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Grain Size and Solid Solution Strengthening in Metals

A Theoretical and Experimental Study

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Doctoral Dissertation

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ABSTRACT

The understanding of the strengthening mechanisms is crucial both in the development of new materials with improved mechanical properties and in the development of better material models in the simulation of industrial processes. The aim of this work has been to study different strengthening mechanisms from a fundamental point of view that enables the development of a general model for the flow stress. Two different mechanisms namely, solid solution strengthening and grain size strengthening have been examined in detail. Analytical models proposed in the literature have been critically evaluated with respect to experimental data from the literature. Two different experimental surface techniques, atomic force microscopy (AFM) and electron backscattered diffraction (EBSD) were used to characterize the evolving deformation structure at grain boundaries, in an ultra low-carbon (ULC) steel. A numerical model was also developed to describe experimental features observed locally at grain boundaries.

For the case of solid solution strengthening, it is shown that existing models for solid solution strengthening cannot explain the observed experimental features in a satisfactory way. In the case of grain size strengthening it is shown that a simple model seems to give a relatively good description of the experimental data. Further, the strain hardening in materials showing a homogenous yielding, is controlled by grain boundaries at relatively small strains. The experimental results from AFM and EBSD, indicate more inhomogenous deformation behaviour, when the grain size is larger. Both techniques, AFM and EBSD, correlate well with each other and can be used to describe the deformation behaviour both on a local and global scale. The results from the numerical model showed a good qualitative agreement with experimental results.

Another part of this project was directed towards the development of continuum models that include relevant microstructural features. One of the results was the inclusion of the pearlite lamellae spacing in a micromechanically based FEM-model for the flow stress of ferritic-perlitic steels. Moreover a good agreement was achieved between experimental results from AFM and FEM calculations using a non-local crystal plasticity theory that incorporates strain gradients in the hardening moduli.

The main philosophy behind this research has been to combine an evaluation of existing strengthening models, with new experiments focused on studying the fundamental behaviour of the evolving dislocation structure. This combination can then be used to draw general conclusions on modelling the strengthening mechanisms in metals.

Keywords: strengthening mechanisms, flow stress, solid solution strengthening, grain size strengthening, micromechanical modelling, AFM, EBSD

"It is sometimes said that the turbulent flow of fluids is the most difficult remaining problem in classical physics. Not so, workhardening is worse."

Sir. A.H. Cottrell

Preface

After working for 3,5 years in the steel industry I started my research work for two main reasons. One was that after my years with practical steel development I really wanted to understand the mechanisms behind the mechanical properties. The other reason was the interesting opportunity to collaborate with researchers from other fields.

I have for a long time found it fascinating that one of the fundamental tools for an engineer and one of the simplest mechanical tests, namely the stress strain curve, obtained from a tensile test, cannot yet be fully predicted, at least not for commercial alloys. Gaining a greater insight into the abstract and mysterious world of dislocations also offers a challenge.

One of the original aims of this research work was to understand and explore the superposition and interaction of different strengthening mechanisms. However, as in a scientific endeavour of this type, the thesis deals with a number of other unexplored problems among the different strengthening mechanisms. Nevertheless a limited literature survey is presented on the different strengthening mechanisms and their interaction/superposition. The research presented here can hopefully contribute towards a deeper understanding of the strengthening mechanisms and set the ground for attacking the problem of superposition.

This thesis consists of an introductory part and the following appended papers:

I Solid Solution hardening - a comparison of two models
Dilip Chandrasekaran
Materials Science and Engineering, A309-310, (2001) 184-189.

II Grain Size Strengthening in Polycrystals
Dilip Chandrasekaran and Kjell Pettersson
Modified version of paper in MRS Proceedings Volume 683E, BB2.8. 1-6, San Francisco, USA, 2001.

III Micromechanical Modelling of Two-Phase Steels
Mikael Nygårds, Dilip Chandrasekaran and Peter Gudmundsson
Modified version of paper in MRS Proceedings Volume 653, Z8.8. 1-6, Boston, USA, 2000.

IV Comparison of Surface Displacement Measurements in a Ferritic Steel using AFM and Non-Local Crystal Plasticity
Dilip Chandrasekaran and Mikael Nygårds
Accepted for publication in Materials Science and Engineering.

V A Study of the Surface Deformation Behaviour at Grain Boundaries in an Ultra Low-Carbon Steel
Dilip Chandrasekaran and Mikael Nygårds
Acta Materialia, vol. 51,(2003) pp. 5375-5384.

VI Grain Size Strengthening at Small Strains – Analysis of Experimental data and Modelling Implications
Dilip Chandrasekaran and Göran Engberg
Submitted to International Journal of Plasticity, 2003.

Stockholm, October 2003

Dilip Chandrasekaran

CONTENTS

Chapter 1

General Introduction

The ultimate dream for a materials scientist is to be able to predict the mechanical behaviour of a material from its composition and microstructure. The fascinating paradox of materials science is that the problem to be solved can be stated in so simple terms but is so difficult to solve. Today we are still quite far from a complete understanding of the mechanical behaviour of metals. The complexity of this problem requires knowledge of mathematics, physics, mechanics and chemistry for its solution.
What is the motivation for understanding and modelling the mechanical properties?
The answer is quite obvious with the ongoing technological development towards more efficient and environmental friendly processes and products, more advanced materials need to be developed at shorter times and at less cost.

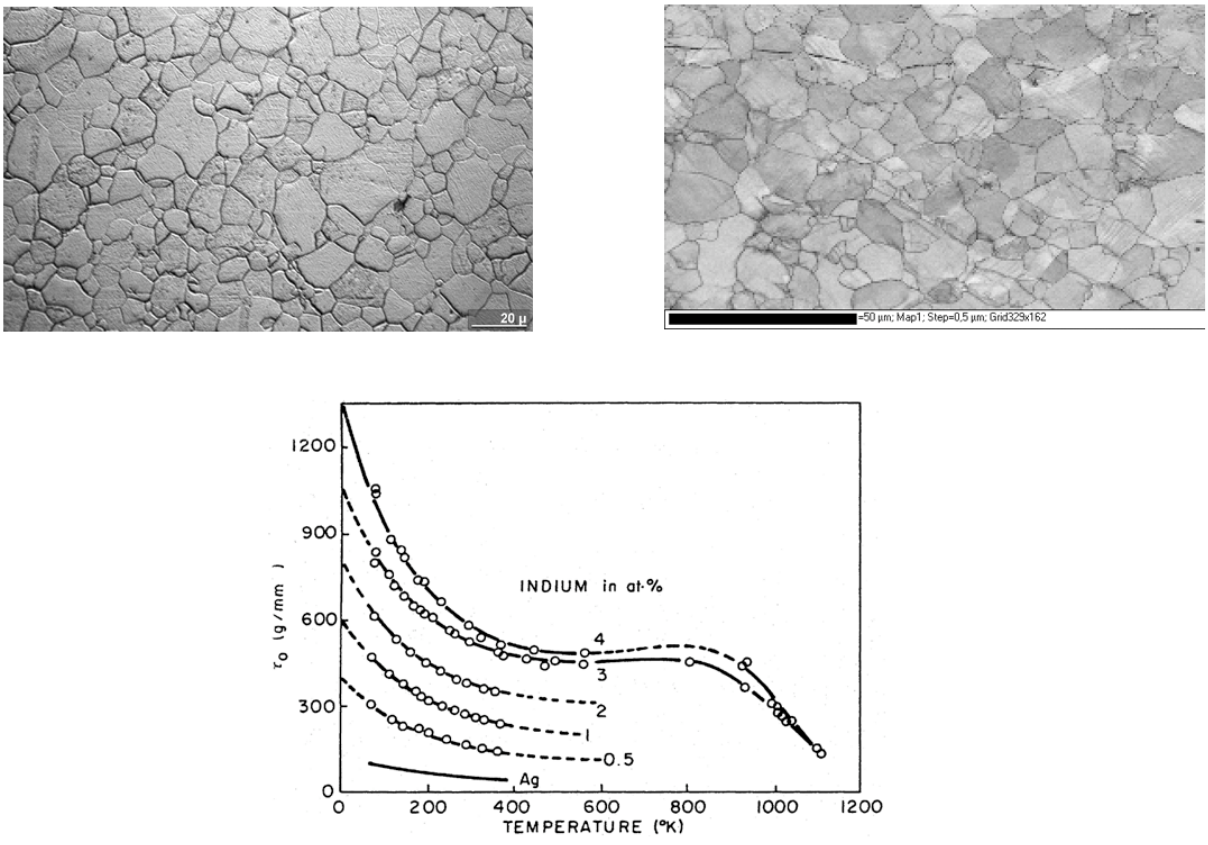


Fig 1.1 Light optical micrograph, showing the grain structure of low-carbon steel (top left). Band contrast image, revealing crystal orientations in deformed ultra-low-carbon steel (top right). Graph showing the variation of the yield stress with temperature in Ag-In alloys (Boser 1972).

Thus, the reasons for developing better models are several;

- Costly experiments can be avoided in the development of new materials
- Improved models are an important tool in the search towards a more fundamental understanding.
- Materials can be tailor-made for different applications.

The basic philosophy behind this thesis, may be represented by Fig. 1.1. In this research work, a study of the microstructure (represented by the micrograph of a carbon steel) is combined with information on the change in crystal orientations (represented by the band contrast image) together with a theoretical analysis of stress strain data (represented by a graph showing the variation of the yield stress with temperature). This covers the scope of this thesis work.

The more specific aim of the research work is to analyze the mechanical behaviour from a fundamental point of view, which would enable us to draw general conclusions concerning the mechanisms behind the strengthening in metals. In short the two main aims of this thesis are:

- To contribute towards a greater understanding of the mechanisms behind grain size and solid solution strengthening.

- To explore and combine different experimental techniques and use them to understand fundamental deformation mechanisms.

The first issue is addressed both by analyzing experimental data in the literature and by developing different types of modelling approaches. The focus of the modelling work has been on analytical models in the literature and their validity and limitations. The experimental work in this thesis was directed towards two different surface characterization techniques for the study of the evolving deformation structure. Ideally in future we should be able to understand and predict the mechanical behaviour of commercial alloys with complex microstructures, from information about the manufacturing process. In this thesis fundamental issues concerning the strengthening of metals are discussed. An attempt is made to answer the following general question:

What is the stress strain behaviour of a deforming metal sample, given its composition and microstructure?

The thesis surveys the different mechanisms contributing to the strengthening in metals. The survey is by no means complete and only two mechanisms, namely grain size strengthening and solid solution strengthening are treated in detail. Other very interesting issues like the superposition and interaction of mechanisms are only discussed briefly.

1.1 Flow stress modelling

This thesis work is based on dislocations, their behaviour and interaction with obstacles. The understanding of the dislocation behaviour in metals is fundamental in understanding and predicting the mechanical behaviour. Ideally the aim when modelling the mechanical properties is to end up with constitutive equations of the following kind:

$$\sigma = f\{\varepsilon, \dot{\varepsilon}, T, microstructure\} \tag{1.1}$$

This kind of formulation is necessary to model and predict the macroscopic properties from a given microstructure and in the development of generalized models for the flow stress that can be applied to solve more complicated "real" problems like, for example, forming or rolling and even machining. The overall view is illustrated in Fig. 1.2. This thesis covers the work on the strengthening mechanisms from a dislocation viewpoint, while the micromechanical modelling is covered elsewhere (Nygårds 2003). In order to develop a flow stress model that can be used to predict mechanical properties and simulate real processes micromechanical modelling and dislocation modelling should be combined together with a microstructurally relevant length scale.

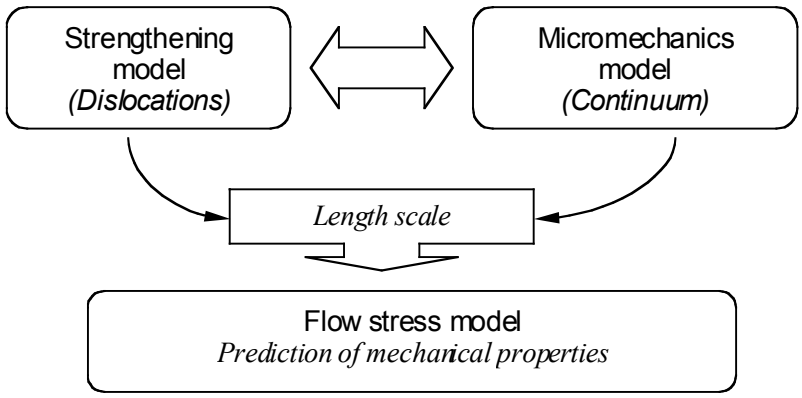


Fig 1.2 A layout of the general idea in modelling the flow stress.

1.2 Length Scales

An important aspect in the development of finite element models for flow stress is the concept of length scales. Classical continuum models do not contain a microstructural length scale, i.e. in such a model the influence of grain size and dislocation structure cannot be taken into account. A number of approaches, where local effects can be taken into account, are now being developed. One difficulty in the development of these approaches is the lack of good experimental information on a microstructral scale. This thesis aims to discuss the issue of the

required length scale in continuum models, both in the light of new experimental information and some modelling results. Some ideas on relevant microstructural features in continuum models are discussed in more detail in papers 3 and 4. A lot of research work is also being directed towards the modelling of mechanical behaviour on different scales using different techniques such as, simulation of discrete dislocations (DD) or molecular dynamics (MD) simulations, just to mention a few examples. The appropriate length scale to be included in such models depends on the type of problem and the desired resolution required.

1.3 Experimental issues

As mentioned earlier, two different surface characterization (2D) techniques namely, atomic force microscopy (AFM) and electron backscattered diffraction (EBSD), are used to study plastic deformation, in this thesis. However, even though plastic deformation is essentially a 3D process a number of difficulties are involved in performing 3D studies of plastic deformation. Perhaps bulk techniques such as 3-dimensional X-ray diffraction (3DXRD) and high-resolution transmission electron microscopy (HRTEM) may be applied but unfortunately these are expensive and demanding methods, when it comes to sample preparation. The experimental methods used in this thesis on the other hand, are relatively inexpensive and require a minimum of sample preparation. Therefore the information from these methods combined with information from the macroscopic bulk behaviour, should prove useful in contributing to a deeper understanding of the inherent mechanisms. A few of the discrepancies between surface and bulk measurements are discussed further in chapter 3.

