

Charles University
Faculty of Mathematics and Physics

HABILITATION THESIS



An acoustic emission study of plasticity in crystalline materials

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Preface

Acoustic emission (AE) is a well-known phenomenon from everyday life and it is commonly linked to “sound of danger” as, for example, a sound of an earthquake, cracking ice, loose snow avalanche, etc. AE is defined as transient elastic waves produced by a sudden release of energy due to a local dynamic change within the material. The wavelength of the emitted elastic waves cannot exceed the maximum size of the material and may vary from the nanometre scale for moving defects in a crystal lattice, such as dislocations, up to the kilometre for earthquake-induced cracking in the Earth’s Crust [1]. Thus, AE is the materials-related phenomenon whose essential role often extends over many scales in time and space (multi-scale).

Historically, the first evidence of AE can be dated to a few thousand years BC, when audible sounds were noted during cooling of ceramics or during plastic deformation of pure tin. In the early 20th century, AE was firstly correlated with deformation behaviour of metals. Jan Czocharlski [2] related tin and zinc cry to twinning and later, A. Portevin and F. Le Châtelier [3] observed AE from a stressed Al-Cu-Mn alloy exhibiting plastic instabilities. Pioneer works about applications of AE in materials engineering [4, 5] published in 1950 are generally recognized as the beginning of modern day AE testing [6].

Recently, in addition to using AE in many engineering applications (structural integrity monitoring, quality control, system feedback, process monitoring, etc.), the performance of contemporary AE facilities and improved possibilities for analysing large amounts of data open window for a detailed study of collective phenomena in condensed matter physics and materials science. The AE technique belongs to the non-destructive *in-situ* methods. It offers integral information from the entire volume of the material about dynamic processes due to the loading of the material. Therefore, it can be used for a study of dislocation motion and twinning which are the main deformation mechanisms in crystalline materials.

In this habilitation thesis, I propose a collection of 10 journal publications, which are thematically divided into two subchapters.

The first subchapter deals with using the AE technique for a study of general aspects of plastic deformation in crystalline materials. The concept of self-organized criticality through a power-law distribution of probability density of the AE energy or dislocation avalanches was applied on the AE data. The power-law distributions were firstly plotted for single crystals with hexagonal and cubic lattice, in order to eliminate the contribution

of grain boundaries to plastic deformation. Then the statistical analyses of the AE data were performed on polycrystalline materials. Various chemical compositions and grain sizes of materials, different loading conditions and the occurrence of a plastic instability in some Al-Mg alloys enable us a comprehensive study of dislocation dynamics under different conditions. The interest of the scientific community in our results is demonstrated by citations in highly ranked journals (Csikor et al., *Science* 318 (2007); Papanikolaou et al., *Nature* 490 (2012); Zhang et al., *Progress in Materials Science* 61 (2014)).

In the second subchapter, the AE features were related to individual deformation mechanisms which occur during mechanical loading of Mg alloys. The combination of *in-situ* and *post-mortem* methods shed more light on the understanding of deformation processes in Mg alloys and it contributed to the design of novel Mg alloys with a high potential for technical applications. Specifically, our attention was paid to the explanation and possible reduction of tensile/compression asymmetry and anisotropy at the macroscopic yield strength in wrought Mg alloys. Experimental data from deformation tests were applied in the strain hardening model which is based on the evolution of dislocation density. Furthermore, the microstructural stability and twinning-detwinning process in extruded Mg alloys were studied in detail. Altogether, in the frame of our cooperation with one of the top rated institutes in research and development of Mg alloys (Helmholtz-Zentrum Geesthacht - formerly GKSS), we have published in this field more than 20 papers in journals with an impact factor and received more than 300 citations (without self-citations).

A brief introduction to the AE technique containing a basic characterisation of AE sources, processing and analysis of the AE signal and the AE terminology can be found in Chap. 1. This literature review provides an introduction to understanding the AE results presented in the Chap. 2.

Chapter 2 contains my commentary on the motivation, experimental procedure and major conclusions of the proposed papers.

In Chap. 3, the general conclusions with the relevance of the findings for the scientific community and future perspectives are presented.

Chapter 4 is the list of publications and Chap. 5 contains reprints of the selected papers.

1 Introduction to acoustic emission

Acoustic emission (AE) is defined as transient elastic waves produced by a sudden release of energy due to a local dynamic change within the material. Local irreversible microscopic events in the material result in dissipative and irreversible macroscopic changes, i.e. plasticity. Thus, the discrete and discontinuous microscopic strain mechanism is represented by the unique AE response and the AE technique can be used to investigate collective processes and their statistics. In general, source mechanisms producing AE in crystalline materials are dislocation slip, mechanical twinning, initiation and growth of cracks, phase transformation in metals, fibre breakage and fibre-matrix debonding in composites, and corrosion. In this habilitation thesis, I am only focusing on AE which occurs during plastic deformation of crystalline metals as a consequence of dislocation slip and mechanical twinning, which are the main deformation mechanisms in metals.

1.1 Acoustic emission sources

1.1.1 Dislocation slip

AE from dislocation motion can be produced by relaxation of the elastic stress field in the lattice caused by the passage and annihilation of dislocations, and radiation of wave energy from accelerating or decelerating dislocations (Bremsstrahlung) [7].

AE is usually detected on the material surface and therefore the magnitude of surface displacement from dislocation motion within a material is a decisive parameter, which is responsible for the accuracy of the AE measurement. Using the Green's function, Scruby et al. [8] calculated the peak amplitude of the surface displacement from the first (longitudinal) wave arrival at the epicentre caused by a growth of dislocation loop in an isotropic material. The loop was assumed to lie in a depth D below the surface inclined at 45° to the tensile axis, and to grow at a constant velocity v from zero radius to the final radius r . The peak displacement amplitude was then calculated as:

$$\Delta U = \frac{brvc_T^2}{Dc_L^3} \quad (1)$$

where b is the Burgers vector, c_T and c_L are the transversal and longitudinal wave velocities, respectively. For a pure Al single crystal (crystal diameter of 4 mm, $c_T = 3200 \text{ ms}^{-1}$, $c_L = 6400 \text{ ms}^{-1}$, $b = 0.29 \text{ nm}$, $v = 200 \text{ ms}^{-1}$, $D = 4 \text{ cm}$), it is obtained $\Delta U = 10^{-13} \text{ m}$. For conventional polycrystalline materials, ΔU is calculated of order 10^{15} m

or less. The piezoelectric transducers are sensitive to a surface displacement not less than 10^{-14} m [9] and thus a passage of a single dislocation can hardly produce detectable AE.

According to Tetelman [10], the released energy due to relaxation of the elastic stress field in the lattice caused by the passage of dislocations is given by:

$$E_g = b\tau d \quad (2)$$

where τ is the applied stress and d is the grain size.

