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Experimental investigation of dislocation avalanches

thesis booklet

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Background

Plastic deformation of crystalline materials is in most cases mediated by dislocations. Dislocations are line-like crystallographic defects formed by a sequence of atoms having an atomic neighborhood incompatible with the lattice structure. The movement of these lines leads to the local plastic deformation of the material in the order of the lattice parameter ($0.3 - 0.5$ nm).

The deformation properties on the micron and submicron scale are fundamentally different from those of bulk materials. In this size regime, plastic deformation is characterized by large spatial and temporal fluctuations (dislocation avalanches). Since the dislocations produce significant long-range stresses $\sigma \propto r^{-1}$, their cooperative motion is complex and complicated. Such avalanche-like behavior of dislocations is still widely studied today, and their deeper knowledge is essential in understanding the plastic behaviour of materials. It is known that the sizes of the strain bursts generated by dislocation avalanches follow a power-law distribution. As such, these events occur on practically every size scale.

A special process of plastic deformation of crystalline materials is twinning. In this process, a material subjected to external load realizes the deformation by changing the orientation of its unit cell. There is some particular misorientation between the parent and twin crystal, which could be created by compression or tension twinning. In these cases, the global orientation change occurs via local, sub-nanometer displacements of individual atoms. The process of twinning occurs by momentary coordinated change of the atoms in a given unit of volume. Nowadays, many open questions about the dynamics of this deformation are still to be answered.

Objective of the thesis

My thesis deals with the experimental investigation of the micron-scale deformation of metals. It was previously shown that the deformation consists of strain bursts well-separated both in space and time, however, this was only true when certain conditions were met. For example, it became detectable in case of appropriate dissolved atom concentration and temperature, as well as on given sample size and experimental setup. The experiments of my research intended to answer the question, how and for what reason the plastic instability of metals occurs and what their dynamics is. New results in this field can provide useful knowledge both in theoretical and applied research, as it also broadens our understanding of the properties of micro-electromechanical systems (MEMS) used in large quantities today in quotidian electronic devices.

Experimental methods

- **Acoustic Emission** (AE) is generally defined as elastic energy spontaneously released during local, dynamic and irreversible changes of the (micro)structure of the materials. Near the AE source, the released energy forms a stress pulse, which propagates through the material as transient elastic waves. The wave component perpendicular to the surface may be detected by piezoelectric transducers, which are coupled to the specimen surface mechanically. The electrical signal from the transducer is first preamplified and then led to the input of the measuring system. Obviously, the waveform of the AE signal is significantly affected by the source mechanism, propagation through the specimen volume and detection by the transducer.
- In a **Scanning Electron Microscope** (SEM), the generated electron beams are focused on the sample surface using magnetic lenses. The surface of the sample can be imaged by collecting information from the electron-matter interaction and scanning it from point to point. The secondary electrons commonly used for imaging originate from the sample. They come from a depth of $1-10$ nm in the sample, that is, SEM is assumed to provide a surface-type of information.
- An SEM, in addition to the electron beam, can be also equipped with an ion beam. In our SEM laboratory one has the possibility to accelerate Ga^+ ions to 30 keV and to focus them on the surface of the sample in a similar way as the electron beam. In the technology of **Focused Ion Beam** (FIB), the possible current values are in the range of 1.5 pA $-$ 65 nA. Imaging can be performed using secondary ions and electrons generated by the ion beam. By increasing

the current of Ga^+ the high-impulse ion beam pulverizes the surface atom by atom, so the sample can be machined on the submicron scale to nm precision.

- As a specific type of electron-matter interaction, the **Electron Backscatter Diffraction** (EBSD) can also be investigated. In this case the focused electron beam penetrates into the sample and inside the sample the electrons scatter in all directions. The scattering intensity depends on the direction. The electrons are scattered inelastically and then interact with an atomic layer, generating a diffraction pattern according to Bragg's law. The shape of these diffraction patterns can be used for the identification of the crystal structure and its orientation.
- The image of a **Transmission Electron Microscope** (TEM) is created based on the interaction between the electrons generated and controlled by the device and the sample. The main difference between SEM and TEM is that in the latter the electron beam is collimated. The beam passes through a thin sample then the elastically scattered electrons generate the diffraction pattern. With the help of TEM, even atomic resolution can be achieved, and one has the possibility to examine the dislocations individually.
- The perfectly ordered single crystals would give **X-ray diffraction** Bragg peaks that are 'infinitely sharp' delta functions. Any kind of disorder in the crystal lattice, however, produce a broadening of the diffraction peaks. From the detailed analysis of the shape of the peak, the properties of the deformation field can be determined. The effects of dislocations and other types of lattice defects can be distinguished from the line profiles. The analysis of the second order restricted moments of the diffraction was used to determine the dislocation densities with an accuracy of a few percent.