Chapter 2

Strengthening Mechanisms

In the following section a general overview of the different strengthening mechanisms in metals will be given. A few other aspects such as the superposition and interaction of different mechanisms will also be discussed. Only a brief overview is given here, as there are a great number of reviews and books on these subjects (Kelly and Nicholson 1971; Kocks, Argon et al. 1975; Nabarro and Duesbery, 2002). The reader is recommended to these for a more detailed study. In this general introduction, two strengthening mechanisms, namely solid solution strengthening and grain size strengthening will be discussed in more detail, as this is the focus of the appended papers. Other mechanisms are only dealt with qualitatively, the idea being to provide an introduction towards a general modelling of the strengthening in metals. A few words will also be said on the superposition of different mechanisms.

2.1 General concepts

A fundamental concept in the discussion of the strengthening behaviour in metals, are dislocations, or line defects. In order to understand the mechanisms behind the different strengthening mechanisms, it is vital to understand the behaviour of dislocations and their interaction with different types of defects. Most of the discussion in this section will be concentrated around the interaction of dislocations with different obstacles and also the effect of external variables, such as temperature and strain rate. One way of describing the strengthening in metals is to evaluate the response of a material due to a prescribed load. The simplest and most used experimental test method is uniaxial tensile testing which results in a stress strain curve. In this chapter and in this thesis, we will restrict ourselves to the strengthening occurring during a tensile test. The fundamental mechanisms discussed are naturally valid for many other problems and applications.

Traditionally the flow stress has been modelled as the sum of the different strengthening contributions, although it is by no means self-evident that the contributions are additative and in some cases not true at all. One can also write the flow stress σ_f, as a sum of two components, one temperature T and/or strain rate $\dot{\varepsilon}$ dependant part σ^*, and one athermal part σ^a.

$$\sigma_f = \sigma^a + \sigma^*(\dot{\varepsilon}, T) \tag{2.1}$$

As will be seen later, dislocations may bypass some of the strengthening obstacles by thermal activation while others are too large to be bypassed unless the stress is higher. Another way of

describing the flow stress is by the following relation, which has been experimentally verified for a number of different metals and alloys. The flow stress τ_f at certain plastic strain is then expressed as,

$$\tau_f = \tau_0 + \alpha G b \sqrt{\rho} \qquad (2.2)$$

where τ_0 is a friction stress, G the shear modulus, b the Burgers vector and α a proportionality constant. As ρ increases during deformation Eq. (2.2) actually predicts work hardening. This relation was first proposed by Taylor and a great number of theories have been proposed in the literature since then, to explain the work hardening behaviour. The friction stress is often given as the linear sum of the other strengthening contributions, such as solutes, precipitates, grain boundaries and Peierls-Nabarro barriers. A more detailed discussion on the superposition of different mechanisms shall be presented later.

2.2 Solid Solution Strengthening

The strengthening effect of solutes is well known and has been investigated by a number of researchers over the years. A number of different interactions[1] exist between solutes and the solvent lattice, but here we will only consider interactions of elastic type which are essentially of two kinds namely, size effects and modulus effects. The former is caused by a size misfit of a solute atom causing strains in the lattice and the latter by differences in shear modulus between solutes and the lattice.

Different theories have been proposed in the literature to explain and model the experimental features of solid solution strengthening and there are a number of excellent reviews (Fleischer 1963; Kocks 1985; Butt and Feltham 1993; Cahn and Haasen 1996) on the subject to which the reader is referred to for a more detailed study. A few important concepts, concerning the modelling of solid solution strengthening will be discussed now. In order to model the experimental information on solid solution strengthening, one requires a model which incorporates the actual strengthening effect of solutes (concentration), with the temperature dependence observed experimentally in the solution-strengthened alloy (Kocks 1985).

One of the classical efforts to model and classify the effect of solute/dislocation interactions was by Fleischer (Fleischer 1963) and a short summary of his approach will be presented here. Fleischer classified the nature of hardening in terms of the distortion a solute atom causes in the lattice. Symmetrical distortions, e.g. substitutional atoms in a fcc-lattice, or asymmetric distortions, e.g. interstitials in bcc (Fleischer and Jr. 1963). The hardening effect of an asymmetric distortion is often an order of magnitude larger. The elastic interaction

[1] Other interactions include chemical-, electrostatic- and stress-induced order locking

between a solute and a dislocation can then be described depending on the type of dislocation (edge or screw). A typical example of a force-obstacle profile is shown in Fig. 2.1 below.

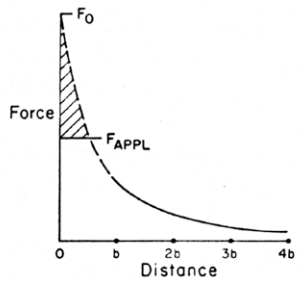


Fig. 2.1 Schematic of a typical force-distance diagram for the case of solid solution strengthening, taken from Fleischer (Fleischer 1967).

If the interaction force is integrated over a certain interaction distance, an energy for the specific obstacle-profile considered, can be defined. This can be identified as the activation energy for the process. By comparing the predicted hardening with experimental information, the controlling mechanism can be evaluated. For instance, the hardening in substitutional copper alloys has been shown by Flesicher, to be controlled by the stress needed to move screw dislocations and this from a combined effect of atomic size and modulus difference (Fleischer 1962).

2.2.1 Dislocation Line Flexibility

Another important concept in solution hardening is the flexibility of the dislocation line. There are two main approaches here, namely Fleischers's (Fleischer and Jr. 1963) and Mott and Nabarro's (Mott and Nabarro 1948). In Fleischer's approach a moving dislocation is assumed to encounter a series of individual discrete obstacles on the slip plane. The spacing L, between these then depends on the flexibility of the dislocation line (see Fig. 2.2). The concept of discrete obstacles is the same as the one originally introduced by Friedel (Friedel 1956) where L is defined from the requirement that the dislocation loop, while passing an obstacle, encounters one and only one new obstacle.

This differs for example, from the earlier treatment by Mott and Nabarro where the resistance to dislocation motion is assumed to stem from an internal stress. In their treatment, a dislocation line in equilibrium under an internal stress will acquire a curved or zigzag shape.

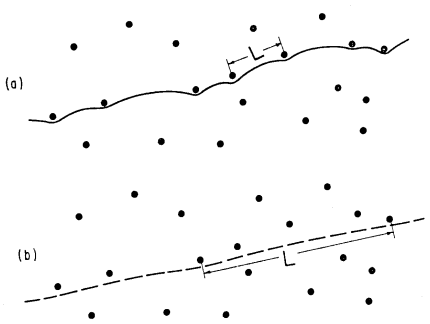


Fig. 2.2 Average solute spacing L depending on the flexibility of a dislocation line, from Fleischer (Fleischer and Jr. 1963).

2.2.2 Concentration Dependency

The concentration dependency in solution hardening, predicted by the different approaches, does not vary much, ranging from parabolic to linear hardening and values in between. Essentially the different solution hardening models proposed in the literature are similar. They all consider solutes as discrete obstacles (except Mott (Mott and Nabarro 1948; Mott 1950) and the hardening is then assumed to stem from differences in size and/or modulus of the solutes. The concentration dependency of the flow stress will then vary depending on how the flexibility of the dislocation line is expressed.

Kocks et al. (Kocks, Argon et al. 1975) have discussed the differences between *Mott-statistics* and *Friedel-statistics*. The former is valid in the case of weak obstacles and concentrated solutions, while the latter for dilute solutions and stronger obstacles. The stress to bypass obstacles may be written in the following general form, as originally introduced by Orowan,

$$\tau = \frac{F_o}{bL} \tag{2.3}$$

where F_o, is the obstacle strength, due to solutes, particles etc, and L is the average spacing between obstacles. Using the above expression and suitable statistics the following expression can be derived for the strengthening effect due to solutes at 0 K:

$$\tau_s = \mu \cdot f^n \cdot c^m \tag{2.4}$$

This expression contains the shear modulus μ, the solute concentration c and a measure of the obstacle strength f. In this form it covers several different theories[2]. The exponent n will vary depending on the assumptions concerning the nature of the obstacles and m depending on how

[2] In this context the statistical theory for solid solution hardening developed by Labusch (Labusch 1970) should be mentioned, this predicts a m-value of 2/3, taking into account local variations of the dislocation line and its interaction with randomly distributed solutes.

the average spacing L is defined. For a completely straight dislocation line, L= b/c and for a more flexible dislocation line, L= $b/c^{1/2}$. The scatter in the experimental information makes it possible to fit different concentration dependencies. For tetragonal distortions, e.g. carbon in bcc-iron, the flow stress is found experimentally to vary proportionally with the square root of the carbon content (Wert 1950) as predicted by Fleischer (Fleischer 1962).

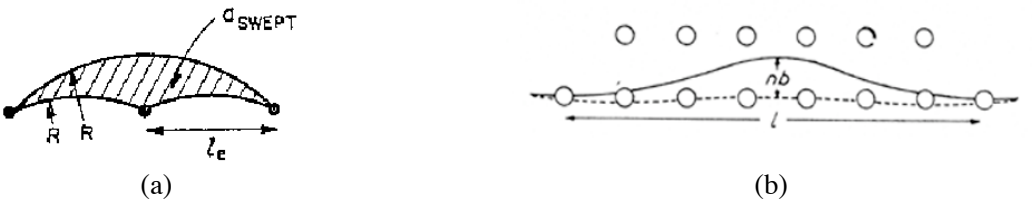
2.2.3 Thermal Activation

In the treatment so far we have not accounted for temperature effects and the treatment presented so far only gives the yield stress at 0 K. At temperatures above absolute zero thermally activated dislocation motion is an important mechanism. This can be seen experimentally by the strong temperature dependency of the yield stress, observed for different alloy systems (Hutchison and Honeycombe 1967; Nakada and Keh 1971). It seems reasonable then, that due to the short-range nature of solute obstacles thermal activation should be an important mechanism. This does not rule out the existence of an athermal solution hardening effect due to solutes indicated in several alloy systems (Kocks 1985).

Experimental observations of the variation of yield stress with temperature sometimes shows a plateau in the yield stress, as can be seen in Fig. 1.1. This is the case, e.g. for Ni-C alloys (Nakada and Keh 1971) and Ag-alloys (Hutchison and Honeycombe 1967). This type of behaviour cannot be explained using a discrete obstacle approach. These shortcomings led to the development of the models of collective type, which have been hence applied to more concentrated solid solutions. The two different approaches in modelling the temperature dependency of the flow stress can be summarised as below:

1. Solutes are treated as discrete obstacles and are overcome by an individual activation event (see Fig. 2.3a) ⇒ Single obstacle models.

2. The dislocation line is locked along its length by solutes and the activation event involves several atoms (see Fig. 2.3b) ⇒ Collective models.



(a) (b)

Fig. 2.3 Difference between (a) a discrete-obstacle approach where the dislocation line encounters only one obstacle at a time i.e. Friedel statistics (Kocks, Argon et al. 1975) and (b) a collective approach where the dislocation line has to breakaway from a row of solutes (from (Feltham 1968)).

Using reaction rate theory an Ahrrenius-type expression can be written for the activation energy ΔG, as a function of strain rate $\dot{\varepsilon}$, and temperature T. In Eq. (2.5) k is Boltzman's constant and $\dot{\varepsilon}_0$ is a pre-exponential factor (related to the Debye frequency) in the order of 10^{12}-10^{14} s^{-1}. The activation energy ΔG, will then be a function of the applied stress τ, and the nature and size of the interaction between the obstacle and dislocation.

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp\left(-\frac{\Delta G}{kT}\right) \tag{2.5}$$

The temperature variation of the flow stress then depends on the assumed obstacle profile and its stress dependency. Kocks et al. (Kocks, Argon et al. 1975) have proposed a phenomenological expression to generalise all discrete-obstacle models. The activation energy ΔG, to overcome a discrete obstacle is then given from the following expression,

$$\Delta G = F_0\left[1 - \left(\frac{\tau}{\hat{\tau}}\right)^p\right]^q \tag{2.6}$$

where p and q are two coefficients, with values depending on the nature of the obstacle. The critically resolved shear stress needed to overcome the obstacle at some temperature T, or at 0 K are represented by τ and $\hat{\tau}$ respectively (assuming strengthening due to only one type of obstacle). Obviously, F_0 can be identified as the activation energy needed at zero applied stress (τ=0).

A number of different collective models have been proposed in the literature (Kocks 1985; Hattendorf and Büchner 1992; Butt and Feltham 1993). These theories usually lead to a more complicated expression for the activation energy as a function of the applied stress. By combining this type of expression with Eq. (2.5) above, the temperature dependency of solid solution strengthening can be modelled.

There are several fundamental differences between a discrete-obstacle approach and a collective approach, and a more detailed discussion can be found in paper 1. One fundamental difference between the different models is presented in Fig. 2.4. Here the stress, normalised by the critically resolved shear stress at 0 K, is shown as a function of the temperature for three different models. It can be noted that for the discrete-obstacle models there exists an upper temperature, T_0, above which thermal activation occurs so easily that no stress is required to bypass the obstacles, while for collective models no such temperature exists.