AE produced by annihilation of dislocations was widely studied by Natsik et al. [11], who considered the escape of dislocations on the material surface, the annihilation of moving dislocations and the Frank-Read source operation. The energy per unit length of a dislocation line released due to annihilation of a dislocation pair is given by:

$$E_{an} = \frac{\rho b^2 u^2}{8\pi} \gamma \ln \frac{d}{b} \quad (3)$$

where, ρ is the material density, u is the relative velocity of dislocations at the instant of annihilation ($u = 0.1 - c_T$ used in the original paper), $\gamma = 1$ for screw and $1 + (c_T/c_L)^4$ for edge dislocations.

AE caused by the radiation of wave energy from accelerating or decelerating dislocations was firstly studied by Eshelby [12] and Kosevich [13]. Kiesewetter and Schiller [14] have proposed that the generation, motion and stopping of dislocations at the obstacle can be assumed as a periodic dislocation vibration with the amplitude constrained by the distance between the source and the obstacle. The released energy due to the Bremsstrahlung of a single screw dislocation vibrating with a frequency of $\omega_0/2\pi$ can be calculated as:

$$E_B = \frac{Gb^2 L A_d^2 \omega_0^3}{10c_T^3} \quad (4)$$

where G is the shear modulus, L is the length of the dislocation line, A_d is the moving amplitude ($A_d = 2\pi v/\omega_0$) and v is the velocity of the dislocation.

A contribution of all three AE sources to the total AE energy from dislocation motion is demonstrated on polycrystalline Al ($\rho = 2700 \text{ kgm}^{-3}$, $G = 2.64 \text{ GPa}$, $\tau = 4 \text{ MPa}$, $u = 1600 \text{ ms}^{-1}$, $d = 80 \text{ }\mu\text{m}$, $L = 80 \text{ }\mu\text{m}$, $A_d = 40 \text{ }\mu\text{m}$, $\omega_0 = 3.1 \times 10^7 \text{ s}^{-1}$). From the calculated energies ($E_g \sim 9 \times 10^{-8} \text{ Jm}^{-1}$, $E_{an} \sim 3 \times 10^{-10} \text{ Jm}^{-1}$, $E_B \sim 5 \times 10^{-11} \text{ Jm}^{-1}$) is clearly seen that a passage of dislocations mostly contributes to the total AE energy.

A detailed overview of dislocation models of AE and experimental results can be found in [9, 15, 16].

1.1.2 Mechanical twinning

Twinning occurs when a portion of a crystal is sheared in a new position, which is a mirror image of the original crystal lattice. Thus, the crystal shears across particular lattice planes in specific directions. A pioneer work about the twinning mechanism of plastic deformation in metals was done by E. Schmid and G. Wassermann in 1928 [17] and since then a large number of twin formation models have been proposed.

As was already mentioned in Preface, twinning belongs to the first observed sources of AE. The twin nucleation is a result of a collective motion of several hundred dislocations and therefore it is an excellent source of AE [15]. It was shown in [18] that the twin growth does not produce detectable AE. Papirov et al. [19] have documented that even the growth velocity of an elliptical twin is in order of 10^{-3} ms^{-1} the surface displacement caused by twin growth is only $\Delta U = 5 \times 10^{-22} \text{ m}$ (Eq. 1). This surface displacement is definitely below the resolution of the AE setup [20].

1.2 Measurement and analysis of acoustic emission

Monitoring of AE is usually performed using a computer controlled AE facility which involves a transducer, preamplifier and variable gain amplifier with a bandpass filter. The AE transducer, which is commonly made from a piezoelectric ceramic such as lead zirconate titanate (PZT), is characterized by operating frequency, sensitivity, environmental characteristics (e.g. low and high temperatures, radioactivity). AE transducers can be categorized as resonant, multi-resonant or broadband types. For a study of plasticity in metals, broadband piezoelectric AE transducers with a flat response in a frequency band from 100 to 600 kHz are typically used. The AE transducer is placed on a surface of investigated material and the detection of AE is realized by the conversion of elastic waves (mechanical movement) into electrical signals. The preamplifier, which is placed close to the transducer in order to minimize interference, ensures high signal/noise ratio. Typically, large transducers are equipped with integrated preamplifiers. The amplifier can operate also as a band pass filter and signals having frequencies lower than 100 kHz are usually suppressed because they can be influenced by noise of various nature. Finally, amplified and filtered AE signals are evaluated using AE parameters. The evaluation of the AE signal is usually based on threshold-level detection and yields a comprehensive set of AE parameters. From the AE signal, amplitude, counts, events, rise time, duration, energy, count and event rate and frequency spectrum can be gathered.

A brief overview of the main AE parameters (following [20]) is given here:

- Event – is a single dynamic process releasing elastic energy
- (Peak) Amplitude – the maximum of the AE signal voltage within an individual event
- Counts – the number of pulses emitted by the measurement circuitry if the signal amplitude is greater than the threshold
- Rise time – the time interval between the first threshold crossing and the signal peak
- Duration – time difference between the first and the last threshold crossing
- AE event – AE event starts by crossing a defined threshold voltage level and the end of the event is detected when the signal remains below the threshold voltage for a period exceeding a Hit Definition Time (HDT). Afterwards, during a Hit Lockout Time (HLT), the AE signal is not parametrized in order to filter out sound reflections.
- AE count rate ($\Delta N_C/\Delta t$) – is the count number per time unit at a given threshold voltage level
- AE event rate ($\Delta N_E/\Delta t$) – is the event number per time unit at a given threshold voltage level
- AE energy – is elastic wave energy released by an AE event. Usually, the evaluation systems calculate the electrical energy as:

$$E = \frac{1}{R} \int_{t_s}^{t_e} U^2(t) dt \quad (5)$$

where R is the electrical resistance of the measuring setup, U is the measured voltage and t is the time (t_s, t_e – start and end time, respectively).

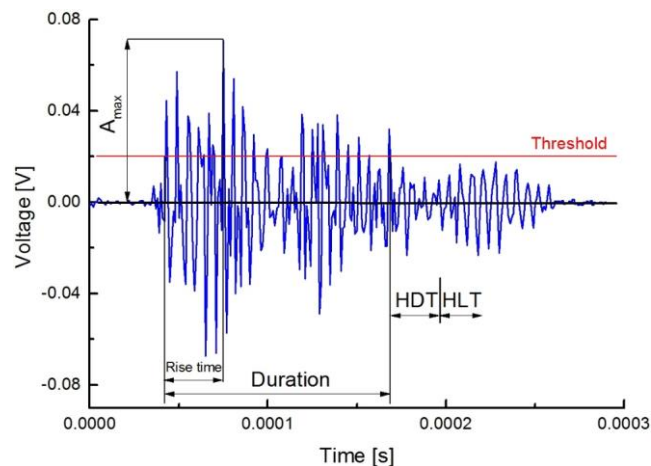


Fig. 1. Parametrization of the AE event

In general, two main forms of AE can be found: continuous and burst emission. The continuous emission (Fig. 2) consists of low amplitude AE generated simultaneously by a large number of sources and the AE signal has a random character. Typically, it is observed during plastic deformation of metallic materials as a result of collective dislocation movement. The burst emission (Fig. 3) occurs as a consequence of an instable fashion of plastic deformation or degradation of materials and is represented by high amplitude signal with a short duration.

