Summary

Dislocations (line defects) and grain boundaries (planar defects) are two types of lattice defects that are crucial to the deformation behavior of metals. Permanent deformation of a crystalline material is microscopically associated with the nucleation and propagation of dislocations, and extensive knowledge of these processes is therefore required to understand and control the mechanical performance. Metals and alloys are most commonly used in their polycrystalline form, i.e. they consist of grains separated by grain boundaries. These grain boundaries, too, greatly affect the mechanical properties as they can impede the movement of dislocations. The actual interaction between dislocations and grain boundaries is a complex phenomenon that may be exposed through various mechanisms. This thesis concentrates on these interaction mechanisms and their effect on the microscopic and macroscopic mechanical properties of the material. The main focus lies on aluminum magnesium alloys, in which particularly the effect of magnesium on the abovementioned mechanisms is studied. Transmission electron microscopy and nanoindentation have been extensively used throughout the experimental work.

Incipient plasticity at grain boundaries

Nanoindentation can be used to introduce deformation, and thereby dislocations, near grain boundaries on a submicrometer scale, while the resulting mechanical response is continuously recorded. Following this principle we have conducted a series of experiments to investigate the incipient plastic behavior of grain boundaries in bicrystals. The advantage of bicrystalline specimens is that the grain boundary parameters are constant and unambiguously defined. Body-centered cubic (bcc) metals were chosen because of their intrinsically high resistance to dislocation transfer, which considerably facilitates the measurement of dislocation – grain boundary interaction phenomena. The response to indentation has been measured as a function of the distance to the boundary in a Mo bicrystal with a coherent Σ3 boundary, a Mo bicrystal with a Σ11 boundary and an Fe-Si bicrystal with a general high-angle grain boundary.
In the Mo bicrystals, the presence of the grain boundary leads to a detectable decrease of the load required to initiate macroscopic plastic deformation. Crystalline specimens in general can be loaded elastically until the critical shear stress for dislocation nucleation is reached under the indenter. This point is characterized by a plateau in the load-displacement curve. The nucleation of dislocations from the $\Sigma 3$ boundary becomes significant when the indentation is made within approximately 300 nm from the boundary. From this interaction range, the critical shear stress for dislocation nucleation at the boundary is estimated to be 1.8 GPa.

Besides the plateau associated with initial plastic deformation, the indentation curves for the Fe-Si bicrystal show another characteristic plateau, the presence of which is strongly correlated to the proximity of the grain boundary. From energetic considerations it is suggested that this plateau is due to a dislocation-based mechanism, in particular dislocation transmission across the boundary. Dislocations pile up between the indenter and the grain boundary until the shear stress at the front of the pile-up becomes large enough to initiate emission of dislocations in the adjacent grain. The length of the pile-up can be estimated from the experimental data; this value can subsequently be used to obtain a quantitative measure for the grain boundary’s resistance to dislocation transmission. The investigated indentation method thus provides the unique opportunity to locally measure the strength of individual grain boundaries.

**Plasticity in ultrafine-grained Al-Mg alloys**

Observation of indentation-induced deformation is commonly performed post mortem by atomic force microscopy (AFM) or transmission electron microscopy (TEM). This indirect approach entails several disadvantages: it is not possible to monitor time-dependent deformation processes during deformation; furthermore, the microstructure observed after indentation may differ substantially from that during indentation due to relaxation processes. The recently developed technique of in situ nanoindentation in a TEM allows for direct observation of indentation-induced dynamical processes and consequently does not suffer from the abovementioned limitations. Using this technique the deformation behavior of Al and Al-Mg thin films with a grain size of several hundreds of nanometers has been studied.

The movement of dislocations through the Al-Mg films proceeds in a jerky fashion due to their interaction with solute Mg atoms (solute drag). The
observed jump distance in an Al-2.6%Mg film is of the order of 50 nm. This value compares well to the theoretical average distance between obstacles in the presence of diffuse interaction. For load-controlled indentation, the solute drag significantly influences the load-displacement curve. In pure Al, several plateaus are observed which are attributed to dislocation nucleation and propagation. These plateaus are significantly smaller or even absent in the Al-Mg alloy films, where dislocation propagation is hindered by the interaction with solutes.