Main results

- **T1: I developed a novel experimental method for the detection and quantification of dislocation avalanches in micropillars.**

The experiments presented in my thesis are based on a self-developed in situ nanoindenter. The nanoindenter, which can also be used in SEM, collects data from three independent sources simultaneously during the deformation of a micropillar. (i) A capacitive displacement sensor measures the elongation of a spring. From this, one gets real-time information about the acting stresses in the pillar and the exact position of the indenter tip, which shows the stress-strain conditions. A further advantage of this spring technology is that the adjustable spring constant allows tuning the response speed of the device. (ii) The attached (and replaceable) piezoelectric acoustic sensor detects the elastic energy released by plastic events. Until nowadays, it has only been possible to investigate these events at the macroscopic level. (iii) The simultaneously created SEM images are also essential for a clear explanation of the deformation mechanism. The establishment of this measurement system has been successful despite the fact that highly localized dislocation events emit extremely weak AE signals due to the relatively small number of moving dislocations.

It has become clear that this device is able to detect extremely weak AE signals generated during micropillar deformation. As part of my research, I extended the capabilities of the device to be able to mount different AE sensors and to collect and analyse AE data. Furthermore, I made it possible to use the stage inside different devices (such as a glove box) as well.

- **T2: I found that the deformation energies associated with dislocation avalanches exhibit a linear connection with the energies of the AE signals, and the statistical properties of the AE signals are analogous to the behaviour observed for earthquakes.**

Based on compression tests on Zn micropillars, strong experimental evidence was obtained that the sources of the detected AE signals were indeed the dislocation avalanches. The cooperative avalanche-like motion of dislocations is indicated by the sudden load drop. Typically, multiple AE events were detected during such a drop. By adding the energies of such AE signals during a given load drop, I found that the released acoustic energy shows an excellent correlation with the energy associated with the load drop. (Fig. 1).

The importance of this correlation is evident if one takes into account, that different response speeds belong to different measurement methods. The time resolution of the AE is at least four orders of magnitude higher than that of a common in situ nanoindentation system. This makes the AE highly suitable for dynamic investigations.

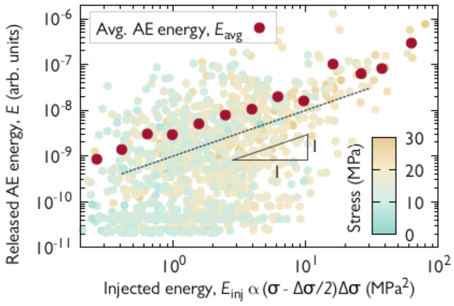

Figure 1: On the x-axis the injected energies of the stress drops are presented whereas the y-axis shows the sum of the energies of those AE events that were detected during the given stress drop.

The most surprising outcome of the experiments is that this process, despite the fundamental differences between deformation mechanisms of metals and that of tectonic plates, was found to be completely analogous to earthquakes.

- **T3: I have shown that ion irradiation of a single crystal reduces the energy dissipated by dislocation avalanches in accordance with the observed localization of the deformation.**

The effect of proton irradiation was investigated by single slip oriented Zn micropillar compressions. The ion irradiation induced evenly distributed dislocation loops with an average diameter of ∼100 nm. Such lattice defects have been found to act as a strong barriers against dislocation movement.

The effect of dislocation loops on the deformation process was identified. Our results confirmed the channeling effect. The ion-induced dislocation loops contributed to the formation of deformation channels in which dislocations can move unhindered compared to the near regions. Furthermore, the distribution of AE signals was found to be insensitive to the presence of channels - dislocation loops - however, the number of detected signals showed a significant dependence. It is likely that the dislocation loops are able to immobilize a given proportion of the mobile dislocations, which explains the reduced amount of

AE signals. Since the energy values of the emitted AE were independent of the existence of deformation channels, I assume that the energies of AE signals are not determined by the number of mobile dislocations, in contrast to the number of AE signals.