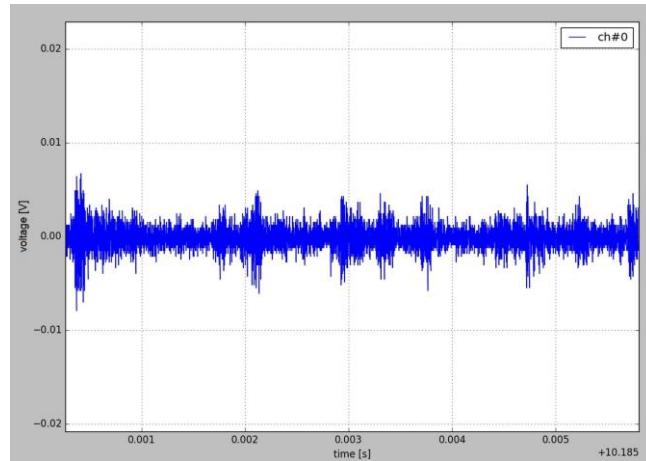


Fig. 2. Example of continuous emission

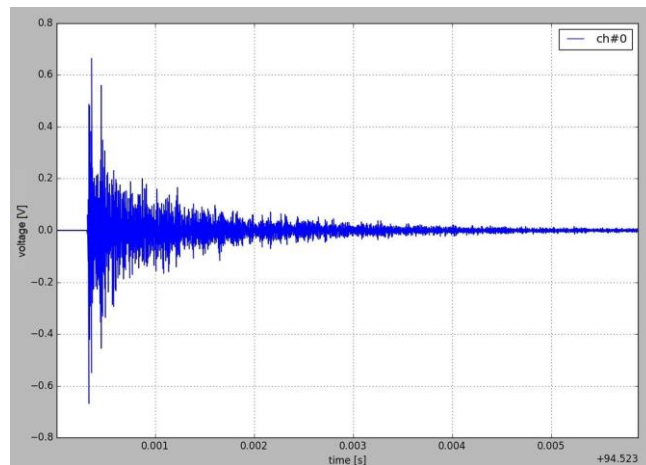


Fig. 3. Example of burst emission

Recently, a continuous storage of the AE data with a high sampling frequency enables additional *post-mortem* analyses of these data using advanced statistical methods. In general, the AE analyses are performed on either individual AE events separated by a threshold level detection or the “time window” AE data, in which a background noise is taken into account. Some advanced analyses of the AE signal are documented in our scientific papers which are presented in the following chapter.

2 Overview of the results

2.1 Critical character of plasticity from acoustic emission measurements

The plasticity phenomena in crystalline solids are linked with the multiplication and motion of crystal defects which results at the microscopic level in heterogeneous and discontinuous plastic flow. Averaging over local random fluctuations leads in most solids to homogeneous and continuous macroscopic plastic flow, even though the microscopic strain mechanism is discrete and discontinuous. In case of inhomogeneous plastic deformation, the serrated plastic flow at macroscopic level is always associated with plastic instabilities in metals as, for example, Portevin-Le Châtelier (PLC) effect [3].

The cooperative motion of a large group of crystal defects organizes into a scale-free pattern of dislocation avalanches which can be characterized by intermittency (crackling plasticity), power law distributions of avalanche sizes [21], aftershock triggering, fractal patterns as well as complex space-time coupling [22, 23]. The scale-free behaviour of dislocation avalanches is indicative of possible manifestation of self-organized criticality (SOC). The concept of SOC was introduced by P. Bak et al. [24] as a general framework for explaining the avalanche-like phenomena in spatially extended dynamical systems [25].

AE represents the collective processes occurring within the material during plastic deformation and therefore the SOC concept can be applied also to the AE data. The first evidence came from AE experiments on deformed ice single crystals that showed power law distributions of AE amplitude and energy [26].

The power law distribution of probability density $P(E)$ of the AE energy E is given by

$$P(E) \sim E^{-\tau_E} \quad (6)$$

where τ_E is the power law exponent and E is calculated according to Equation (5) for individual AE events.

Weiss et al. [26] have presented an approach in which the energy dissipated by viscoplastic deformation during individual AE events is assumed to correspond to the maximal amplitude of the events $E \sim A^2$ (Eq. (5)). Therefore, a power law distribution of avalanche amplitudes

$$P(A^2) \sim (A^2)^{-\tau_A} \quad (7)$$

is often used in the present form for a characterization of collective dislocation dynamics.

[A1] T. Richeton, P. Dobroň, F. Chmelík, J. Weiss, F. Louchet: *On the Critical Character of Plasticity in Metallic Single Crystals*, Materials Science and Engineering A – Structural Materials Properties Microstructure and Processing 424 (2006) 190 – 195. IF: 1.490

In this paper, the concept of self-organized criticality through a power-law distribution of probability density of the AE energy was applied on the AE data which were detected during tensile tests of hexagonal closed packed (hcp) single crystals. Specifically, Cd and Zn single crystals of various crystallographic orientations with respect to the loading axis were tested at a constant crosshead speed and at room temperature (RT) in order to activate specific slip systems and/or twinning. By using single crystals, the contribution of grain boundaries to plastic deformation was eliminated and the AE parameters were directly linked with deformation mechanisms.

In all our experiments, plastic deformation was accompanied with the AE activity exhibiting a strong intermittent character. We identified two basic sources of AE (dislocation slip avalanches and twinning), which correspond to two different types of AE waveforms. The AE signals recorded in the stage of easy basal glide exhibited low AE maximum amplitude and substantially smaller duration by comparison with the AE signals associated with twinning.

Furthermore, we separated AE events on the basis of a (somehow arbitrary) threshold on $A_0/E^{1/2}$ (A_0 – maximum amplitude) in order to distinguish twinning from dislocation slip. Events with low $A_0/E^{1/2}$ values were called T-type events and events with high $A_0/E^{1/2}$ values S-type events. Of course, the real proportion of twinning in the two different groups remained unknown. Nevertheless, this arbitrary distinction enabled us to verify that the probability distribution of AE event energies remains unchanged whether the analysis was made from a population of AE events exhibiting a supposedly small (S-type) or large (T-type) proportion of twinning.