During indentation of the ultrafine-grained Al film, extensive movement of both low- and high-angle grain boundaries is observed. The occurrence of this deformation mechanism, which under uniform stress conditions is restricted to nanocrystalline materials, is attributed to the high stress gradients involved in sharp indentation. In contrast to the observations in pure Al, no such movement of high-angle grain boundaries is found in any of the Al-Mg alloy films. Since this apparent pinning effect is observed for Mg concentrations both below and above the solubility limit in Al at room temperature, it is considered to be due to solute Mg. Presumably the presence of Mg atoms alters the atomic structure of the grain boundary, which in turn changes its local stress field. Low-angle grain boundaries are not susceptible to this pinning effect, since they can be regarded as a periodic arrangement of dislocations with negligible mutual interaction. The stress required to move a low-angle boundary is therefore much lower than for a high-angle boundary.

With grain sizes decreasing below several hundreds of nanometers, the deformation of metals is increasingly accommodated by grain boundaries rather than conventional dislocation mechanisms. The observation that grain boundaries may be effectively pinned by solute Mg is therefore very interesting from the perspective of the development of ultrafine-grained and nanocrystalline aluminum alloys.

**Superplasticity in coarse-grained Al-Mg alloys**

The final chapter of the thesis concentrates on deformation of Al-Mg alloys with a substantially larger grain size of about 70 µm. Under specific conditions of strain rate and high temperature, these alloys show superplastic behavior. Superplastic materials can be deformed to very high elongations prior to failure, usually in excess of a few hundred percent, at a relatively low flow stress. This characteristic property makes them very attractive for the production of components with a large freedom of design. The hallmark of superplastic
deformation is a high strain-rate sensitivity of the flow stress, which suppresses necking during deformation and consequently allows high elongations to be reached.

In coarse-grained Al-Mg alloys, superplastic deformation is accomplished by viscous glide of dislocations. In this mechanism, the interaction between dislocations and solute Mg atoms accounts for the high strain-rate sensitivity. The viscous glide is accompanied by dynamic reconstruction of the microstructure, the appearance of which depends on the deformation parameters and can be both detrimental and beneficial to the ductility. These reconstruction mechanisms have been investigated by analyzing the microstructure of the alloys as a function of tensile strain at different strain rates and temperatures.

Dynamic recrystallization is dominant at strain rates in excess of $10^1 \text{s}^{-1}$ and results in rapid coarsening of the microstructure and premature failure. The optimum strain rate for superplasticity of the coarse-grained Al-Mg alloys lies around $10^2 \text{s}^{-1}$; in this regime, dynamic recovery prevails, leading to values of the maximum elongation in excess of 300%. During dynamic recovery, grain refinement occurs by the formation of subgrain boundaries and low-angle grain boundaries. TEM observations show that subgrain formation proceeds slowly, presumably due to the low stacking fault energy in Al-Mg compared to pure Al. During initial straining, subgrains are formed primarily along the original grain boundaries. A uniform substructure is established at a strain of the order of 1.

With a high optimum strain rate of approximately $10^2 \text{s}^{-1}$, the coarse-grained Al-Mg alloys are an attractive alternative to conventional fine-grained superplastic Al-Mg alloys. Superplastic deformation of these fine-grained alloys is attributed to grain boundary sliding, which generally requires low strain rates of $10^{-4}$ to $10^{-3} \text{s}^{-1}$ in order to be accommodated by diffusion processes. An additional advantage of superplasticity based on viscous glide is that this mechanism has virtually no grain size dependence and therefore the preparation of such materials is less complex.

In summary, the research has provided new insights into the interaction between dislocations and grain boundaries on various length scales, in which specifically the effect of Mg in Al-Mg alloys on these interaction mechanisms has been clarified.