adv. Eng. Mater.*, vol. 18, no. 1, pp. 127–131, Jan. 2016, doi: 10.1002/adem.201500068.

[32] T. G. Langdon, ‘The mechanical properties of superplastic materials’, *Metall. Trans. A*, vol. 13, no. 5, pp. 689–701, May 1982, doi: 10.1007/BF02642383.

[33] P. Hähner, 'A generalized criterion of plastic instabilities and its application to creep damage and superplastic flow', *Acta Metall. Mater.*, vol. 43, no. 11, pp. 4093–4100, Nov. 1995, doi: 10.1016/0956-7151(95)00091-9.

[34] J. D. Bressan and B. Baudelet, 'Theoretical investigations on the constitutive equations for superplastic materials', *J. Mater. Process. Technol.*, vol. 31, no. 1–2, pp. 217–224, May 1992, doi: 10.1016/0924-0136(92)90022-K.

[35] M. Considère, 'The use of iron and steel in structures', vol. 9, pp. 574–769, 1885.

[36] E. W. Hart, 'Theory of the tensile test', *Acta Metall.*, vol. 15, no. 2, pp. 351–355, Feb. 1967, doi: 10.1016/0001-6160(67)90211-8.

[37] R. B. Figueiredo and T. G. Langdon, 'Record Superplastic Ductility in a Magnesium Alloy Processed by Equal-Channel Angular Pressing', *Adv. Eng. Mater.*, vol. 10, no. 1–2, pp. 37–40, Feb. 2008, doi: 10.1002/adem.200700315.

[38] R. Panicker, A. H. Chokshi, R. K. Mishra, R. Verma, and P. E. Krajewski, 'Microstructural evolution and grain boundary sliding in a superplastic magnesium AZ31 alloy', *Acta Mater.*, vol. 57, no. 13, pp. 3683–3693, Aug. 2009, doi: 10.1016/j.actamat.2009.04.011.

[39] R. K. Islamgaliev, R. Z. Valiev, R. S. Mishra, and A. K. Mukherjee, 'Enhanced superplastic properties in bulk metastable nanostructured alloys', *Mater. Sci. Eng. A*, vol. 304–306, pp. 206–210, May 2001, doi: 10.1016/S0921-5093(00)01440-4.

[40] M. F. Ashby and R. A. Verrall, 'Diffusion-accommodated flow and superplasticity', *Acta Metall.*, vol. 21, no. 2, pp. 149–163, Feb. 1973, doi: 10.1016/0001-6160(73)90057-6.

[41] Y. Umakoshi *et al.*, 'The role of dislocations in high-strain-rate superplasticity of an Al–Ni–misch metal alloy', *Acta Mater.*, vol. 46, no. 13, pp. 4469–4478, Aug. 1998, doi: 10.1016/S1359-6454(98)00159-1.

[42] R. B. Figueiredo, M. Kawasaki, C. Xu, and T. G. Langdon, 'Achieving superplastic behavior in fcc and hcp metals processed by equal-channel angular pressing', *Mater. Sci. Eng. A*, vol. 493, no. 1–2, pp. 104–110, Oct. 2008, doi: 10.1016/j.msea.2007.06.090.

[43] R. Von Mises, 'Mechanik der plastischen Formänderung von Kristallen', *Z. Für Angewande Math. Mech.*, no. 8, pp. 161–185, 1928.

[44] J. P. Hirth, *Theory of dislocations*, 2nd ed. Malabar, FL: Krieger Pub. Co, 1992.

[45] E. O. Hall, 'The Deformation and Ageing of Mild Steel: III Discussion of Results', *Proc. Phys. Soc. Sect. B*, vol. 64, no. 9, pp. 747–753, Sep. 1951, doi: 10.1088/0370-1301/64/9/303.

[46] N. J. Petch, 'The cleavage strength of polycrystals', *J. Iron Steel Inst. Lond.*, vol. 173, pp. 25–28, 1953.

[47] G. Kurz, J. Bohlen, D. Letzig, and K. U. Kainer, 'Influence of Process Parameters on Twin Roll Cast Strip of the Alloy AZ31', *Mater. Sci. Forum*, vol. 765, pp. 205–209, Jul. 2013, doi: 10.4028/www.scientific.net/MSF.765.205.