A time clustering of AE events discovered that a T-type event can trigger an S-type event and vice-versa and so it highlights mutual interactions between twinning and slip. Avalanches of gliding dislocations induce stress rearrangements which may trigger twinning events and conversely, by changing locally the orientation of the crystal and consequently the local stress field, twinning may trigger dislocation glide avalanches.

Analyses of the AE energy distributions using the probability density function (PDF) of the AE event energies (Eq. 7) for twinning and dislocation slip were performed. Both deformation mechanisms show the same power law distribution for the PDF of their AE

energies with the exponent $\tau_E = 1.5 \pm 0.1$ which indicated no distinction between twinning and dislocation slip from this point of view.

Our results were compared with those found on hcp ice single- and polycrystals with an average grain size varying from 260 μm to 5 mm in creep test [27]. Compression creep tests (constant load) were carried out at 0.54 and 0.80 MPa at -10°C . One test was done at -3°C to be much closer to the melting point. The exponent of the power law distributions for all experiment was similar to those, which was observed for metallic single crystals deformed in tension at RT. These results strongly suggest that the general features of the observed intermittent flow regime are of general nature in plastic deformation of crystalline materials.

[A2] J. Weiss, T. Richeton, F. Louchet, F. Chmelík, P. Dobroň, D. Entemeyer, M. Lebyodkin, T. Lebedkina, C. Fressengeas, R.J. McDonald: *Evidence for Universal Intermittent Crystal Plasticity from Acoustic Emission and High-resolution Extensometry Experiments*, Physical Review B. 76 (2007) 224110. IF: 3.172

To support the concept of the universality of time-correlated intermittency and power law scaling in avalanche statistics, we decided to extend the AE observations of hcp single crystals described in [A1] to Cu single crystals with the face-centered cubic (fcc) unit cell and to use the high-resolution extensometry as a complementary method.

In hcp single crystals favourable oriented for the basal slip, a typical stage I characterized by a low-stress plateau was followed by hardening due to the activation of non-basal slip systems (stage II). The transition from stage I to stage II represented by a rapid increase in hardening coefficient ϑ ($\vartheta = d\sigma/d\varepsilon$) did not lead to different power law distributions of AE amplitudes ($\tau = 2.0 \pm 0.1$). However, plasticity in hcp metals is anisotropic and only small contribution of non-basal slip to overall plastic strain in stage II is expected. To study a contribution of multi-slip (a concurrent activity of several slip systems) on macroscopic strain without the presence of twinning, Cu single crystals were investigated. Again, the power law distribution of AE amplitudes exhibited the same exponent $\tau = 2.0 \pm 0.1$, which indicates the universality of power law scaling in avalanche statistics, irrespective of the level of strain hardening.

The high-resolution extensometry was used for a study of multislip in Cu single crystals. Several deformation tests in strain rate range from 5×10^{-5} to $5 \times 10^{-3} \text{ s}^{-1}$ were performed and the local displacements detected by extensometry were derived to obtain the

displacement velocities. The velocities exhibited a jerky character and PDF for their peak values was drawn. The peak velocity values were consistent with power law scaling of AE amplitudes and the same exponent $\tau = 2.0 \pm 0.1$ was observed.

Furthermore, the time correlation using a multifractal analysis of displacement velocities was performed. The observed wide range multifractal spectrum with a weak dependence on the applied strain rate indicated long-range time correlation between dislocation avalanches and the universality of the underlying dynamics.

We also studied an effect of the finite size on the development of the largest dislocation avalanches. Experimental results were explained by a motion of dislocation avalanches spreading over lamellar structures (occurrence of slip bands) in which a cut-off strain increment decreases with increasing sample size. The critical dynamics of intermittent plasticity is limited at large scales by the smallest sample dimensions as long as obstacles to dislocation motion (grain boundaries, precipitates, etc.) do not confine avalanche propagation to small volumes. This interpretation can explain why in polycrystalline materials the intermittency and scaling of strain are not present at macroscopic length scales.

[A3] M.A. Lebyodkin, I.V. Shashkov, T.A. Lebedkina, K. Mathis, P. Dobroň, F. Chmelík: *Role of superposition of dislocation avalanches in the statistics of acoustic emission during plastic deformation*, Physical Review E 88/4 (2013) 042402. IF: 2.326

In this study, the effect of the superposition of dislocation avalanches on the experimental determination of the underlying power-law statistics was investigated. In other words, we were interested in how the possible overlap of the analysed AE events can affect the results of the statistical analysis of the experimental data. A continuous storage of the AE data with a high sampling frequency enables *post-mortem* analyses of these data. By varying the event individualization parameters (U_0 – threshold voltage, HDT, HLT – more details in Chap. 1.2) a different set of AE events can be extracted from the same recorded signal.

The individualization of AE events was performed on MgZr (hcp structure) and AlMg (fcc structure) alloys. All deformation tests were performed in tension at RT. Three MgZr alloys with a various content of Zr (0.04 wt%, 0.15 wt%, and 0.35 wt%) and having the average grain size of 550, 360, and 170 μm respectively, were tested at a constant strain

rate. The AlMg alloy exhibiting a plastic spatial-temporal instability also known as PLC effect was studied at various constant strain rates.

All alloys showed a strong AE activity. In the MgZr alloys, AE was produced by a combination of dislocation slip and twinning. In the AlMg alloy, the PLC effect was the main source of AE. Afterwards, the sets of AE events were subjected to the power law analysis.

In the MgZr alloys, the variation of the threshold voltage U_0 had a very slight influence on the slope of the power law distribution. Increasing U_0 truncated low amplitude events (cut-off of the left-hand part of the power law scaling) and thus led to an impoverishment of the statistical sample. The influence of time parameters (HDT, HLT) on the power law distribution was even weaker than that of U_0 .

In the AlMg alloy, the application of the AE event parametrization was more complicated because of the PLC effect. This plastic instability is known to generate lasting AE events, most likely due to the merging of many hits because of successive triggering of many dislocation avalanches.

The obtained power law exponent was higher (2 - 3) by comparison to that observed in MgZr (1.5 - 2). The experiments on AlMg also made it possible to compare the data obtained in similar strain intervals and for the same choice of individualization parameters but for different strain rates. This comparison revealed some tendency to an increase in the power law exponent with decreasing strain rate. However, the avalanche overlapping does not only depend on the strain rate but it is a complex result of the action of various factors, including the noise level and the parameters used to extract avalanches. The robustness of the avalanche statistics regarding their overlap made it possible to confirm the hypothesis that the plastic deformation manifests a universal avalanche-type nature on the scale relevant to AE, reflected in the scale-invariant character of the distribution of AE amplitudes. Anyway, we have shown that the power-law exponents do not take on a universal value for all microscopic mechanisms of plastic flow. Moreover, they are sensitive to the microstructure, e.g., grain size, under condition of action of the same mechanism.