- **T4: Based on the experiments performed on pure aluminum and its Mg alloy, I found that with the help of the concurrent application of AE measurements and micromechanical tests, the plastic instabilities of different origins can be distinguished at the micron scale.**

  Three different deformation mechanisms were investigated on cubic single crystals. The presence of small amounts of Mg alloying atoms decisively influenced the deformation mechanism. From the measurements of Al3Mg showing the Portevin-Le Chatelier (PLC) effect, I concluded that the mechanism of the deformation is not affected by the sample size, nor by the number of mobile dislocations. In the case of a larger micro-sample, due to the higher number of dislocations involved in the deformation, dislocation avalanches with energies exceeding the AE detectability limit are more likely to occur. With this measurement, AE data directly provided information on micron-scale dislocation processes.

  Without dissolved atoms, the length scale determining the deformation (the distances characterizing the dislocation-dislocation interaction) was larger than the examined sample sizes, so the deformation properties were also influenced by the sample size. At this time, regardless of orientation, the number of detectable AE events surprisingly increased for the smaller micropillars. Furthermore, I received a large number of AE events in double-slip oriented cases contrary to the case of multiple-slip orientation. These data support that the larger sample size and the higher number of activated slip systems provide a greater number of possibilities for dislocation reactions that can reduce the energy emitted by the dislocation avalanches below the detection limit.

- **T5: The process of twinning was investigated by micropillar compression experiments, which allowed the observation and measurement of different time scales related to the dynamics of twinning.**

Hexagonal close packed materials can deform frequently by twinning. In my studies, the twinning of pure Mg single crystal was investigated. Three different time scales were successfully associated to twinning. At the onset of deformation, the instantaneous crystallite formation was followed by a rapid lateral grain growth, then a slow vertical grain boundary propagation took place. We were the first to measure the velocity of lateral grain growth.

My other new results belong to the slow process of dynamics for micropillar twinning, which describes the vertical growth of the twin grain. The experiments unanimously showed that under the testing conditions, the twin crystallite does not grow further after reaching a given size, instead, a new twin is formed, and then at the end of the deformation, at high stresses, the crystallites merge together.

- **T6: Based on the AE measurements of microsamples with different structures and compositions, I found that the most important parameter in the statistics of AE signals is primarily determined by the nature of the dominant deformation mechanism.**

The values of the statistical parameters of dislocation avalanches are currently under discussion. Accurate measurement of the statistical values is complicated due to several aspects. On the one hand, it is known that these values are sensitive to almost every parameter of the experimental setup, such as temperature, strain rate, control of the deformation, and precondition of the sample. Based on my work, AE signals are highly suitable for the characterization of dislocation avalanches. Therefore, instead of the conventional stress-strain analysis, the AE data can be used to characterize them.

The diagram of the summarized AE data from my PhD research (Figure 2.) contains the energy distributions of AE events belonging to different deformation mechanisms. The different colours refer to different deformation mechanisms. Data points with the same colours, however, do not take into account the differences in the indentation rate, pillar sizes, and geometry, since these parameters were found not to have significant effects on the AE energy distributions. In the following discussion, I compare data from different experiments. The comparison is valid because the data from different experiments were streamed by the same setup and under identical conditions, such as the indentation control mode, device stiffness, temperature, and the lateral flexibility and hardness of the punch as well as the distance between the AE source and detector.

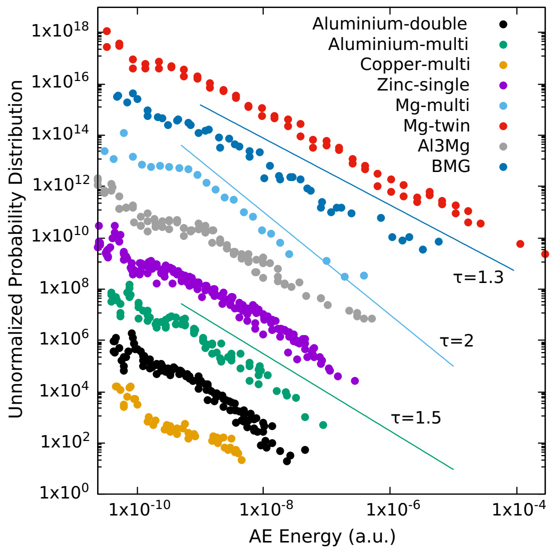

Figure 2: Shifted probability distribution densities of the AE energies, which detected during different (color marked) deformation mechanisms

A common aspect of the studied AE energy distributions is their scale-free nature as well as their deviation from the power law behavior at low energies. The universality of the low-energy deviation was expected in our case because the sensitivity of the AE equipment used during the different measurements was the same. This behaviour expresses the sensitivity of AE detection. Furthermore, no sign of high-energy cut-off was found, although I found four orders of magnitude difference between the largest events detected.