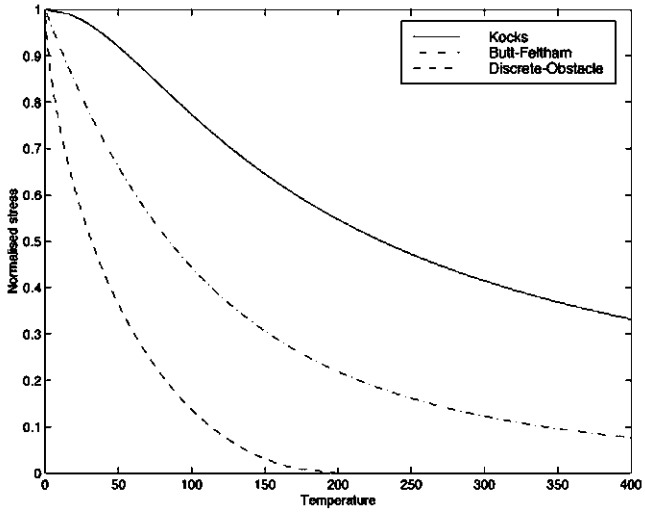


Fig. 2.4 Normalised stress $\frac{\sigma}{\sigma_0}$ as a function of temperature as predicted by a Discrete-Obstacle (--) and two collective models, Butt-Feltham (·-) (Butt and Feltham 1993) and Kocks (-) (Kocks 1985).

2.2.4 Modelling of Solid Solution Strengthening

In order to evaluate the predictive capability of the different approaches discussed above, model predictions were compared with experimental data for three different alloy systems. The discrete-obstacle approach was compared with a collective model proposed by Kocks (Kocks 1985). Rather than adjusting model parameters to the experimental information, reasonable values were calculated and tested. A detailed discussion of the results can be found in paper 1, and we shall discuss some of the main results now.

A comparison between model calculations and experimental data for a Cu-Mn single crystal system and a Ni-C polycrystal system is shown in Figs. 2.5a and 2.5b. As can be seen, the collective model (Kocks) seems to reproduce the experimental data for the two systems remarkably better than the discrete-obstacle model. The third system studied was a Nb-Mo single crystal system and neither approach was found capable to describe the experimental data satisfactorily here. The reason for the poor description of this system is probably due to the influence of other strengthening mechanisms, as discussed in more detail in paper 1.

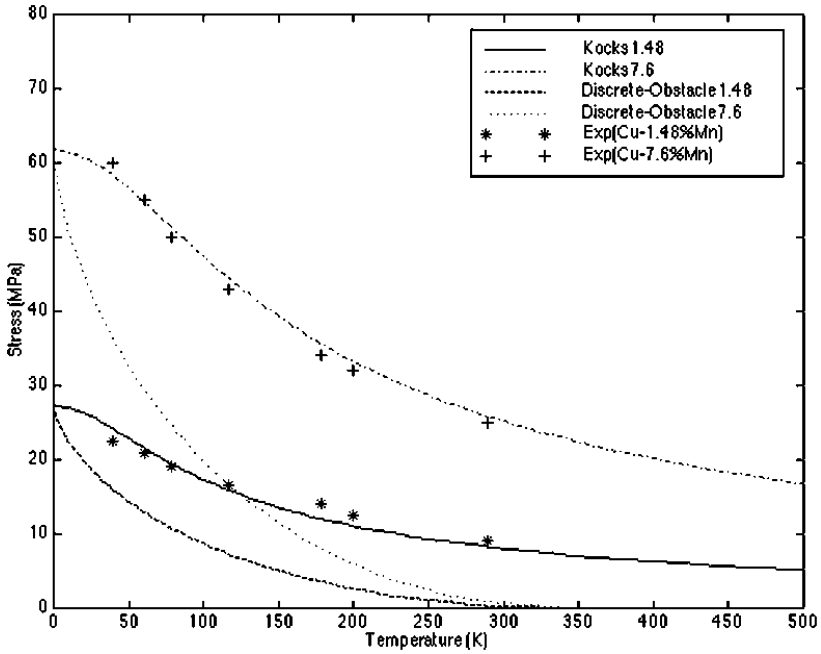


Fig. 2.5a Comparison of a Discrete-Obstacle model and a Collective model (Kocks) with experimental data for Cu-Mn single crystal alloys taken from (Wille and Schwink 1986).

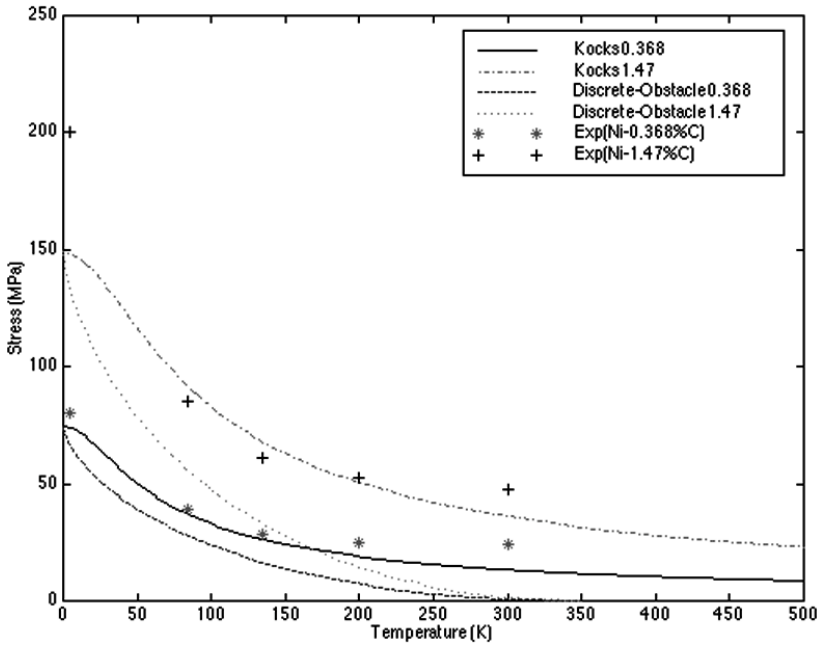


Fig. 2.5b Comparison of a Discrete-Obstacle model and a Collective model (Kocks) with experimental data for Ni-C polycrystal alloys taken from (Nakada and Keh 1971).

Although the discrete-obstacle model has a straightforward physical meaning, where the strengthening effect is caused by misfit strains due to differences in size and/or in shear modulus between solutes and matrix atoms, there are a number of drawbacks. For example, the total interaction energy between a single solute and a dislocation at 0 K, F_0, can hardly depend on the solute concentration. Therefore T_0 (as defined earlier and a direct function of F_0, given from Eqs. (2.5 – 2.6)) must also be concentration independent. As a result, the yield stress predicted by the model, will level out at the same temperature T_0, independent of the concentration. This is in conflict with the experimental data in Fig. 2.5, which indicates that a plateau in yield stress is reached at higher temperatures. In the model proposed by Kocks, the actual strengthening mechanism is more difficult to visualise, although the plateau behaviour can be described fairly well. On the other hand, despite the existence of strong experimental evidence of large strength contributions due to differences in size and modulus (Fleischer and Jr. 1963), no such effects are included in the collective model by Kocks.

To conclude, the experimental data for the systems studied, is better described by a model accounting for a collective overcoming of solutes, rather than overcoming of discrete obstacles. A discrete-obstacle approach includes the experimentally observed strengthening due to differences in size/modulus, but is not capable of describing the experimental information on solid solution strengthening, especially at higher temperatures. A complete description of solid solution strengthening requires a model that can incorporate size/modulus effects with a collective overcoming of solutes, especially at higher temperatures and concentrations.

2.3 Grain Size Strengthening

The strengthening in polycrystals due to grain boundaries has been experimentally established ever since Hall (Hall 1951; Petch 1953) proposed his relation between the grain size and the yield stress. The Hall-Petch relation (given below) has been found to be valid for a number of different systems, both for pure metals and alloys, over quite a large range of grain sizes.

$$\sigma = \sigma_0 + k \cdot d^{-\frac{1}{2}} \qquad (2.7)$$

In the above equation σ is the (upper or lower) yield stress or flow stress, σ_0, is the contribution from other strengthening mechanisms, d is the grain size and k a constant, often known as the Hall-Petch constant. In order to explain the experimental observations of the Hall-Petch effect, several different types of mechanisms have been proposed in the literature. This is discussed in detail in paper 2 and a short summary will be given here. Of the different models to explain the Hall-Petch behaviour, three fundamentally different approaches can be identified, namely pile-up models, dislocation density models and composite models.

2.3.1 Pile-up Models

One of the earliest attempts to explain the Hall-Petch behaviour was the pile-up model by Hall (Hall 1951), with subsequent modifications by Petch (Petch 1953) and Cottrell (Cottrell 1964). The basic idea is that dislocations are assumed to pile-up against a grain boundary, thereby causing a stress concentration. When the stress concentration equals a critical stress, assumed to activate new dislocation sources, yielding starts in the next grain. The simplest pile-up we can imagine is a single-layer pile-up, as illustrated in the figure below.

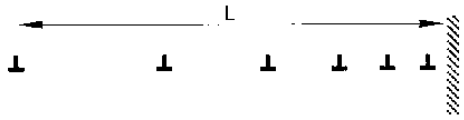


Fig. 2.6 An illustration of a classical pile-up, visualised as a number of edge dislocations piled up at a grain boundary.

The number of dislocations in a single-layer pile-up, as a function of the applied stress and pile-up length, has been derived by Eshelby et al. (Eshelby, Frank et al. 1951). The pile-up length is then proportional to the grain size and going through the algebra we can write the tensile shear stress as:

$$\tau_s = \tau_0 + \sqrt{k_1 \frac{\tau_c \tau b}{\tau}} \cdot d^{-\frac{1}{2}} \qquad (2.8)$$

This relation is identical to Eq. (2.7) earlier, if the square root can be identified with k in Eq. (2.7), with d as the grain-size and k_1 as a constant. The value of k_1 depends on the nature of the pile-up and the assumption coupling the length of the pile-up with the grain size. There are several attractive features with this theory. It gives an explanation for the sharp yield point behaviour in low-carbon steels and it is consistent with the inhomogeneous nature of plastic yielding in these steels. The major drawbacks are that it is not really applicable to all systems (e.g. fcc-metals) and there are no direct observations of pile-ups reported in the literature. It should be mentioned that a number of more complicated dislocation configurations have been proposed in the literature (Li and Chou 1970) although the main features are essentially the same.

2.3.2 Dislocation Density Models

Another approach to explain the Hall-Petch effect and also to explain the observed grain-size dependency at higher strains are the different dislocation density models. They are all based on Ashby's original model (Ashby 1970) of which a very brief outline will be given here.

Ashby based his model on the assumption that the strengthening due to dislocations can be separated into two different contributions, namely that from statistically stored dislocations ρ^S, and that from geometrically necessary dislocations ρ^G. The former quantity is grain-size independent while the latter depends on the grain size. This leads to the following expressions for ρ^S and ρ^G,

$$\rho^S = m\frac{C_1\varepsilon}{bL^s} \tag{2.9a}$$

$$\rho^G = m\frac{C_2\varepsilon}{bd} \tag{2.9b}$$

where $\bar{m}$ is the average Taylor factor and d is the grain size. The dislocation density ρ^S is governed by the geometrical slip distance L^S, in the interior of the grains where the deformation is assumed to be uniform. The non-uniform deformation in the grain boundary region is accommodated by the introduction of geometrically necessary dislocations. These can be seen as the strain bearers needed to account for the plastic incompatibilities in-between grains (Ashby 1970), as illustrated in Fig. 2.7 below.

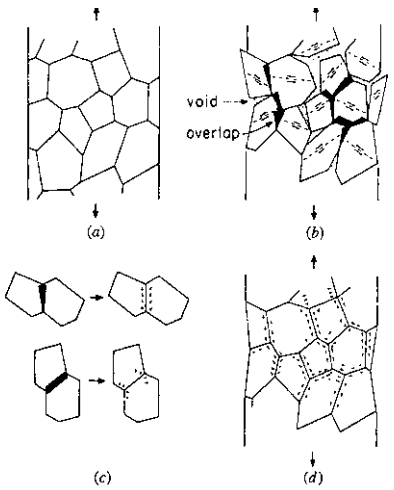


Fig. 2.7 Deformation of polycrystal grains in an uniform manner, causing voids and overlaps (top right), this are corrected by the introduction of geometrically necessary dislocations (bottom right), taken from Ashby (Ashby 1970).

The flow stress can then, in the usual fashion, be expressed as proportional to the square root of the total dislocation density, which leads to:

$$\sigma = \sigma_0 + C\bar{m}\tau^{\frac{3}{2}}\sqrt{\left(\frac{C_1 b}{L^S} + \frac{C_2 b\varepsilon}{d}\right)} \tag{2.10}$$

In the case where grain boundary strengthening dominates, $L^S >> C_1 b$, i.e. the deformation is inhomogeneous, (i.e. $\rho^G > \rho^S$) the above equation reduces to,

$$\sigma = \sigma_0 + C' \sqrt{\rho} \sqrt{C_2 b} \cdot d^{-\frac{1}{2}} \qquad (2.11)$$

where the dislocation-dislocation interaction and the Taylor factor are included in C'. The factor in front of $d^{-1/2}$ can then be identified as k in Eq. (2.7). In Ashby's original paper (Ashby 1970) the constant C_2 in Eq. (2.9b) is given as 0.25, depending on the assumptions concerning the number of dislocations needed at the grain boundaries. The parameter C_2 can be viewed as a measure of the creation rate of geometrically necessary dislocations at grain boundaries.

2.3.3 Composite Flow Stress Models

A third type of approach is the idea of describing the flow stress as the sum of the contribution from grain boundaries and the contribution from grain interiors. A number of different variants have been proposed (Hirth 1972; Thompson, Baskes et al. 1973; Meyers and Ashworth 1982). One such model will very briefly be outlined here.

Thompson et al. (Thompson and Baskes 1973; Thompson, Baskes et al. 1973; Thompson 1975; Thompson 1975) developed a model to describe the Hall-Petch behaviour of fcc-metals by combining concepts from Ashby's model with a composite-type model (Hirth 1972). They assumed the dislocation density in the grain boundary region ρ^G, to be inversely proportional to the grain size but independent of strain. In their expression, the statistical density of dislocations was estimated to be inversely proportional to the geometrical slip distance, L^S. The contributions to the flow stress from the different area fractions were then added, using a rule of mixtures. Assuming the area of the grain boundary region as L^S/d, this leads to the following expression for the flow stress:

$$\sigma = \sigma_0 + \left(1 - \frac{L^S}{d}\right)\frac{K_1}{L^S} + \frac{L^S}{d} K_2 d^{-\frac{1}{2}} \qquad (2.12)$$

When L^S approaches d, the grain size, i.e. at very small strains, the above expression reduces to the form of Eq. (2.7), with K_2 equal to k. The physical significance of K_2 is not very clear, but it should basically have the same meaning as C_2 in Ashby's model although the interpretation is not as straightforward.