Statement of the author's contribution to the publications [A1-A3]

Based on my previous studies of the AE response of metals exhibiting a wide variety of deformation modes, I have significantly contributed to the proposals of all experiments and also to physical interpretations of the results. Particularly, to apply the SOC concept to the AE data [A1, A2], which were detected during plastic deformation of metallic single crystals. By that time, the SOC concept was used only for a study of dislocation slip avalanches in rocks or ice crystals. Therefore, the main idea was to study the critical character of plasticity in metals as a result of dislocation slip and mechanical twinning.

I performed all deformation and AE experiments, and most of the following AE data processing [A1, A2]. I contributed to both papers by discussions of AE results and I also took part in the manuscripts preparation.

I have participated in the study presented in [A3], which essentially consisted of the AE data streaming (in-situ) and the statistical analysis of the stored AE data (post-mortem). I performed deformation tests with concurrent AE measurements on the polycrystalline MgZr alloy and I also realized the AE signal processing. Furthermore, I contributed to physical interpretations of the results in the paper.

2.2 Plasticity and acoustic emission in novel magnesium alloys

Magnesium alloys, as prospective lightweight materials, are still intensively studied in order to achieve mechanical properties required for advanced applications. The mechanical behaviour of Mg alloys is quite different from conventional metallic materials with cubic structure (e.g. Al alloys) due to their hcp crystallographic lattice. It strongly depends on the c/a ratio of lattice parameters [28]. If the c/a is larger than the ideal $\sqrt{8/3}$ ratio, in which atoms are as close as possible to one another, the densest packing of atoms is on basal planes and plastic deformation is preferentially realized by dislocation glide in them along $\langle a \rangle$ direction (Fig. 4). If the ratio is smaller than the ideal one, the close-packed planes are prismatic and pyramidal ones, and the dislocation glide in them along the $\langle c+a \rangle$ direction will be favoured.

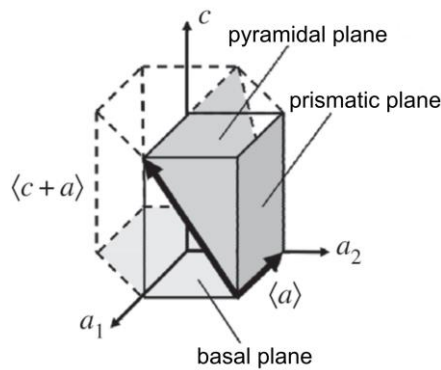


Fig. 4. Slip systems for the hcp lattice

Mg has the c/a ratio approximately equal to the ideal value and deformation behaviour is more complex than those described for both previous cases. According to the von Mises criterion [29], the compatibility of deformation in polycrystals requires at least five independent slip systems. However, in the hcp lattice of Mg, there are only two independent easy slip systems at room temperature (RT) on the basal (0001) plane ($\langle a \rangle$ direction), which is perpendicular to the c -axis. Therefore, basal slip does not produce any elongation or shortening parallel to the c -axis. To accommodate straining along the c -axis, either prismatic or pyramidal slip system ($\langle c+a \rangle$ direction) must be activated. Due to very high values of the critical resolved shear stress (CRSS) for their activation at RT, additional deformation mechanism called twinning must occur. Twinning is the dominant deformation mechanism in Mg alloys at RT and provides inelastic shape changes in the c -direction. It is an inhomogeneous process caused by local stress concentrations due to accumulated dislocations and depends on microstructure and a direction of the applied stress. In the tensile test of Mg single crystals along the c -axis, twins with $(10\bar{1}2)$

symmetry plane along $\langle 10\bar{1}0 \rangle$ direction are favoured. During compression test along the c-axis, twins with $(10\bar{1}1)$ symmetry plane along $\langle 10\bar{1}2 \rangle$ direction and with $(10\bar{1}3)$ symmetry plane can be activated [30].

The activation of individual deformation mechanisms is temperature dependent. CRSS for non-basal slip systems decreases with increasing temperature and above 220°C, it is so low that twinning is not necessary to be activated to fulfil the von Mises criterion [31].

Mechanical properties of wrought Mg alloys are significantly influenced, besides of hcp lattice, by a grain size, strong initial texture, solute segregation and precipitation [32]. The design of novel Mg alloys with advanced mechanical properties requires a thorough understanding of their role in plastic deformation. Therefore, in our studies, we have focused on the explanation of key issues using various *in-situ* and *post-mortem* methods. In particular, individual deformation mechanisms exhibit specific AE features and thus the active deformation mechanisms during loading of Mg alloys were monitored by the AE technique. Furthermore, the experimental results from deformation tests were applied in the theoretical model, which was based on evolution of dislocation densities. Thus, a combination of experimental and theoretical approach can be found in the following papers.

[B1] J. Bohlen, P. Dobroň, J. Swiostek, D. Letzig, F. Chmelík, P. Lukáč, K.U. Kainer: *On the Influence of the Grain Size and Solute Content on the AE Response of Magnesium Alloys Tested in Tension and Compression*, Materials Science and Engineering A – Structural Materials Properties Microstructure and Processing 462 (2007) 302 – 306. IF: 1.457

In this study, we focused our attention on the understanding of tension-compression asymmetry at the macroscopic yield strength (YS) and on a possible reduction of this undesirable mechanical behaviour.

Extruded bars of Mg-Al-Zn alloys were prepared by indirect and hydrostatic extrusion in order to reveal active deformation mechanisms during tension and compression of Mg alloys with various Al content and a different average grain size. A strong basal texture with grains having the basal planes preferentially oriented parallel to the extrusion direction (ED) as a result of extrusion process was taken into account.

Deformation tests were carried out in tension and compression at a constant strain rate of 10^{-3} s^{-1} at RT with a concurrent AE measurement. To easily compare the AE activity

in Mg alloys with various Al content, the AE results were discussed for Mg alloys with a similar average grain size. The AE count rate was detected at two threshold levels in order to distinguish low and high amplitude AE signals, which can be attributed to collective dislocation motion and twinning, respectively.

The grain size dependence of the activation of deformation mechanisms in the alloys was described according to the Hall–Petch law [33]. The dependence of the YS σ on the average grain size d is given as

$$\sigma = \sigma_0 + kd^{-1/2} \quad (8)$$

where σ_0 is a friction stress for dislocation movement. The slope k , called the “Hall–Petch strength coefficient”, depends on the orientation relation between the interacting grains as well as the critical shear stresses of the activated deformation mechanisms in both grains.

It was observed that the Hall–Petch slope k is more pronounced for compression than for tension and increases for both tension and compression with increasing content of aluminium.