[48] M. Masoumi, F. Zarandi, and M. Pekguleryuz, 'Microstructure and texture studies on twin-roll cast AZ31 (Mg–3wt.%Al–1wt.%Zn) alloy and the effect of thermomechanical processing', *Mater. Sci. Eng. A*, vol. 528, no. 3, pp. 1268–1279, Jan. 2011, doi: 10.1016/j.msea.2010.10.003.

[49] N. S. Barekar and B. K. Dhindaw, 'Twin-Roll Casting of Aluminum Alloys – An Overview', *Mater. Manuf. Process.*, vol. 29, no. 6, pp. 651–661, Jun. 2014, doi: 10.1080/10426914.2014.912307.

5. List of publications

[JS1] J. Vrátná, M. Janeček, J. Čížek, D.J. Lee, E.Y. Yoon, H.S. Kim: Mechanical properties and microstructure evolution in ultrafine-grained AZ31 alloy processed by severe plastic deformation, Journal of Materials Science, 2013, Vol. 48, pp. 4705–4712. DOI: 10.1007/s10853-013-7151-x

[JS2] J. Vrátná, B. Hadzima, M. Bukovina, M. Janeček: Room temperature corrosion properties of AZ31 magnesium alloy processed by extrusion and equal channel angular pressing, Journal of Materials Science, 2013, Vol. 48, pp. 4510–4516. DOI: 10.1007/s10853-013-7173-4

[JS3] J. Stráská, M. Janeček, J. Čížek, J. Stráský, B. Hadzima: Microstructure stability of ultra-fine grained magnesium alloy AZ31 processed by extrusion and equal-channel angular pressing (EX-ECAP), Materials Characterization, 2014, Vol. 94, pp. 69-79. https://doi.org/10.1016/j.matchar.2014.05.013

[JS4] J. Stráská, M. Janeček, J. Gubicza, T. Krajňák, E.Y. Yoon, H.S. Kim: Evolution of microstructure and hardness in AZ31 alloy processed by high pressure torsion, Materials Science & Engineering A, 2015, Vol. 625, pp. 98–106. http://dx.doi.org/10.1016/j.msea.2014.12.005

[JS5] J. Stráská, J. Stráský, P. Minárik, M. Janeček, B. Hadzima: Continuous measurement of m-parameter for analyzing plastic instability in a superplastic ultra-fine grained magnesium alloy, Materials Science & Engineering A, 2017, Vol. 684, pp. 110-114. http://dx.doi.org/10.1016/j.msea.2016.12.027

[JS6] P. Minárik, M. Zimina, J. Čížek, J. Stráská, T. Krajňák, M. Cieslar, T. Vlasák, J. Bohlen, G. Kurz, D. Letzig: Increased structural stability in twin-roll cast AZ31 magnesium alloy processed by equal channel angular pressing, Materials Characterization, 2019, Vol. 153, pp. 199-207. https://doi.org/10.1016/j.matchar.2019.05.006

[JS7] D. Dvorský, J. Kubásek, M. Roudnická, F. Průša, D. Nečas, P. Minárik, J. Stráská, D. Vojtěch: The effect of powder size on the mechanical and corrosion properties and the ignition temperature of WE43 alloy prepared by spark plasma sintering, Journal of Magnesium and Alloys, 2021, Vol. 9, Issue 4, pp. 1349-1362. https://doi.org/10.1016/j.jma.2020.12.012

[JS8] J. Kubásek, P. Minárik, K. Hosová, S. Šašek, M. Knapek, J. Veselý, J. Stráská, D. Dvorský, M. Čavojský, D. Vojtěch: Novel magnesium alloy containing Y, Gd and Ca with enhanced ignition temperature and mechanical properties for aviation applications, Journal of Alloys and Compounds, 2021, Vol. 877, 160089. https://doi.org/10.1016/j.jallcom.2021.160089

[JS9] P. Minárik, K. Hosová, S. Šašek, J. Kubásek, J. Veselý, R. Král, M. Čavojský, J. Stráská, D. Vojtěch: Ignition-resistant Mg-2Y-2Gd-1Ca alloy for aviation applications, Journal of Alloys and Compounds, 2023, Vol. 948, 169683. https://doi.org/10.1016/j.jallcom.2023.169683

6. Reprints of the selected papers