2.3.4 Modelling of Grain Size Strengthening

The different models presented earlier were developed to explain specific features of the experimental systems studied. Certain factors should be kept in mind when modelling grain size strengthening. For example different stress strain behaviour (sharp or smooth yielding) is caused by different mechanisms and have therefore to be modelled separately. The

inhomogeneous yielding in low-carbon steels is due to the propagation of Lüders bands, a process that depends, among other quantities, on the grain size. This has to be taken into account in the modelling of the upper and lower yield stress in these steels. On the other hand, fcc-materials that yield more homogeneously, can be modelled using one of the approaches presented above, at least at the yield point. An alternative treatment is presented in paper 2. There are a number of parameters in the different models and the physical significance of these, are not always so clear. Concerning pile-up models, more complicated dislocation configurations than a simple single-layer pile-up, are possible (Li and Chou 1970). It is also generally very difficult to observe pile-ups and other dislocation configurations experimentally in these materials at room temperature. This due to the easy occurrence of cross-slip (Engberg 1979).

Another important issue is the role of grain boundaries during the initial stages of plastic deformation. There are certain indications of higher dislocation activity around grain boundaries than in grain interiors (Hansen and Ralph 1981; Hansen 1985; Jago and Hansen 1986). Grain boundary source mechanisms have also been proposed as an alternative to pile-up models (Li 1963). Although this mechanism cannot be assumed to act as a dislocation generator, one can visualise the creation of a single dislocation that can then interact with other existing dislocations, leading to the propagation of plastic deformation from grain to grain.

This brings us to the next issue, the grain size strengthening observed at higher strains. This effect at higher strains has been observed in both low-carbon steels (Bergström and Hallén 1983) and in copper (Hansen 1985). The results also indicate, a grain size strengthening effect present at higher strains, which is independent of strain. This is not consistent with any of the models presented here. This point will be treated more extensively in section 2.3.5. Generally speaking, the grain size is not a particularly good variable at higher strains where the flow stress is rather a function of the dislocation substructure and its evolution. Moreover at higher strains the change in grain shape and texture evolution must be accounted for.

Another important factor is the influence of temperature and solute content on the Hall-Petch constant. Several suggestions have been made in the literature on the possible effect of solute carbon (or nitrogen) on the Hall-Petch constant and a number of researchers (Russel, Wood et al. 1961; Wilson 1967) have suggested that the presence of carbon atoms at grain boundaries should influence the unpinning stress, although the exact mechanism is not specified. If the upper yield stress can be seen as the stress needed to unpin locked dislocations, it seems likely that an increased carbon content, should lead to stronger locking. In this context it is interesting to discuss the effect of temperature on the Hall-Petch constant. A process involving unlocking from interstitial solutes should be thermally activated and several investigations have been concerned with the temperature dependence of the Hall-Petch constant (Embury 1971). Experiments on low carbon steels show that for quenched and aged

specimens k has a strong temperature dependence while in slowly cooled specimens k is relatively insensitive to temperature (Dingley and McLean 1967; Embury 1971). These results suggest a stronger temperature effect in specimens with a larger solute content, i.e. with a stronger locking effect.

Solutes can also influence the Hall-Petch behaviour in materials showing smooth yielding. A number of investigators have reported an increase in the Hall-Petch constant with increasing alloying contents, especially in copper (Hall 1970) and in nitrogen alloyed austenitic stainless steels (Norström 1977; Gavriljuk, Berns et al. 1999). In the light of the models discussed here it is hard to incorporate a direct effect of solutes into any of them. An indirect effect on the hardening behaviour is possible, there being some evidence that nitrogen changes the slip character (Gavriljuk, Berns et al. 1999) and enhances planar slip during the deformation of austenitic steels. There is also an effect of nitrogen increasing the stacking fault energy (Gavriljuk, Berns et al. 1999). These factors could possibly influence the grain size strengthening in an indirect fashion.

2.3.5 A phenomenological and analytical treatment of grain size strengthening

As mentioned earlier, one interesting observation from the experimental data is the grain size strengthening observed at higher strains, which is not captured satisfactorily in the models described earlier. This point is the focus of Paper 6, where experimental information from different alloys is analysed in more detail, using a classical single parameter work hardening model. A brief summary and discussion of the results will be given here.

The starting point of our discussion is the assumption that strain hardening is controlled by the grain size at small strains and by the inherent dislocation structure at larger strains. In the following, we will focus on materials exhibiting a homogenous yielding on a macroscopic scale, i.e. having a smooth stress strain curve. If the evolution of dislocation density with strain, is expressed in the following general fashion suggested by many authors e.g. Bergström (Bergström 1970) or by Kocks and Mecking (Mecking and Kocks 1981),

$$\frac{d\tau}{d\gamma} = \frac{\overline{m}}{b}\left(\frac{k_1}{d} + k_2 \cdot \sqrt{\rho}\right) - k_3 \cdot \rho \qquad (2.13)$$

where b in the above equation is the Burgers vector, $\overline{m}$ is the Taylor factor and k_1, k_2 and k_3 are three dimensionless constants characteristic of the material under consideration. The first term, in the equation, is strain independent and varies only with the grain size, d. The second term represents the multiplication of dislocations with increasing strain and the last term the annihilation and remobilisation of dislocations at larger strains. This type of phenomenological description has proven to be very useful in the past. One implication of Eq.

(2.13) is that the mean free path of dislocations at very small strains, is a function of the grain size, d. With increasing strain, the mean free path will decrease, due to dislocation-dislocation interactions. At still larger strains, the increase in dislocation density will level out due to the annihilation of dislocations. This can be compared with the discussion on Ashby's model earlier in section 2.3.2. If the above equation is combined with the general expression for the flow stress given in Eq. (2.2), we can, after certain reformulation, express the strain hardening, $d\sigma/d\varepsilon$ as:

$$\frac{d\sigma}{d\varepsilon} = C_1 \cdot \frac{1}{d} \cdot \frac{1}{(\sigma - \sigma_0)} + C_2 \qquad (2.14)$$

C_1 and C_2 are here given by:

$$C_1 = \frac{\bar{m} k_1}{b} \cdot \frac{(\bar{m} \alpha G b)^2}{2} \qquad (2.15a)$$

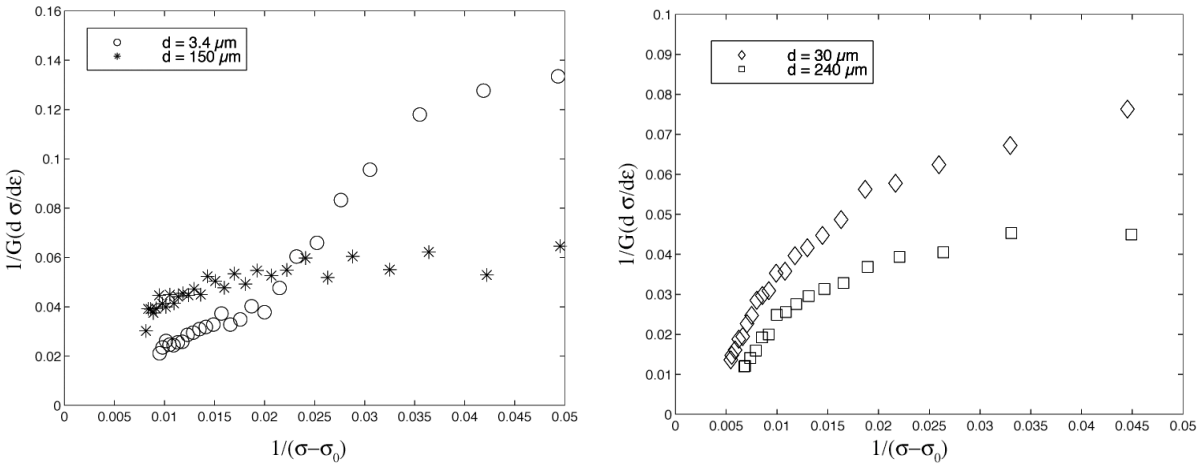
$$C_2 = \frac{k_2}{2} \bar{m}^2 \alpha G \qquad (2.15b)$$

In this derivation, we have omitted the third term in Eq. (2.13), assuming relatively small strains (up to 10 %) and thereby neglecting recovery effects. Equation (2.14) predicts a gradual decrease in the strain hardening with increasing flow stress (i.e. increasing strain) and the initial slope will depend on the grain size. We may also solve analytically the differential equation (2.13), which is fairly straightforward, assuming once again that we can neglect recovery of dislocations at small strains. Unfortunately it is not possible to express the dislocation density as an explicit function of the strain, as we would like. The integration of Eq. (2.13) combined with Eq. (2.2) leads finally to the following expression for the strain as a function of the flow stress.

$$\varepsilon = \frac{2b}{\bar{m}} \left[\frac{d}{k_1} \frac{(\sigma - \sigma_0)}{\bar{m} \alpha G b} - \frac{k_1}{d \cdot k_2^2} \ln\left[1 + \frac{d \cdot k_2}{k_1} \frac{(\sigma - \sigma_0)}{\bar{m} \alpha G b} \right] \right] \qquad (2.16)$$

A more detailed derivation of the expressions above and a lengthier discussion can be found in Paper 6. In the following section the described treatment is compared with experimental data for two different systems, an iron-titanium alloy (bcc) and pure copper (fcc). In the first type of evaluation, the experimental data was replotted with the strain hardening, $d\sigma/d\varepsilon$, as a function of one over the difference in stress, $1/\sigma - \sigma_0$. This is shown for the different systems in Figs. 2.8a-b. The figures can be interpreted in the following way. At low strains i.e. to the right hand side of the Figs. 2.8a-b, the experimental information for the two different grain

sizes are well differentiated indicating that the strain hardening at the initial stages of plastic deformation is controlled by the grain boundaries. Although, according to Eq. (2.14), there should be a linear dependency between, $d\sigma/d\varepsilon$ and $1/\sigma-\sigma_0$, that is not reflected in the Figs. 2.8a – b. Furthermore the grain size dependency predicted by Eq. (2.14) is much stronger than what is seen in Figs. 2.8a – 2b. This large discrepancy is partly due to the difficulties in evaluating the experimental data from the literature. Another important factor could be the difference in the work hardening behaviour with grain size. Firstly, there is the influence of texture, the experimental data does not indicate the initial texture for the different grain sizes and the initial texture can vary for the different grain sizes. Secondly, it has also been reported in the literature (Gracio and Fernandes 1989) of the difference in substructure evolution with grain size. It was shown there for copper, that large grained samples ($d > 60$ μm) behaved more like single crystals compared to fine-grained. The findings in the literature along with the discrepancy noted here, indicate that a more sophisticated approach, than Eq. (2.14), is needed. The focus of this study is not to give a complete description of the influence of grain size on the work hardening, but to emphasise that the rather simple-minded approach presented here is sufficient to draw useful conclusions. The results still indicate that the strain hardening, at the initial stages of plastic deformation, is controlled by the grain boundaries.

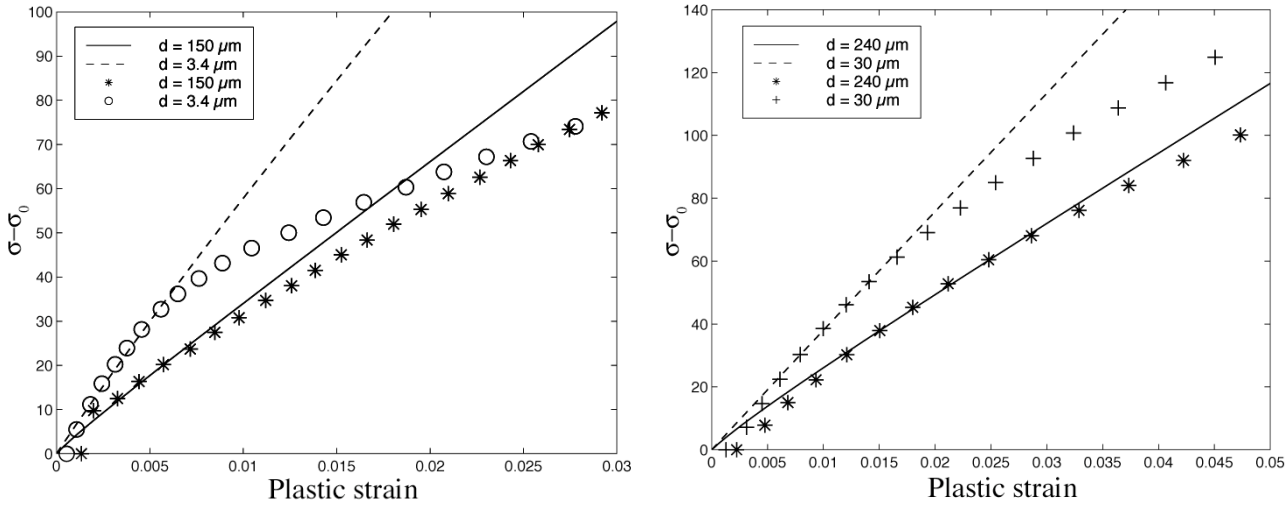


(a) (b)

Fig. 2.8. Stress strain data (from Fig. 1 in Paper 6) replotted as the strain hardening $d\sigma/d\varepsilon$ vs. $1/(\sigma-\sigma_0)$ for (a) copper and (b) Fe-0.2 w%Ti

At higher strains, i.e. the left hand side of the figures, the experimental information for the different grain sizes, fall on the same line. This latter observation is in line with our earlier reasoning, that at larger strains we have an evolving dislocation structure and dislocation-dislocation interactions will control the strain hardening behaviour.

