Interactions between Dislocations and Grain Boundaries
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1.1 Lattice defects in metals

Although metals have been used for structural applications since the copper and bronze ages, the physical mechanisms governing their mechanical properties remained unknown until the end of the nineteenth century. Experimental observations at that time revealed that permanent deformation of metals proceeds by the formation of slip bands, in which part of the material is sheared with respect to the other part [1]. After the X-ray experiments by Laue in 1909, it was inferred that such deformation behavior could be explained by shearing of the crystal on rational crystallographic planes. The theoretical shear stress required to initiate plastic deformation in this way was calculated by Frenkel [2] to be approximately one-fifth of the elastic shear modulus. However, experimental values for the shear strength of metals were found to be several orders of magnitude smaller. The justification of this discrepancy was one of the major challenges in materials science in the beginning of 1930s.

In 1934, it was realized independently by Orowan [3], Polanyi [4] and Taylor [5] that the disagreement between the theoretical and experimental values of the shear stress can be explained by the concept of a dislocation, or line defect, in the crystal. Shearing of a crystal containing dislocations may occur by movement of the dislocations along lattice planes. This process involves merely the displacement of atoms close to the dislocation core, rather than the displacement of an entire lattice plane, and therefore requires a much lower shear stress. Since the 1950s, numerous observations of dislocations have been made by a broad range of experimental techniques, of which transmission electron microscopy is the one that is most widely used to date.

The importance of dislocations to the field of materials science and engineering lies in the fact that they are the carriers of plastic deformation in crystalline materials [6,7]. The mechanical properties of metals may therefore be tailored by altering the extent to which dislocations can nucleate, propagate or interact. For example, the high hardness and yield strength of many alloys is
achieved by introducing obstacles to dislocation motion, such as solute atoms or second phase particles.

Since metals and alloys are most common in their polycrystalline form (i.e. they consist of many crystals separated by planar defects termed grain boundaries), the interaction between dislocations and grain boundaries is of particular interest. Grain boundaries act as obstacles to dislocation motion as conveyed through the classical Hall-Petch relation \([8,9]\) describing the increase in yield strength of polycrystalline metals with decreasing obstacle distance. However, plastic deformation of such materials involves a wide range of interaction phenomena between dislocations and grain boundaries, which are still subject to extensive research. Moreover, with the ongoing miniaturization of devices and materials, length scales have come within reach at which the mechanisms by which deformation proceeds change drastically. A thorough understanding of such mechanisms is required to improve the mechanical properties of advanced materials. In line with this ultimate objective, the work presented in this thesis is aimed at clarifying the interaction mechanisms between lattice defects from the perspective of their relevance to the mechanical behavior of metals.

### 1.2 Scope and outline of the thesis

In Chapter 2, the physical concepts of dislocations and grain boundaries are introduced, and some basic interaction mechanisms between them are discussed. The remainder of the chapter is concerned with the experimental methods used throughout this thesis. The main microscopic techniques employed are transmission electron microscopy and electron backscatter diffraction, and their aspects that are relevant to the observation of dislocations and grain boundaries are reviewed. Subsequently, the key principles of nanoindentation are introduced. Nanoindentation is a relatively new technique for mechanical testing at submicrometer scales and has seen a great amount of development over the past twenty years. In the work described in this thesis, it is used to locally introduce dislocations near grain boundaries in a controlled way, and to measure the resulting mechanical response.

Chapter 3 presents a nanoindentation study of the incipient plastic behavior of grain boundaries in body-centered cubic metals. The experiments comprise the systematic measurement of the mechanical response to indentation at submicrometer distances from well-defined grain boundaries in bicrystalline
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specimens. Body-centered cubic metals have been chosen because of the inherently high resistance of grain boundaries to dislocation transmission in this crystal structure. The results show that characteristic features in the indentation measurements can be used to determine the maximum shear stress that a grain boundary can sustain and thus provide a unique method for locally probing the strength of individual grain boundaries.

A major drawback of experimental and theoretical research in the field of dislocations is that most of the work has been concentrated on static structures. Obviously, the dynamics of moving dislocations are more relevant to the deformation of metals. Nuclear spin relaxation methods in the rotating frame have been developed as a complementary tool for studying dislocation dynamics in metals [10]. A strong advantage of this technique is that it detects dislocation motion in the bulk of the material, as opposed to in situ transmission electron microscopy, where the behavior of dislocations may be affected by image forces due to the proximity of free surfaces. However, information about the local response of dislocations to an applied stress cannot be obtained by nuclear spin relaxation and therefore in situ transmission electron microscopy remains a valuable tool in the study of dislocation dynamics. Direct observation of dislocation behavior during indentation has recently become possible through in situ nanoindentation in a transmission electron microscope. In Chapter 4, we use this novel technique to study deformation mechanisms in Al and Al-Mg alloys with grain sizes of the order of a few hundred nanometers. At these grain sizes, stress-induced movement of grain boundaries is an important deformation mechanism in pure Al. In Al-Mg however, the grain boundaries are found to be effectively pinned by solute Mg atoms. Such pinning effects may significantly enhance the mechanical properties of ultrafine-grained or nanocrystalline alloys.

Chapter 5 deals with the mechanical behavior of Al-Mg alloys with a substantially larger grain size. Under specific conditions of strain rate and elevated temperature, coarse-grained Al-Mg alloys exhibit superplastic properties, i.e. they show very high elongations prior to failure, typically in excess of a few hundred percent. This makes them attractive candidates for the production of components with a large freedom of design. The physical mechanisms by which coarse-grained superplastic alloys deform are markedly different from those involved in conventional superplasticity of fine-grained materials. In particular, they allow for much higher forming rates, which is a considerable advantage from the perspective of commercial viability. Chapter 5 concentrates on the deformation mechanisms responsible for the superplastic properties of coarse-
grained Al-Mg alloys. To this end, the microstructure and dislocation substructure of the alloys are analyzed as a function of the deformation parameters. The observations are discussed in relation to dynamic reconstruction mechanisms and their influence on the ductility of the alloys.

References

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3. E. Orowan, Z. Phys. 89 (1934), 605
4. M. Polanyi, Z. Phys. 89 (1934), 660