For tensile test, the characteristic peak of AE at YS was explained by a massive dislocation multiplication. The following decrease of the AE count rates was ascribed to an increasing density of forest dislocations which reduces the flight distance and the free length of moving dislocations.

For compression test, a “broader” peak of AE at YS and a more rapid subsequent decrease in the AE activity was observed. This “broader” AE peak was explained by a twinning activity. Twin boundaries of newly created twins acted as non-dislocation obstacles for dislocation motion, and therefore they considerably reduced AE.

From our observations, it was concluded that the tension-compression asymmetry at YS is a geometrical effect of twinning. Owing to a strong basal texture, twinning can better contribute to the macroscopic strain in compression than in tension. The tension-compression asymmetry decreased with decreasing grain size. The grain size reduction leads to a decrease in the flight distance and the free length of moving dislocation. Corresponding to this, the AE count rates decreased. We attributed this to the fact that massive dislocation slip, as well as twinning, did occur with lower activity. A high elongation to fracture of 15 – 20% in tension and 10 – 15% in compression indicated that the deformation obeys the von Mises criterion (necessity to have at least 5 independent slip modes [30]). It was not possible to state which mechanisms were activated, but it can be assumed that non-basal slip, in general, has to be active. Grain boundary sliding for finer-

grained samples could also be considered. Both mechanisms will not contribute to the tensile-compression asymmetry and also not produce any additional AE.

Furthermore, the addition of Al led to a decrease in the tension-compression asymmetry. However, the influence of solute Al on the twinning activity as well as the influence of precipitates on the activity of twinning requires further investigations.

[B2] P. Dobroň, J. Bohlen, F. Chmelík, P. Lukáč, D. Letzig, K.U. Kainer: *Acoustic Emission during Stress Relaxation of Pure Magnesium and AZ Magnesium Alloys*, Materials Science and Engineering A – Structural Materials Properties Microstructure and Processing 462 (2007) 307 – 310. IF: 1.457

It was shown in [B1] that the tension-compression asymmetry at YS in wrought Mg alloys can be reduced by grain refinement and/or using Al as an alloying element in the Mg-Zn system. In those fine-grained Mg alloys, a microstructural stability under mechanical stress becomes an important point even at RT.

In this paper, we studied the microstructural stability of pure Mg and three Mg-Al-Zn alloys fabricated by the extrusion process using the so-called Kaiser effect in AE during repeated stress relaxations. The Kaiser effect is described as the absence of detectable AE at a fixed sensitivity level, until previously applied stress levels are exceeded [5]. The violation of the Kaiser effect indicates microstructure changes (such as recovery or recrystallization) during unloading or stress relaxations.

Tensile tests with and without stress relaxations were performed at RT and at a constant strain rate of 10^{-4} s^{-1} . Stress relaxations were realized so that the machine was stopped after each 2% of strain and the specimen was allowed to relax for 300 s. Concurrently, AE was monitored on the basis of two threshold level detection.

It was found that, besides a hardening effect due to increasing amount of alloying elements (mainly Al), a pronounced post-relaxation effect, manifested by a small yield point (a stress increment after reloading indicating ageing during relaxation) followed by a serrated flow curve (indicating the PLC effect, i.e. dynamic strain ageing), after each stress relaxation in the Mg-Al-Zn alloys appears. The post-relaxation effect increased with increasing amount of alloying elements and it was strain dependent.

The violation of the Kaiser effect was seen in pure Mg and in the Mg alloy with a low Al content, which demonstrates an unstable microstructure during stress relaxation. The AE activity was observed, even after the relaxation started. This indicates that plastic

deformation continues by recovery due to climb of dislocations, which can lead to re-opening of already closed dislocation sources, collective dislocation movement and very probably through occasional twinning during the stress relaxation. The effect of solute locking during stress relaxation seemed to be negligible. On the contrary, the Mg alloys with a higher Al content obey the Kaiser effect, which shows that the solute locking is efficient enough to stabilize the microstructure during the relaxation through diffusion of alloying elements to dislocations. The pronounced AE peak corresponding to the small yield point after the relaxation (missing in pure Mg) was explained by dislocation breakaway from solute atmospheres after reloading.

[B3] P. Dobroň, F. Chmelík, S. Yi, K. Parfenenko, D. Letzig, J. Bohlen: *Grain Size Effects on Deformation Twinning in an Extruded Magnesium Alloy Tested in Compression*, Scripta Materialia 65/5 (2011) 424 – 427. IF: 2.699

Even though wrought Mg alloys exhibit the fine-grained and stable microstructure, their deformation behaviour can be still insufficient for using the alloys in technical applications. The reason is an inhomogeneous microstructure after forming, which results in anisotropy of mechanical properties.

In this study, the influence of a microstructure with a broad grain size distribution on the occurrence of twins during compression was investigated using *in-situ* and *post-mortem* methods. The evolution of the microstructure during testing was first recorded using the AE technique. Tests were then repeated to successively greater strains along the stress–strain curve in order to observe changes in the microstructure during testing. Electron backscattering diffraction (EBSD) was used to characterize local microstructures and textures with special regard to the type and distribution of twins.

We documented in the paper that the AE measurement shows several onsets of dynamic energy release related to the activity of deformation mechanisms in an environment where a decrease in the AE count rates would be related to work hardening. The EBSD analysis confirmed that (10–12) extension twinning is the underlying mechanism and that twinning is favoured in the larger grains of the microstructure. As plastic deformation proceeds, it also occurs in smaller grains. At a later stage of the deformation experiment, the absence of AE confirmed that twin nucleation ceases and twin growth takes place.

[B4] P. Dobroň, J. Balík, F. Chmelík, K. Illková, J. Bohlen, D. Letzig, P. Lukáč: *A study of mechanical anisotropy of Mg-Zn-Rare earth alloy sheet*, Journal of Alloys and Compounds 588 (2014) 628 – 632. IF: 2.999

Besides the extrusion process, discussed in publications [B1-B3], Mg alloys are very often used in a form of rolled sheets. Mechanical properties (especially strength, ductility, and formability) of Mg alloy sheets are significantly influenced by a strong texture with the orientation of basal planes almost parallel to the sheet plane. The strong basal texture is responsible for tension-compression asymmetry and in-plane anisotropy of mechanical properties. Recent studies on the texture development in Mg–Zn alloys sheets have shown that rare earth (RE) elements additions can significantly reduce the final rolling texture, which results in changes of the deformation behaviour and mechanical properties. The final texture of magnesium alloy rolled sheets can also be affected by annealing and/or recrystallization.

It is generally accepted that the tensile in-plane deformation in Mg alloy sheets is initially realized by dislocation glide in basal planes which later must be accomplished by a contribution of dislocation glide in non-basal planes and/or twinning.