In the second type of evaluation, the analytical solution to Eq. (2.13) i.e. Eq. (2.16), was compared with the experimental stress strain data. This is presented in the Figs. 2.9a-b. As can be seen, there is a reasonable fit of the analytical solution to the experimental data at low strains. This once again indicates the validity of the described approach. Reasonable values were used for the constants in the different expressions and the values for these can be found in Paper 6.



(a) (b)

Fig. 2.9 Comparison of stress strain data (from Fig. 1 in Paper 6) with predictions from Eq. (2.16) for (a) copper and (b) Fe-0.2 w%Ti

2.3.6 A numerical model of grain size strengthening

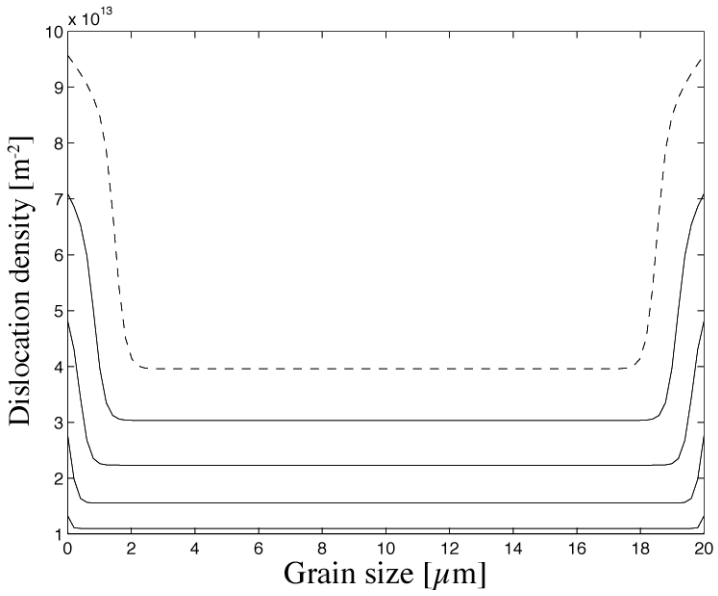
The treatment described in the previous chapter is useful when modelling the macroscopic properties, such as the flow stress, but it cannot capture local effects at grain boundaries. One interesting local feature is the evolution and propagation of gradients in dislocation density. An attempt to experimentally measure the evolving deformation structure is presented in chapter 3. In order to capture these effects, a simple, one-dimensional model was developed. The model aims to describe the evolution of dislocation density with strain and distance, within an average grain. Strictly speaking the model can be seen as an extension of the classical single parameter work hardening model that has been discussed earlier. The mathematical formulation of the developed model with a more extensive discussion of the results and a comparison with experimental data, can be found in Paper 6. Only a brief outline of the key assumptions behind the model and some results will be presented here.

The aim is to derive an expression describing the evolution of dislocation density with strain within a grain. This leads finally to the following partial differential equation, derived with the assumptions presented below:

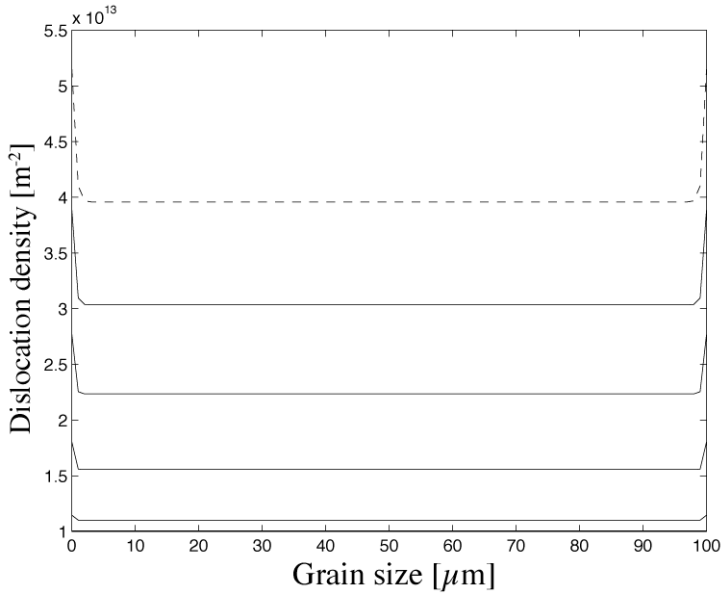
$$\frac{\partial \rho}{\partial \varepsilon} + A_1 \frac{\partial \rho}{\partial z} = \bar{m} k_2 \sqrt{\rho} \qquad (2.17)$$

- A dislocations flux is defined as a product of, $\bar{v}$, the average velocity of dislocation motion and ρ the total length of dislocations per unit volume.
- An extra contribution to the strain hardening is assumed due to the extra generation of dislocations at grain boundaries.
- Generation of dislocations within grains is accounted for by including a source term, where the strain hardening is assumed to be proportional to the square root of dislocation density.
- An explicit finite difference method using superposition was used to numerically solve the PDE in Eq. (2.17).

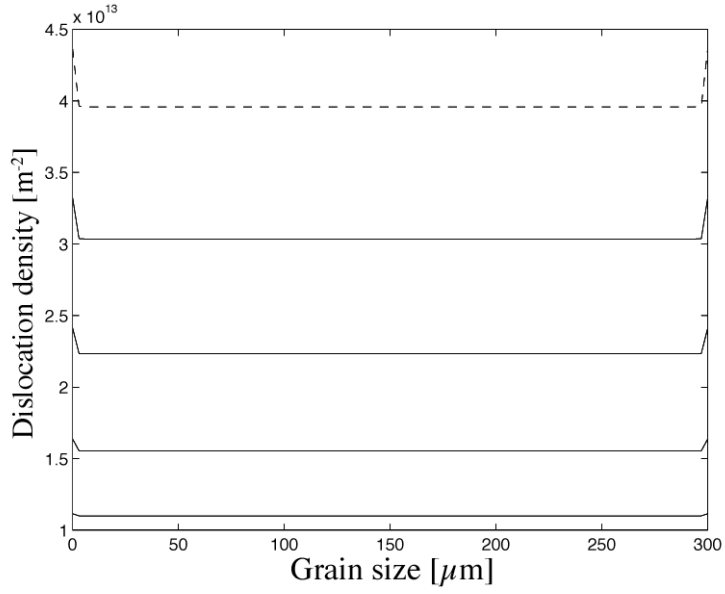
The constants in Eq. (2.17) above, together with the parameters used in the simulations are presented in Paper 6. In the Figs. 2.10a-c, dislocation density profiles within an average grain are shown, for different levels of plastic strain, up to 4 %.



(a)



(b)



(c)

Fig. 2.10 Evolution of dislocation density profiles, within an average grain, for 0, 0.2, 1, 2, 3 and 4% plastic strain when the grain size, d is (a) 20 μm, (b) 100 μm and (c) 300 μm.

As can be seen, by comparing Fig. 2.10a with Fig. 2.10c, the influence of grain boundaries on the evolution of dislocation density is much larger, when the grain size is small (d = 20 μm) compared to when the grain size is large (d = 300 μm). Also the dislocation density level is much higher for small grain sizes, as can be expected. In other words there is a greater

contribution to the strain hardening from grain boundaries. This is also in agreement with experimental measurements from the literature, of the variation of dislocation density with grain size, where fine-grained samples show a steeper increase in dislocation density with strain (Keh and Weissmann 1963; Hansen 1985). Other results from the simulations and comparisons with experiments, along with a more comprehensive discussion of the implications of the model can be found in Paper 6.

In conclusion there are a number of factors that should be included in a satisfactory model for grain size strengthening. A few points have been discussed here and some modelling ideas have been presented. The discussions here have been directed towards materials exhibiting homogenous yielding behaviour, where the presented modelling approaches seemed to provide a satisfactory explanation. In such case, the grain size strengthening at small strains seems to be controlled by grain boundaries. In the case of grain size strengthening in materials showing a sharp yield point more work is needed. One interesting area to explore is the nucleation and propagation of Lüders bands, by performing careful experiments and using new techniques like AFM and EBSD.

2.4 Precipitation Strengthening

The strengthening effect due to finely dispersed particles has been known for a long time. It is also one of the methods most extensively used in the development of higher strength in commercial alloys. A great deal of research has been published on the mechanisms and applications of precipitation hardening. The basic requirements for this kind of strengthening is that through a heat treatment (dissolution - quenching - ageing) in a suitable alloy system, end up with a microstructure consisting of finely dispersed particles in a matrix. These particles then resist the motion of dislocations, thereby increasing the strength level.

There is an enormous amount of literature on different alloy systems for precipitation and the effects of precipitation strengthening on different properties, like toughness, ductility and creep. We will concentrate on the mechanisms behind the effect of precipitation strengthening on the yield strength. Precipitation strengthening can be treated in a similar way as was earlier presented for solid solution strengthening. In fact many of the theories presented earlier were developed for strong obstacles (i.e. precipitates). Among these are the concept of the flexibility of the dislocation line and the concept of discrete obstacles. There are several ways, by which a dislocation can bypass obstacles namely,

- by shearing through them
- by bowing out between them (the Orowan-mechanism)
- by cross-slip or climb

The last mechanism is only interesting at higher temperatures and will not be discussed further here. The first two mechanisms are competing ones and are valid at different particle dispersions.

Extensive theories have been developed for a number of different mechanisms, including;

- chemical hardening
- stacking fault strengthening
- strengthening due to differences in shear modulus
- coherency strengthening (due to coherency strains around particles)
- order strengthening (due to ordered precipitates)

It must emphasised that in many cases two or more mechanisms may contribute to the strengthening (the superposition of different mechanisms will be discussed later). It is not within the scope of this short introduction to go into the details of these different mechanisms and for a more comprehensive treatment on the subject, the reader is referred to Brown et al. (Brown and Ham 1971) and Ardell (Ardell 1985).

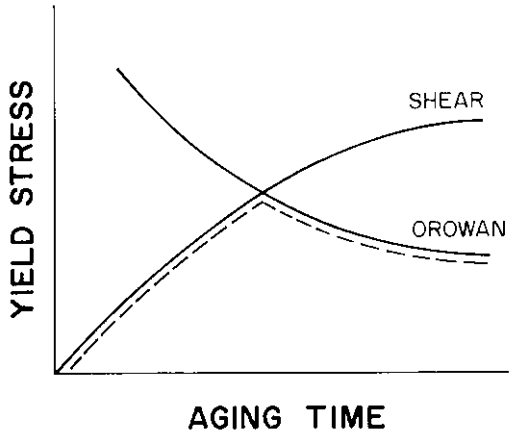


Fig. 2.11 The transition between shearing and the Orowan mechanism at a certain aging time (or particle radius) at a constant volume fraction, from (Meyers and Chawla 1984).

From the basic Orowan-equation (Eq. (2.3)), the different models for the strengthening due to precipitates, can be derived. The crucial point while modelling experimental data is to know how the particle strength F_p, varies for different mechanisms and with different particle sizes. There is also a transition between the shearing and Orowan mechanism, which depends on the strength of the particles, as is illustrated in Fig. 2.11. The other major problem is how to describe the flexibility of the dislocation line, L, or to understand quantitatively how the dislocation line interacts with particles. Independent of the model, L will be a function of the particle strength. In conclusion, a number of factors must be taken into account to

successfully describe and model the strengthening due to precipitates, the important questions to consider are listed below:

- What is the specific nature of the dislocation-particle interaction? (Orowan, order strengthening, modulus strengthening etc)

- What is the effective obstacle spacing? (obstacle distribution, different statistics Friedel, Mott)

- How to account for the influence of obstacles with different strengths? (superposition of strengthening contributions)

- How to account for the interaction with other strengthening mechanisms? (e.g. Orowan bypassing should cause an increase in the dislocation density)

Finding the answers to these (difficult) questions should help in including the important parameters in a model for precipitation hardening.

2.5 Peierls-Nabarro Strengthening

Another type of strengthening mechanism, which is important above all in bcc systems, is the resistance to dislocation motion due to lattice friction. This can be understood as the lattice resistance, when a dislocation moves in an otherwise perfect lattice, see Fig. 2.12. This resisting force, which naturally depends on the binding forces between atoms, is called the Peierls force or Peierls-Nabarro force. An evaluation of the Peierls stress depends on the actual force-distance relation between individual atoms, information which perhaps is only available through atomistic simulations. Different treatments of the derivation of the Peierls stress can be found elsewhere, but we will follow the treatment from Kocks et al. (Kocks, Argon et al. 1975) and discuss a few basic concepts. Different approximations for describing the Peierls potential (i.e. the force-separation relation) have been proposed and two important approximations are the sinusoidal potential and the anti-parabolic potential (Kocks, Argon et al. 1975). The interesting question is, what is the critical energy for the dislocation line to move between different energy configurations? This will naturally be a function of the applied stress and the process will be thermally activated.

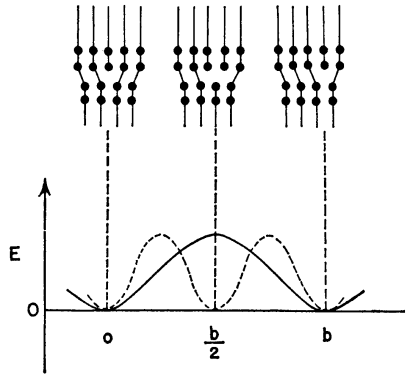


Fig. 2.12 Schematic picture of the energy of an edge dislocation core as a function of its position in the lattice showing two possible periodic variations of energy with position from (Weertman and Weertman 1964)

Different models have been proposed, for the critical configuration of the dislocation line but the most important ones are:

- the nucleation of a bulge on the dislocation line or
- the nucleation of a pair of kinks

A more detailed discussion on this subject can be found in Kocks et al., (Kocks, Argon et al. 1975) and Dorn-Rajnak (Dorn and Rajnak 1964). The activation energy for the two processes can be derived as a function of the applied stress. Interestingly enough, there turns out to be little variation in the activation energy, depending on the chosen potential (sinusoidal or anti-parabolic) or the assumed process (bulge or double-kink). Kocks et al. (Kocks, Argon et al. 1975) have proposed a phenomenological expression to summarise the influence of lattice friction, where activation energy is given as a function of the stress (shown in Fig. 2.13 below). The treatment is similar to that of solid solution strengthening. Due to the very short-range nature of the Peierls-Nabarro force, it is essentially a thermally activated mechanism. Thus from an expression for the activation energy and applying activation theory, the Peierls stress can be derived as a function of temperature. This is analogous to the case of solid solution strengthening.