No significant differences were found in the exponents of the AE distributions between cases where deformation occurs via single (Zinc-single) or multiple dislocation slip (Aluminum-double, Aluminum-multi, Copper-multi, Al3Mg). In such cases, where the deformation takes place by dislocation avalanches, the power law's exponent was near $\tau \approx 1.5$. However, when the deformation mechanisms were mixed – microtwinning and dislocation slip – (Mg-multi), the value of the exponent rised to $\tau \approx 2$. Furthermore, when the deformation process was due to the twinning alone (Mg-twin), or the deformation mechanism was completely different as in metallic glasses (BMG), (formation and propagation of shear bands) the exponent became $\tau \approx 1.3$.

In summary, the value of the power law exponent τ of the AE distribution can be concluded not to be directly affected by the material's form. According to my data, the primary influence on the exponent was exerted by the type of the main deformation mechanism. The exponent was found to be the same in cases where the deformation took place by dislocation slip, regardless of how the main interactions between dislocations occurred.

- **T7: Based on the AE measurements of microsamples with different structures and compositions performed at identical experimental conditions, I found that all mechanisms examined that impede dislocation movement reduce the number of detectable AE signals.**

Additional experiments were performed on single crystal and nano grained polycrystalline Al30Zn micropillars. According to our results, in the nanocrystalline case where the deformation occurred by grain boundary sliding mechanism, no AE events were detected. However, in the single-crystal case, where the deformation was caused by an avalanche-like motion of the dislocations, several AE events were found. According to our results, which have been substantiated by several experiments, the origin of the AE signals triggered by the dislocation avalanche is the elastic energy release caused by the dislocation avalanche. Here I managed to extend this result. If we consider the energy raised by the deformation unit (e.g., dislocation motion, grain boundary slip, shear band propagation, etc.) as the source of the AE event, we obtained a theory that can be confirmed by our experiments. Based on this, since the process of grain boundary sliding is slow, uncoordinated, and operated on the short range, no acoustic activity is expected from this deformation mode.

The amount of the detected AE signals in our experiments not only supports the traditional characterization methods but also amends and modifies them. The number of AE signals induced by dislocation avalanches also strongly depends on the deforming volume, the number of activated slip systems, and the quality and quantity of the lattice defects. This strong relationship may also mean that there is still no clear and absolute definition for acoustically active materials and structures.

The higher the number of slip systems, the lower the number of dislocation events that generate detectable AE events. In the presence of solute atoms, I found that the number of detectable AE signals dropped by about half. This can be explained by the effect of the increased amount of obstacles on the energy released during the dislocation avalanches. The effect of reducing the amount of AE of the extra lattice defects was observed in the case of zinc and aluminum single crystals as well. These lattice defects have differences in both quality and quantity, so they reduce the AE rate to varying degrees. In the case of Zn, the number of AE was reduced to a quarter because of the dislocation loops. The greater reduction is due to the diffusivity of the dissolved atoms, causing the dynamic interaction between dislocations and dissolved atoms.

The number of the AE events generated by dislocations is also affected by the number of active slip systems involved in deformation. In the case of multiple slips, due to the possibility of crossing each other's path of the moving dislocations, and the additional dislocation reactions that occur, I expect a reduction in the mean free path of the dislocations. This could be responsible for the observed decrease in the AE rate.

The author's selected publications

[A1] Péter Dusán Ispánovity, **Dávid Ugi**, Gábor Péterffy, Michal Knapek, Szilvia Kalácska, Dániel Tüzes, Zoltán Dankházi, Kristián Máthis, František Chmelík and István Groma. *Dislocation avalanches are like earthquakes on the micron scale.* Nature Communications 13.1 (2022), 1975. [1]

[A2] Kristián Máthis, Michal Knapek, Filip Šiška, Petr Harcuba, **Dávid Ugi**, Péter Dusán Ispánovity, István Groma és Kwang Seon Shin *On the dynamics of twinning in magnesium micropillars.* Materials & Design 203 (2021), 109563.

[1] with shared first authorship