In this study, deformation mechanisms, which occur during tensile testing along rolling and transversal direction (RD and TD) of Mg-Zn-RE (ZE10) alloy sheet, were examined using AE and EBSD. The novelty consists also in the use of the strain hardening rate and the AE count rate *vs.* the flow stress plots for the sheets deformed in RD and TD to analyse the strain hardening behaviour.

The Mg alloy sheet displayed a relative weak texture with the orientation of basal planes tilted away from the sheet plane (normal direction, ND). The intensity of basal planes in the (0002) pole figure significantly varied with respect to RD and TD. The basal poles exhibited broader tilt and split towards TD rather than to RD, which is not usually observed in conventional magnesium alloy sheets.

A qualitative difference in the initial stages of the strain hardening rate *vs.* the flow stress plots for straining in RD and TD was found. In the early straining stages, the strain hardening rate first decreased rapidly then increased with increasing stress and subsequently, it again decreased with stress. This lift was less pronounced and narrower for the RD sample than for the TD one.

From concurrent AE measurements, we concluded that collective dislocation processes start at a lower flow stress in RD than in TD, which points out that the primary

(basal) slip mechanism can be easier activated in RD. Furthermore, the AE count rate remained practically constant during the wide lift for the TD sample, which indicated the presence of twinning. The EBSD analysis, performed on four samples which were deformed to different strains, confirmed higher twinning activity in the TD samples. With respect to facts that the twin nucleation is an excellent source of detectable AE activity [15] and the twin growth does not produce AE, contrary to the twin nucleation [18, 19] we also concluded that the wide lift for the TD sample is linked with the twin nucleation, whereas the narrow lift with much smaller twin fractions observed in the RD sample can be explained by the twin growth.

[B5] J. Balík, P. Dobroň, F. Chmelík, R. Kužel, D. Drozdenko, J. Bohlen, D. Letzig, P. Lukáč: *Modeling of the work hardening in magnesium alloy sheets*, International Journal of Plasticity 76 (2016) 166 –185. IF: 5.623

Based on results presented in **[B4]**, we have done in this paper a comprehensive study of deformation behaviour of two Mg alloy sheets with different chemical compositions and textures using theoretical and experimental methods. Microstructure and texture of the ZE10 sheet are described in **[B4]**. The Mg-Al-Zn (AZ31) sheet, in as received and stress released condition, exhibited homogeneous microstructure with broader tilt and split of basal poles towards RD rather than to TD (the opposite trend is in ZE10).

Special attention was paid to analysis in the so-called large strain region in which the stable secondary deformation regime has taken over following the primary basal glide. A strain hardening model, based on the generalized Kocks-Mecking approach [34], was fitted to the observed deformation behaviour, including the mechanical anisotropy of the Mg alloy sheets. The model enables the determination of the work hardening coefficient, considering the glide obstacles of both dislocation and non-dislocation origin; the distribution of grain orientations was also considered.

$$d\tau = \mathcal{G}(\tau_d, \dot{\gamma}, s) d\gamma = \left(\frac{A}{\tau_d} + B - C\tau_d \right) d\gamma \quad (9)$$

where $\tau = m\sigma$ is the resolved shear stress, $d\gamma = d\varepsilon/m$ is the slip increment within a grain, m is the Schmid factor,

$$\tau_d = \tau - \tau_y = m\sigma - \tau_y \quad (10)$$

τ_d is the glide resistance component due to the density of stored glide dislocations, and τ_y is the non-dislocation yield stress caused by, e.g., the Peierls-Nabarro stress, the Hall-Petch

contribution of grain boundaries, or solute and precipitation hardening. The parameter A is connected with the interaction of dislocations with the non-dislocation obstacles and it is expected to increase with increased solute content and/or the presence of precipitates. The parameter B relates to the work hardening due to the interaction of dislocations and the parameter C relates to recovery due to cross slip.

The onset of plastic activity within individual grains was conditioned not only by their Schmid factors but also by a level of "pre-deformation" (parameter u), i.e., a contribution to the resolved slip resistance stemming from the previous thermo-mechanical treatment (forming, annealing). Hence, the condition of plasticity was formulated by

$$m\sigma - \tau_y = \tau_d(\gamma) \geq u \quad (11)$$

where the immediate dislocation stress, τ_d , is raised over its starting value u via the work hardening and the slip γ actually elapsed by the grain. The maximum, saturated value $\tau_{d,s}$ cannot be exceeded in any grain, being given by the condition $\mathcal{G}(\tau_{d,s}) = 0$ in relation (9).

The basic work hardening model was primarily developed for materials with a cubic lattice structure, in which twinning does not occur. For our Mg alloys, twins are incorporated into the model as non-dislocation obstacles and the nucleation of twins is not taken into account. Therefore, the AE activity was monitored during the test and in the large strain region in which the model was applied only twin thickening was confirmed.

We supposed that the obstacles necessary to establish the common stage II hardening are not created ($B = 0$) for the present texture and prismatic slip conditions. The complete hardening is produced only by some fixed obstacles ($A > 0$). The apparent negative A detected in some investigated materials is caused by the twin thickening.

[B6] D. Drozdenko, J. Bohlen, S. Yi, P. Minárik, F. Chmelík, P. Dobroň: *Investigating a twinning–detwinning process in wrought Mg alloys by the acoustic emission technique*, Acta Materialia 110 (2016) 103–113. IF: 5.058

The tension-compression asymmetry in wrought Mg alloys with a strong basal texture was already discussed in **[B1]**. Nevertheless, during reverse loading of the wrought Mg alloys the so-called twinning–detwinning process can be observed. Compression loading leads to the 86.3° lattice reorientation caused by extension twins, detwinning could be activated in the twinned volumes during subsequent tensile loading. This process is characterized by a thickness reduction or disappearance of existing twin lamellae; that is, the twin lamellae are rotated back to the parent matrix orientation.

In this study, we analysed this process using the AE dataset in detail. Particularly, nucleation of various types of twins and the influence of twins produced by pre-straining on the subsequent hardening behaviour were investigated in the extruded ZE10 and AZ31 magnesium alloys. A bimodal microstructure of AZ31 and a fully recrystallized microstructure of ZE10 enable to study an influence of grain size on a twinning–detwinning process.

Following conclusions can be drawn:

It was shown that twin nucleation is grain size dependent and during reverse loading along the ED, detwinning is preferred at the expense of nucleation and growth of new twins. Furthermore, we found that twinning and detwinning, in terms of twin boundary mobility, are not grain size dependent.

During twin growth and detwinning, the AE response exhibited events with lower amplitudes than during twin nucleation. Therefore, neither thickening nor thinning of twins obviously contributed to the AE response.