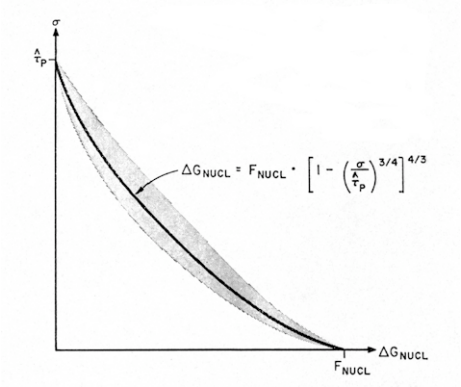


Fig. 2.13 The range of reasonable relations for the activation energy, $\Delta G_{NUCL}(\sigma)$ of nucleation over a Peierls barrier, and a central phenomenological relation. The different models fall within the shaded band, from Kocks et al. (Kocks, Argon et al. 1975).

2.6 Deformation Strengthening

As mentioned earlier, the flow stress has been found experimentally to vary with the square root of the dislocation density. We will not here go into the details of the many models for work hardening, but discuss a few basic concepts. This as an introduction to paper 3 where one simple approach is implemented into a FEM-model. There have been a number of different attempts to model the deformation hardening. Most of them starting from Eq. (2.2) presented earlier, which as mentioned before is experimentally well verified. If the flow stress is written in the following general way,

$$\sigma = \sigma_0 + \bar{m}\alpha\mu b\sqrt{\rho} \tag{2.18}$$

where ρ is the average dislocation density and $\bar{m}$ is the average Taylor factor. The σ_0 term then contains the contribution from the other strengthening mechanisms, which are assumed to be strain independent. The crucial point in different hardening models is to describe the evolution of the dislocation density with strain and this can be done in a number of ways. The evolution of the dislocation density ρ with strain ε, can for example be written in the following general way (Nes 1998):

$$\frac{d\rho}{d\varepsilon} = \frac{d\rho^{+}}{d\varepsilon} + \frac{d\rho^{-}}{d\varepsilon} \tag{2.19}$$

The first term can be seen as a measure of dislocation multiplication, while the second negative term accounts for the recovery of dislocations and becomes more important at higher strains. The dislocation multiplication term, is usually assumed to be inversely proportional to the mean free path, S, of dislocations. The mean free path S, can be related to specific features in the microstructure. For example in polycrystal fcc-materials, S can be assumed to be

proportional to the grain size at the yield strength. This assumption leads to a grain size strengthening effect, as discussed earlier, and in more detail in paper 2 and paper 6. For a pearlitic structure, S can be assumed to be proportional to the interlamellar spacing, as proposed in Ashby's original model (Ashby 1970) and this has been incorporated in a FEM-model, as presented in paper 3. Various theories (Bergström 1970; Roberts and Bergström 1973; Mecking and Kocks 1981; Bergström 1982; Kuhlmann-Wilsdorf 1985; Nes 1998; Kuhlmann-Wilsdorf 1999) have been proposed to describe and understand work hardening and most of these include the terms presented in the above expression. The different approaches lead to slightly different expressions depending on the assumptions concerning the interaction and mobility of dislocations.

2.7 Superposition of Strengthening Mechanisms

Probably the most interesting and most complex part of the modelling the strengthening mechanisms is to account for the interaction and superposition of different strengthening mechanisms. This is also the area where not much literature exists. Kocks et al. (Kocks, Argon et al. 1975) discuss the question of superposition and also present different expressions for adding different strength contributions. Brown (Brown and Ham 1971) and Ardell (Ardell 1985) also discuss these issues in the case of precipitation hardening. The most common metod is to simply add the contributions, so called linear superposition;

$$\sigma = \sigma_1 + \sigma_2 \tag{2.20}$$

Generally speaking, when the structural scales of the different contributions are not widely different, linear superposition cannot be assumed to be valid. On the other hand, when the mechanisms are of sufficiently different length scales, e.g. Peierls-Nabarro barriers and grain boundaries, where the former is on the scale of atomic distances and the latter is on the scale of micrometers, linear addition should be valid. Another way of summing the different contributions to the flow stress commonly proposed in the literature, (Kocks, Argon et al. 1975) is Pythagorean superposition or the sum of the squares of the contributions from different obstacles as:

$$\sigma = \sqrt{\sigma_1^2 + \sigma_2^2} \tag{2.21}$$

This is often used for the case with two sets of discrete obstacles of equivalent strengths, but with varying densities, (Kocks, Argon et al. 1975) as in the case of precipitation strengthening with two different types of precipitates (Ardell 1985). Compared to linear addition, where the obstacle strengths are added linearly, in the latter model the obstacle densities are added linearly. A third case of interest is two sets of obstacles of the same density, but with different strengths. A statistical treatment of this case (actually for misfitting solute atoms above and

below the slip plane) has been given by Labusch (Labusch 1970) leading to the following equation:

$$\tau = \left[\tau_1^{\frac{3}{2}} + \tau_2^{\frac{3}{2}} \right]^{\frac{2}{3}} \qquad (2.22)$$

One difficulty that arises when we have obstacles of similar strengths and both are subject to thermal activation is that small differences in the shape of their respective force-obstacle profiles can lead to large differences in the way dislocations can bypass the obstacles. Most superposition models neglect or assume that there is no interaction between strengthening mechanisms. This is not valid in many cases (Nembach 1992). For example, as a consequence of the Orowan mechanism, dislocation loops are left behind at precipitates. This should increase the dislocation density and influence the strain hardening behaviour. One can also imagine solutes influencing the mechanisms behind Peierls-Nabarro strengthening. The process of nucleation and propagation of kinks could very well be influenced by the presence of solutes.

In conclusion it can be seen that the superposition of strengthening mechanisms is not at all straightforward and a number of different aspects have to be taken into account. Unfortunately, there is no general method to approach this problem and a careful analysis has to be done for each case to find the appropriate model. There are a number of ways to evaluate experimental data in a clever way, in order to distinguish and seperate between different active mechanisms, and this seems to be one approach towards modelling the superposition (Kocks, Argon et al. 1975; Kocks 1979). Another approach is through computer simulations, on a more fundamental level, which is of course limited to relatively small and idealised systems.

Chapter 3

Experimental Techniques and Methodology

As mentioned earlier, the aim of this work is to understand and model the flow stress of metals. One important step is to understand what happens on a microstructural scale when a metal is deformed plastically. In this work, the focus is on the deformation behaviour at relatively small strains around the yield stress. All the different strengthening mechanisms we have discussed earlier (with the exception of deformation strengthening) affect only the yield stress[3]. As we have seen earlier the grain size influences the dislocation strengthening at small strains. In the following sections the experimental procedures used in this thesis will be presented and the most essential results will be discussed. A more detailed description and discussion of the experimental work and also comparison with results from a non-local crystal plasticity model, can be found in Papers 4 and 5.

3.1 Experimental procedure

The purpose of the experimental work in this thesis is to characterize the evolving deformation structure at relatively small strains. In-situ studies, where the microstructure can be characterised by appropriate experimental techniques such as, transmission electron microscopy is probably the best method. This enables a study of the dislocation dynamics and deformation mechanisms. Unfortunately, as mentioned earlier this is a demanding and expensive technique. Another method, which is employed here, is interrupted tensile testing. A polished metal surface is characterised prior to deformation and then subjected to different levels of tensile strain. After each strain level the same region in the sample (i.e. the same grains) is again examined and characterised. The different regions in the samples are identified using micro indentations as markers. In this way the evolving deforming microstructure can be monitored at small strains.

Towards this, two different surface techniques namely, atomic force microscopy (AFM) and electron back-scattered diffraction (EBSD) were applied. The focus, of the experimental work, was to study the behaviour at grain boundaries and especially the differences in behaviour between small and large grains. An important subtask is to compare the results from AFM and EBSD and evaluate they if can be used to study the deformation characteristics in a quantitative fashion, was another motivation of this study.

[3] It can be argued that precipitation strengthening should and in fact does influence the flow stress.

In the first case (Paper 4), a low carbon hot rolled steel, with an average grain size of 11 μm, was studied using AFM, after different amounts of plastic strain up to 3.4 %. The change in the measured surface profiles with strain was characterised and compared with a non-local crystal plasticity model. The details of the model used and other results from the comparisons are available in Paper 4. In the second case (Paper 5), recrystallised samples of an ultra-low-carbon (ULC) steel were characterised with AFM and EBSD. The two samples, a coarse grained (CG_3) with a grain size of 60 μm and a fine grained (FG_1) with a grain size of 14 μm were studied after different amounts of plastic strain up to 10 %. Full details about the experimental set up can be found in paper 5. A short introduction to the two techniques used and the results obtained from the measurements will be presented in the coming sections.

3.2 Electron backscattered diffraction (EBSD)

3.2.1 General description

Electron back-scattered diffraction (EBSD) is a powerful tool that is most useful in studying deformed and recrystallised microstructures is. The principle is based on the acquisition of diffraction patterns from bulk samples in a scanning electron microscope (SEM). By analyzing the Kikuchi diffraction patterns from back-scattered electrons, the crystal orientation at each measured point can be determined. Normally the measurements are conducted in a conventional SEM but nowadays a field emission gun (FEG) SEM with a much higher resolution is the preferred system. This is combined with some software to calculate the crystal orientations at each measuring point from the measured diffraction patterns. Depending on the type of problem and the accuracy needed the step size of the scan can be varied and a large enough area can be covered. The technique is extremely suitable in measuring grains/sub-grains after recrystallisation and deformation processes. A variety of different parameters can be evaluated from these kinds of measurements and an excellent review on the technique and its applications can be found elsewhere (Humphreys 2001). A number of microstructural parameters are now routinely available and used for characterisation, since many times it is superior to conventional techniques. A few useful parameters will be introduced here. One type of basic information that can be obtained from EBSD measurements are orientation imaging maps, i.e. the crystal orientation at every measured point over an area in the sample.

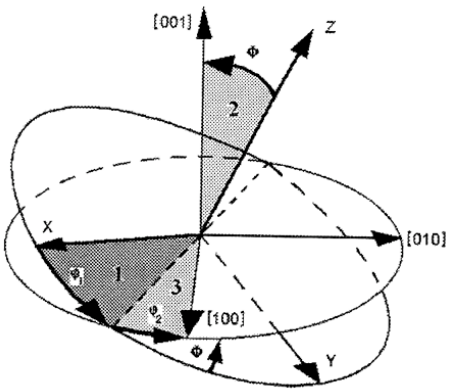
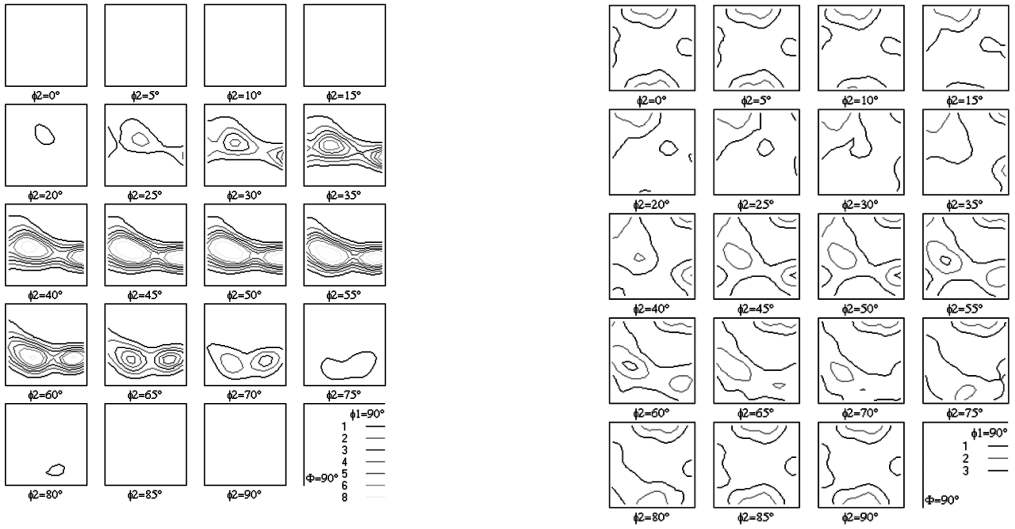


Fig. 3.1 Illustration of the commonly used convention of Euler angles, using a first rotation about the z-axis, a second rotation about the rotated x-axis and finally a third rotation about the rotated z-axis (Magnusson 2000).

In polycrystalline metal samples it is convenient to relate the absolute orientation at each point to the deformation geometry on a macroscopic scale, e.g. the geometry of a rolling process. A number of different definitions are used in the literature to represent the crystal orientations and one commonly used are the Euler angles (as shown in Fig. 3.1). To define the crystal orientation at a single point, two different coordinate systems are required one local, coinciding with the crystallographic axes and one global, for the sample. The Euler angles (φ_1, Φ, φ_2) describe the rotations needed to make the two coordinate systems coincide (Magnusson 2000). In this way a distribution of orientations or the texture in a sample can be defined. A uniform distribution of orientations corresponds to a random texture. The texture of a polycrystalline metal sample is the result of the manufacturing process and thus the texture contains information about deformation history. On the other hand the texture also has strong influence on the mechanical properties. There are a number of different ways to visualise the texture or the distribution of orientations in a material. One common method is by pole figures, where the measured distribution and intensity of important crystallographic directions and planes in a sample, are presented in a stereographic projection. Another way to summarise the overall texture, is by an orientation distribution function (ODF), which is a three dimensional mathematical function describing the intensity at each point in Euler space. Usually one displays 2D sections of the cube of Euler space, with contours showing the intensity, as shown in Fig. 3.2.



(a) (b)

Fig. 3.2. ODF:s describing the global texture for the fine-grained (a) and the coarse grained (b) sample with the <111> // ND. The α-fibre is seen in the φ_2=45 cross-section.

In this figure the ODF for an ultra low carbon steel, is shown for two different grain sizes. It can be observed that the fine-grained sample shows a much stronger texture compared to the coarse grained. In this case the former sample has undergone a much larger deformation prior to recrystallisation, which thus gives this sample a much sharper texture. The important feature in this figure is the φ_2 = 45° section, where the so-called α-fibre, typical of the recrystallisation texture in these steels, is seen. This type of texture, with the <111> direction parallel to the normal direction, is desirable since it leads to the excellent forming properties in these steels

3.2.2 Description of evaluated parameters

A region in a polycrystalline material can be characterised and different grains and grain boundaries identified in the way described earlier. In such an orientation imaging map (OIM), interesting features such as grain boundaries, sub grain boundaries and other changes in orientation can be visualised. This makes EBSD an excellent method to evaluate the average grain size. The grain sizes in the two ULC steel samples examined in this study were evaluated from EBSD measurements to be 60 μm and 14 μm respectively. The orientations in OIMs can be colour coded according to the respective Euler angles at each point and grain boundaries can be introduced as, lines between measurement points having a large enough difference in orientation. In the OIMs shown in Figs. 3.3a-d, grain boundaries are defined as black lines, with a difference in orientation (or a misorientation) of more than 10° between neighbouring measured points. Sub grain boundaries shown as white lines, correspond to a

difference between neighbouring points of more than 2° (see Fig. 3.3). Examples of such OIMs and an interpretation will be presented in the next chapter and full details are available in Paper 5.

Another interesting parameter evaluated here is the intra-grain misorientation. This is defined in the following way; after assigning orientations to all measured points and identifying grain boundaries as described previously, an average orientation in each grain can be evaluated. The intra-grain misorientation is then defined as the deviation from the average orientation, within a grain. In this way an average intra-grain misorientation value per grain, can be defined and the grains can be colour coded accordingly. Thus the intra-grain misorientation can be seen as a statistical measure of the reorientation occurring within grains. The change in the intra-grain misorientation with strain can be seen as a measure of the dislocation storage and therefore a good measure of the local deformation behaviour.

3.2.3 Results from EBSD measurements

The EBSD measurements were performed in a LEO Gemini 1530 FEG-EBSD. In the Figs. 3.3a-d, OIMs where the grains in the two ULC steel samples were colour coded according to the Euler angles, are shown after different amounts of deformation. The step size in the scans presented here, varied between 2.5 and 0.5 μm.

It can be observed in Figs. 3.3a-b that after 0.2% strain, orientations within grains are almost uniform. After 10% strain (Figs. 3.3c-d) however, there is an increase in the occurrence of other orientations (colours) within grains. This is especially visible near certain grain boundaries indicating rotations within grains. Also the large increase in the number of sub-grain boundaries can be seen, at the highest strain. This indicates an increased rotation within grains, leading to more inhomogeneous deformation. These do not however, cover entire grains but instead are predominantly present in the vicinity of grain boundaries and triple-junctions. In other words, at regions which are strongly stressed and where the generation of dislocations can be expected to be larger the rotation is larger. In Figs. 3.3e-f, average intra-grain misorientation maps, of the same regions as before, are shown after 10 % strain. The grains have been colour coded according to the average intra-grain misorientation value within each grain, giving each grain a uniform colour. It can be observed that a number of grains in the fine-grained sample show intra-grain misorientations larger than five degrees and are thus coloured grey. The average intra-grain misorientation, for each region in the respective sample, was also evaluated quantitatively at each strain level. This is shown in Fig. 3.4, where the average intra-grain misorientation shows a linear increase with macroscopic strain. This further emphasises that the intra grain misorientation can be seen as a good measure of the local deformation behaviour.

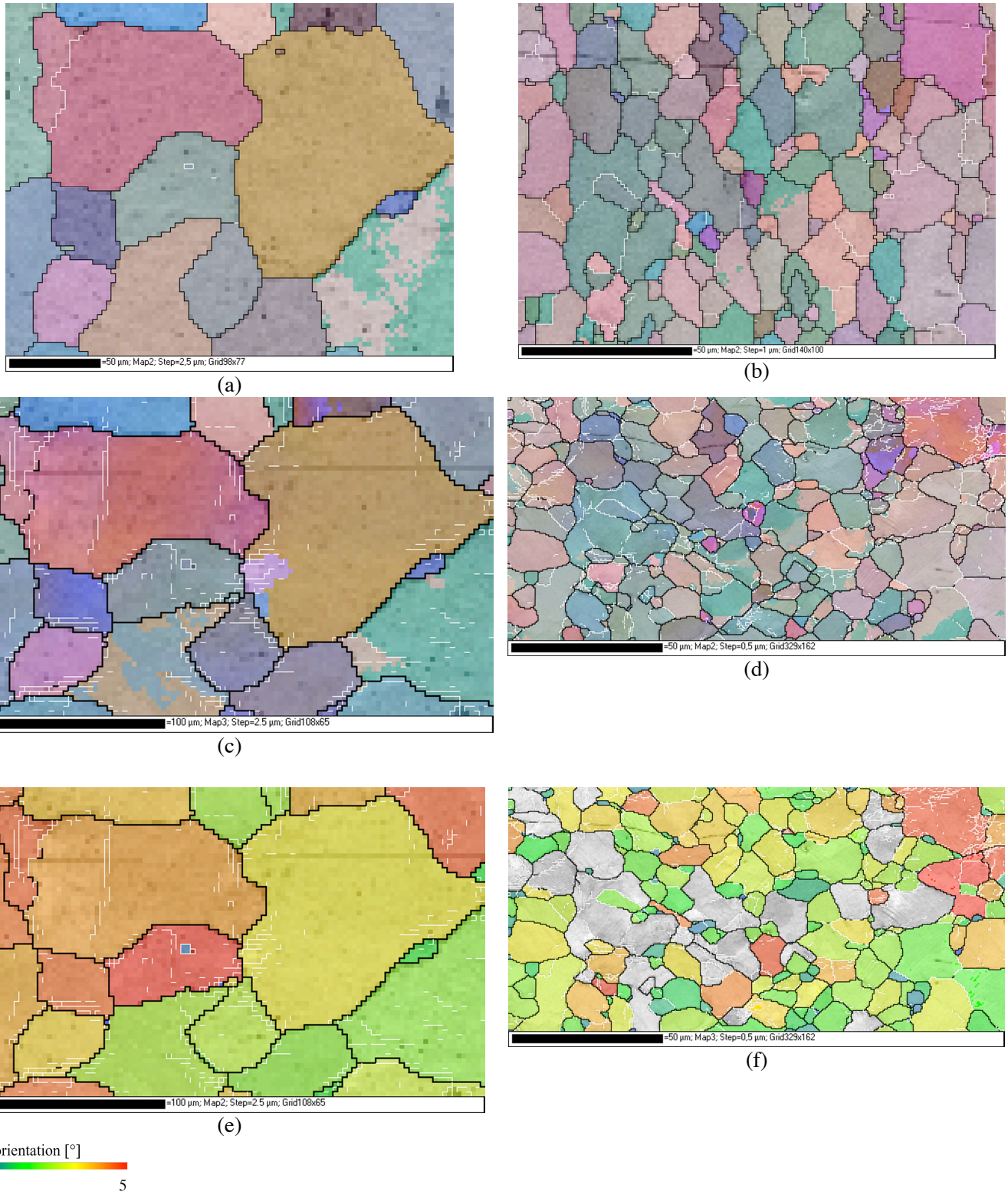


Fig. 3.3. Change in orientation with strain, colour coding defined by Euler angles, for (a), CG_3(after 0.2%), (b), FG_3(after 0.2%), (c) CG_3(after 10%), (d) FG_1(after 10%). Misorientation maps after 10% strain of (e) CG_3, (f) FG_1.

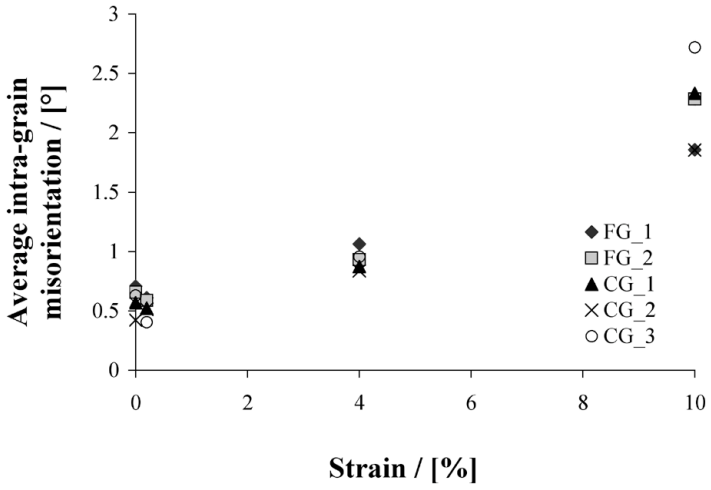


Fig. 3.4 Change in average intra-grain misorientation with strain for the different regions, in the two samples (attention should be paid to the relative change in average intra-grain misorientation not absolute values as there is a certain spread in the individual orientation measurements).

A further statistical analysis (where all grain measurements smaller than 5 pixel points were removed) of the EBSD-data showed two other interesting features. Firstly, both the average intra grain misorientation and standard deviation values of the misorientation measurements were larger for the fine-grained sample. This is consistent with the results presented in Figs. 3.3e and 3.3f, where only grains in the fine grained sample showed intra-grain misorientation values larger than 5°. Secondly, within each sample, the intra-grain misorientation was larger for large grains compared to small. This latter observation is partly an artefact of the misorientation measurements but also indicates a greater tendency for intra grain rotation and inhomogeneous yielding to occur within large grains in a sample. One possible explanation for the larger misorientation values in the fine-grained sample could be due to the extra strain hardening contribution from grain boundaries, in line with the discussion earlier in chapter 2.3.5.

3.3 Atomic force microscopy (AFM)

3.3.1 Experimental principle

Another comparatively new technique is atomic force microscopy (AFM). Since the invention in 1986, the technique has been extensively developed and is finding more and more useful applications in the field of materials science (Wittborn 2000). The principle is quite simple. The surface of the sample is probed by a sharp tip, a couple of microns long and often less than 100 Å in diameter. The tip is located at the free end of a cantilever, that is 100 to 200 μm

long. The tip can either be in contact with (Contact mode) or very near the surface (Non-contact mode). Forces acting between the tip and the surface will cause a bending of the cantilever proportional to the resultant force. The topography of the sample will influence the forces acting on the tip and thus also the deflection of the cantilever (see Fig. 3.5).

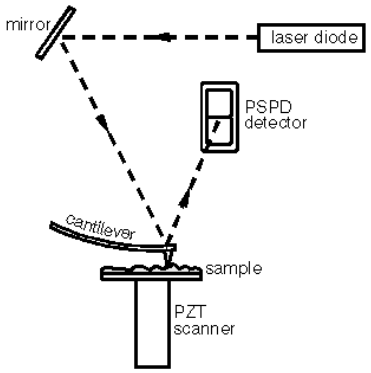


Fig 3.5 Schematic illustration of the principle of AFM. The deflection of the cantilever is monitored via a laser beam, as the tip scans over the surface.

By monitoring and measuring the deflection of the cantilever, a map of the surface topography can be generated. Usually a silicon nitride tip is used for the measurements. The great potential of AFM is the excellent lateral resolution, which can be as low as 0.2 nm, depending on the surface quality. The experimental set-up is schematically shown in Fig. 3.5.

Another advantage of AM compared to other characterization methods is that comparatively large areas, up to 75x75 µm, can be scanned.

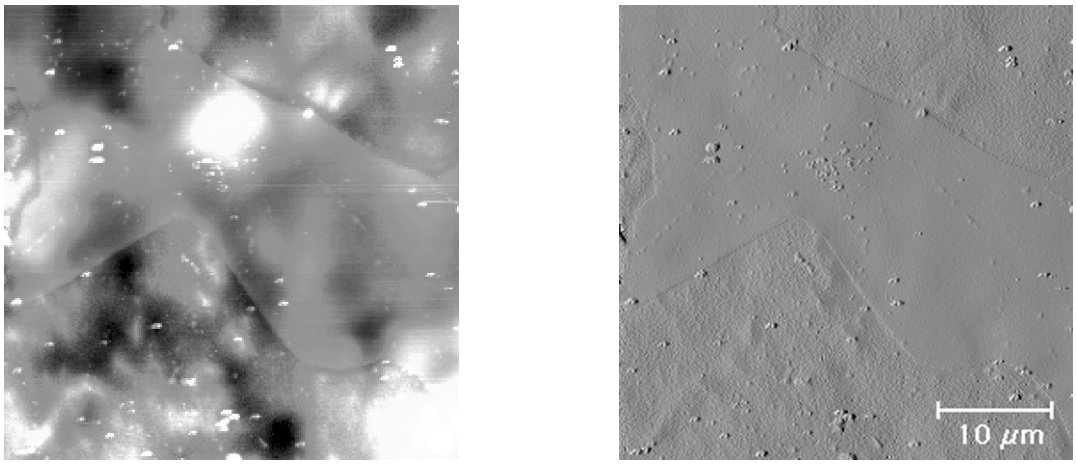
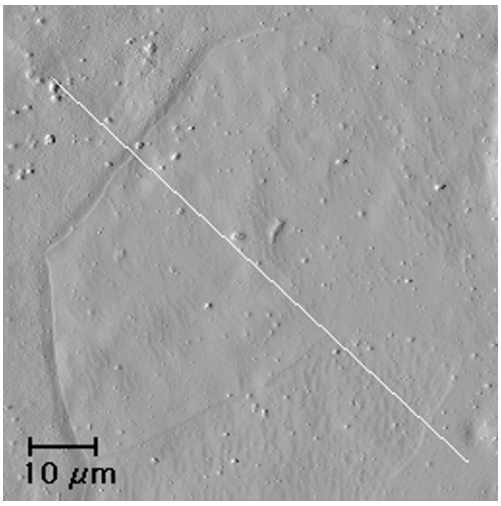


Fig. 3.6 An example of the two different images obtained from AFM, on the left hand side, the image created from the actual height measurement, on the right hand side the image created from the feedback signal. Measurement done on an ultra low carbon steel.