New compression and extension twins appeared after completing the detwinning process at a later stage of plastic deformation. This was because higher stresses are required for their nucleation by comparison with the detwinning mechanism. Newly created twins contributed to strain accommodation in relation to the orientation of the parent grains. Moreover, during tensile loading, another type of extension twins nucleated by comparison with those that are active during pre-compression. The nucleation of twins at this stage of deformation resulted in an additional AE count rate peak.

[B7] K. Horváth, D. Drozdenko, K. Máthis, J. Bohlen, P. Dobroň: *Deformation behavior and acoustic emission response on uniaxial compression of extruded rectangular profile of Mg-Zn-Zr alloy*, Journal of Alloys and Compounds 680 (2016) 623 – 632.
IF: 3.014

The novelty of this paper lies in the determination of the dominant deformation mechanism in a given time period during compression of wrought Mg alloy using the adaptive sequential k-means (ASK) clustering of the raw AE signal.

The extruded Mg-Zn-Zr (ZK10) alloy in a form of the rectangular profile allows fabricating specimens in the three major directions: extrusion, transversal and normal (ED, TD, ND). Compression tests with concurrent AE measurements were carried out at RT with a constant strain rate of 10^{-3} s^{-1} . The deformation tests were repeated and stopped at specific stress values in order to follow a development of the texture during loading. The novel algorithm developed by

Pomponi and Vinogradov [35] was applied to the AE data and observed results were used for interpretation of a complex deformation behaviour in wrought Mg alloys.

The ZK10 alloy exhibited texture having basal planes oriented perpendicular to ND. A small angular spread of basal planes towards TD was observed.

The recorded waveform streaming data were sectioned into consecutive frames (“time windows”). After the Fast Fourier Transformation (FFT) of the signal, the Power spectral density (PSD) function was calculated for each frame. The features of the PSD in the first frame define Cluster 1. PSDs in the consecutive frames are analysed one-by-one. If the statistical properties of a given PSD are similar to those in an already existing cluster, this PSD is assigned to this cluster. If not, a new cluster is established. The conditions for new cluster forming are based on *k*-means method and clustering is realized in the 7-dimensional space.

When the clustering procedure was completed, a dominant AE source mechanism was assigned to each cluster. This assignment consists of three basic steps:

1. Checking the time of the appearance of the elements in a given cluster. For example, since the recording of AE data is always launched *before* the starting of the deformation test, the elements in Cluster 1 naturally belong to the background noise.
2. Checking characteristic features of the PSDs, as energy, frequency distribution etc. For example, the twin nucleation usually emits AE signal with larger energy than that produced by a collective dislocation motion.
3. Comparison of the results with the supplementary data, as texture measurement, microscopy, theoretical modelling etc.

It was revealed that twinning is preferential in the ED and TD compression. This results in a lower number of twins which have a higher impact as revealed from the energy of the respective cluster as well as the more pronounced work hardening. In the case of the ND compression twinning plays a minor role. It is also not preferentially active as seen from the ASK analysis as well as from the gradual change of the texture.

Statement of the author's contribution to the publications [B1-B7]

I have proposed the concept of all experiments and I also significantly contributed to physical interpretations of the results presented in all seven publications.

I performed all deformation tests and AE measurements with the following data processing [B1-B5] or I guided my Ph.D. students through these experiments and AE analyses [B6-B7]. In [B1], I took part in manuscript preparation, especially in discussion of the AE results with respect to microstructure. I wrote the papers [B2-B4] with a contribution of my colleagues, which consisted of X-ray and EBSD measurements and fruitful discussions of the results. In [B5], I also contributed to the discussion of the experimental data and I wrote a part of the paper. I revised the papers [B6, B7] of my Ph.D. students.

3 Conclusions and future perspectives

Publications [A1–A3] have significantly contributed to the understanding of dislocation dynamics in single- and polycrystalline materials using statistical analysis of the AE data. As far as I know, we were the first who applied the concept of self-organized criticality through a power-law distribution of the AE energy on metals. We studied a power-law distribution in a large amount of metals with respect to their crystal structure, chemical composition, and grain size. The loading mode (tension, compression, creep) and strain rate were also taken into account. In all experiments, it was observed a power-law distribution of the AE energy with the exponent of 1.5 – 2.0. These results strongly supported the idea that the general features of the intermittent flow regime are of general nature in plastic deformation of crystalline materials.

In [A3], a unique concept of continuous storage of the sampled AE signal with additional *post-mortem* statistical analyses of the AE data was presented. The uniqueness of the work lies in a study how the AE event individualization parameters influence the results of the statistical analysis of the experimental data. Thus, these data, until then unavailable, clarify the role of the setup of the threshold voltage and time parameters in power-law statistics.

Nowadays, investigations of effects related to plastic deformation at micrometre scale (size effect) are highly attractive and therefore I have been involved in a study of the size effect in thin Al wires, Al foams, and micro-pillars by using the AE technique.

The study of physical processes in the Mg alloys presented in [B1-B7] significantly contributes to knowledge of their deformation behaviour. Several tasks related to the microstructure evolution and resulting mechanical properties of novel Mg alloys with enhanced properties were solved.

In [B1], the tension-compression asymmetry at the macroscopic yield strength in extruded Mg alloys with a various content of Al was documented. We proposed the reduction of this undesirable asymmetry in mechanical behaviour using Mg alloys with a fine-grained microstructure. Grain refinement can lead to instability of microstructure under stress. It was documented in [B2] that higher content of Al in alloy stabilizes the microstructure in extruded Mg-Al-Zn alloys. Using AE and EBSD analyses presented in [B3], we, as the world first, proved experimentally the dependence of twinning activity on the grain size in wrought Mg alloys. In [B4, B5], a comprehensive study of deformation behaviour of Mg sheets with different textures can be found. Experimental results were also applied in the strain hardening model. The fundamental result lies in that prismatic slip with a double slip limit is preferred over the other potential slip systems (basal, pyramidal) and tensile twinning in the range of large strains.

My recent scientific activities in this field of research are focused on a study of the twinning-detwinning process and the impact of solute segregation and precipitation along dislocations and grain boundaries on twinning. The interaction between twin nucleation and twin propagation as well as dislocation glide on one hand and solute or precipitates, on the other hand, are key issues for a knowledge-based development of materials with improved properties. To achieve these goals, new algorithms for AE signal processing were used. The first results are presented in papers [B6, B7] which were written by my Ph.D. students.

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4 List of publications

- [A1] T. Richeton, **P. Dobroň**, F. Chmelík, J. Weiss, F. Louchet: *On the Critical Character of Plasticity in Metallic Single Crystals*, Materials Science and Engineering A – Structural Materials Properties Microstructure and Processing 424 (2006) 190 – 195. IF: 1.490 DOI: 10.1016/j.msea.2006.03.072
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5 Reprints of the selected papers