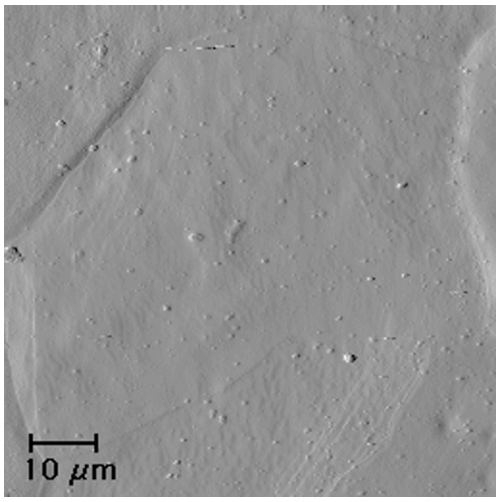
The information generated from an AFM-scan consists of two images; one is the direct measurement of the height from the sample registered by a piezo-electric tube mounted on the cantilever. The other image is generated from the feedback signal, which strives to keep the cantilever at a constant deflection. An example of the two types of images is shown in Fig. 3.6.

3.3.2 Results from AFM measurements

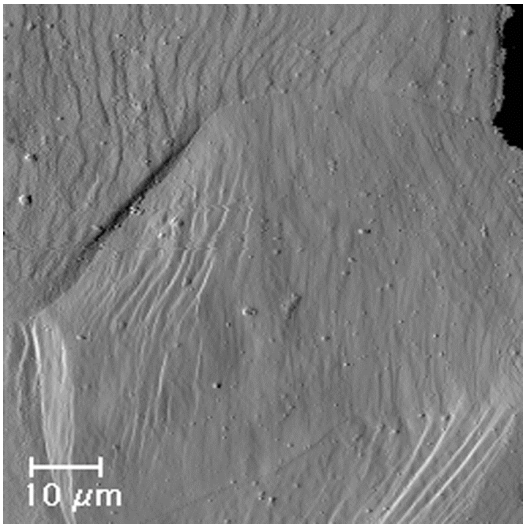
As described above, an ULC steel, with two different grain sizes was characterized with contact mode AFM (D3000 Nanoscope from Digital Instruments with a silicon nitride tip), after different amounts of plastic strain. After each strain increment the same regions in each sample were characterized by AFM. In the Figs. 3.7a - f different types of information are presented. In Figs. 3a - 3d AFM-scans from the fine grained (FG) and the coarse grained (CG) sample are shown before and after different amounts of strain.



(a)



(b)



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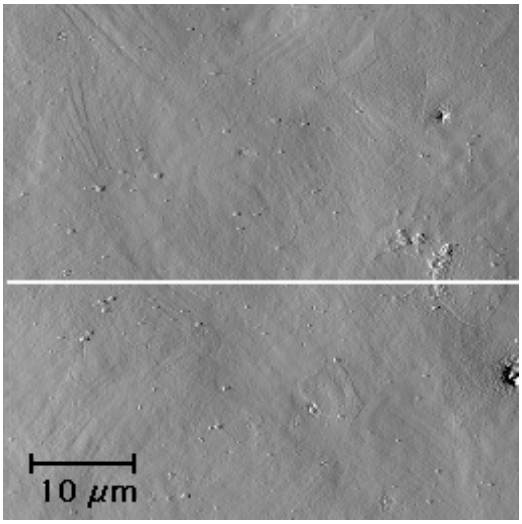
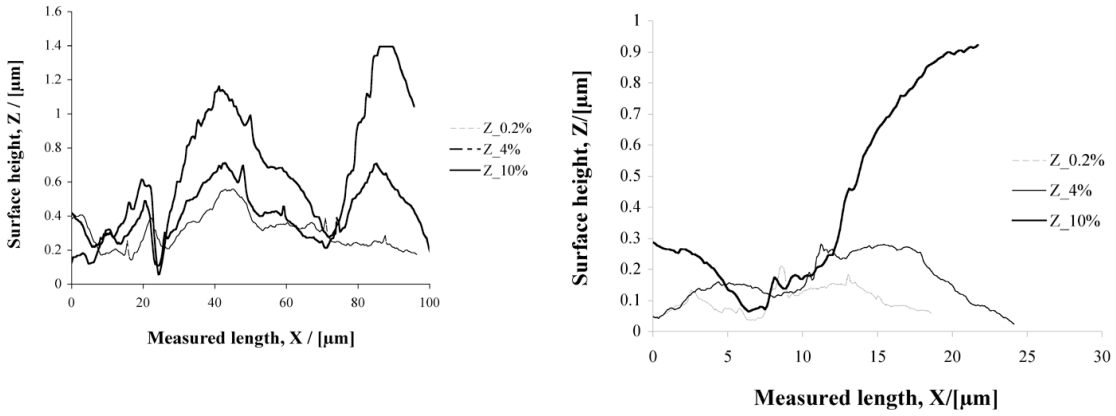


(d)



(e) (f)

Fig 3.7 Typical AFM-scan of regions in the different samples after different strains, (a) CG_3(after 0.2%), (b) CG_3(after 4 %), (c) CG_3(after 10%), (d) FG_1(after 4%). Variation of surface profiles at different strains over the corresponding lines in Figs. 3.7a - d: (e) CG-3 and (f) FG-1.

The corresponding roughness profiles over the lines displayed in Fig. 3.7, are shown in Figs. 3.7e-f. As can be observed, most changes occur in the last deformation step, as illustrated by the slip lines at grain boundaries after 10 % strain (Fig. 3.7c). The roughness profiles show, both an increase in surface height and also a steepening of gradients, with increasing strain. A more quantitative measure of the surface roughness is the standard deviation of all height measurements within a region. This is shown in Fig. 3.8, where the variation of out-of-plane displacements for different regions in the two samples, have been plotted. The standard deviation can be seen to increase fairly linearly with strain. This is also in agreement with the results from the EBSD measurements presented earlier.

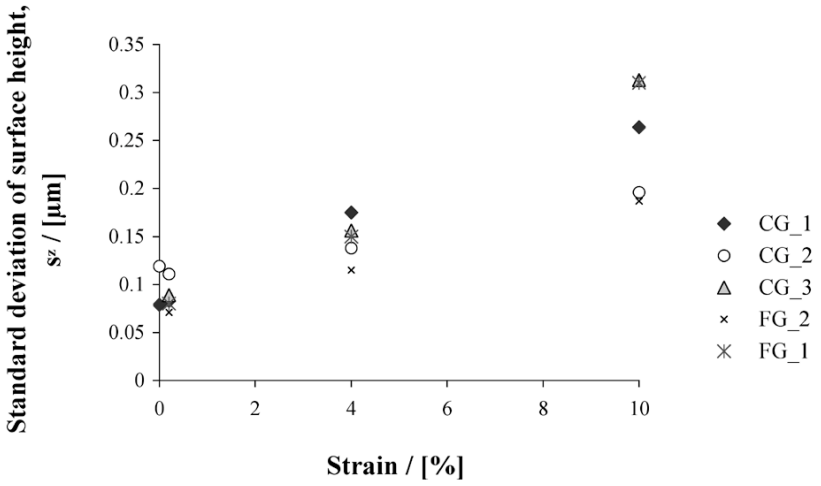
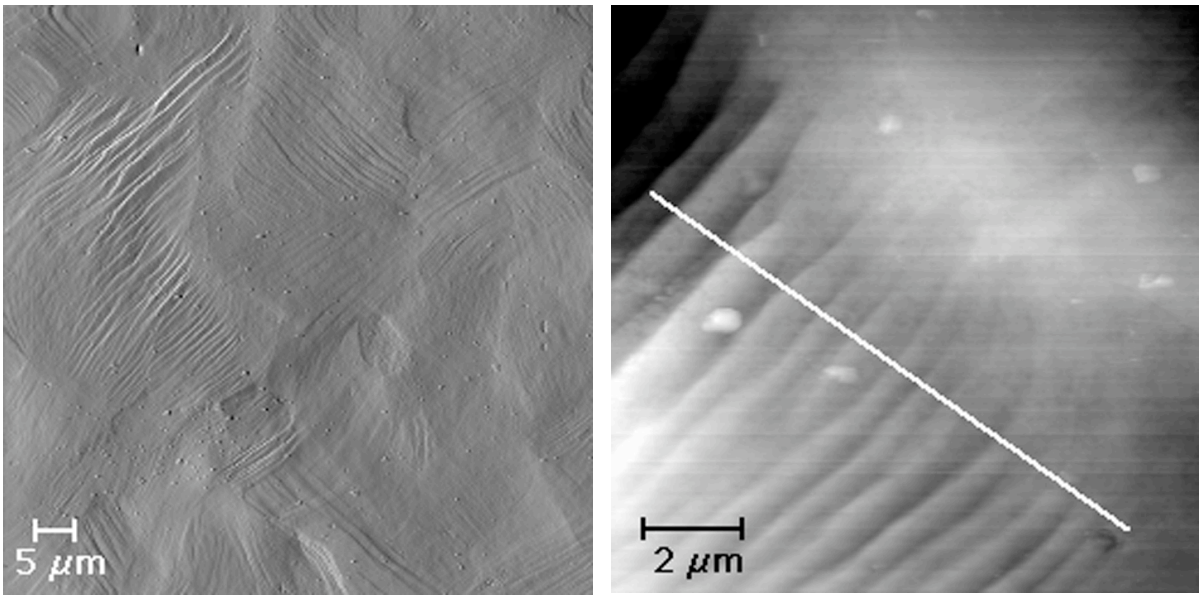
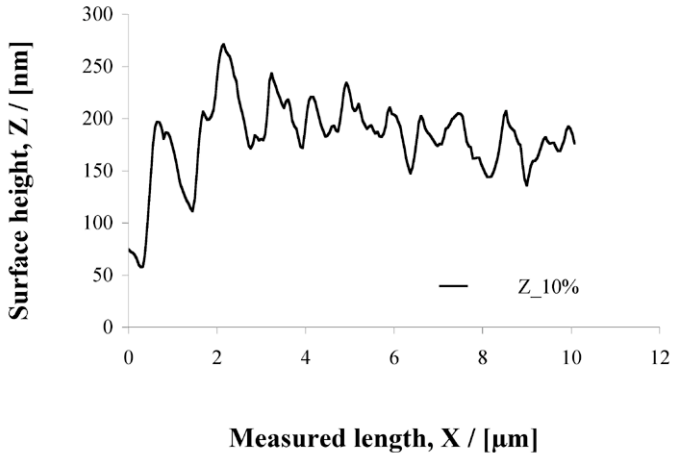


Fig 3.8 Change in overall roughness with strain, represented by the standard deviation, s_z, for the different regions in the two samples.

Another interesting feature that can be visualised with AFM is the individual slip lines, which can be measured quite accurately. In Fig. 3.9a, an overview of a deformed region in the fine-grained sample is shown after 10 % plastic strain and the slip lines can clearly be seen. At higher magnifications, individual slip lines can be resolved and measured and this is shown in Figs. 3.9b-c. The height of individual slip lines can be assumed to correspond to the number of dislocations slipping on the slip plane. In this case a step height of 50 nm, should correspond to roughly 200 individual dislocations, which can then be seen as a measure of the accumulated strain.



(a) (b)



(c)

Fig 3.9 AFM-scan of region in the fine-grained (FG) sample showing the existence and evolution of slip lines after 10% strain. (a) Overview of deformed region, (b) magnification of slip lines seen in previous scan, (c) measurement of surface profile over slip lines as shown in b.

A more thorough discussion of the AFM results and also further comparisons with the EBSD experiments and with a numerical model for grain size strengthening (presented in chapter 2), can be found in papers 5 and 6, in the appendix.

Surface profiles, from AFM measurements on a low carbon hot rolled steel, also compared well with results from a non-local crystal plasticity model. This is discussed in more detail in Paper 4.

3.4 Discussion of the experimental results and concluding remarks

The main aim in using the experimental techniques described above is to get a more complete picture of the evolution of the deformation structure at grain boundaries with strain. A few important points will be mentioned here. One obvious issue, as pointed out earlier, is that surface characterization (2D) techniques have been used to understand plastic deformation, an essentially 3D-process. The problem is to interpret the role of a free surface on the evolution of plastic deformation. It can be argued, that a free surface gives the grains on the surface, an extra degree of freedom compared to bulk grains. On the other hand it is very difficult to study the behaviour of bulk grains. Bulk methods such as TEM also have the disadvantage of only covering a small number of grains and essentially only 3DXRD gives the possibility of studying the same bulk region in a sample, during deformation. The strength of the research work presented here is that the same grains are studied after each deformation step, thereby giving the opportunity to follow the deformation behaviour on a local scale. In order to get some idea of the differences between bulk and surface behaviour, the fine grained ULC steel sample deformed to 10% strain, was polished down to a third of the original thickness and studied with EBSD. The results showed that both the average and standard deviation values, of the intra-grain misorientation, are slightly smaller compared to the earlier presented surface measurements. The order of magnitude is still the same. This seems reasonable since bulk grains should have less freedom of rotation compared to surface grains. It should be emphasised that this comparison is by no means conclusive, as the texture is not necessarily the same at the surface and in the bulk, for example. Further studies are needed to explore the differences between bulk and surface measurements.

A few words on the uncertainties and causes of error with the experimental techniques presented here. In the case of AFM the absolute limit in resolution is given by sharpness of the tip, as mentioned earlier. Other sources of error in the measurements are sample preparation and background noise in the AFM equipment used. These factors will cause a certain error when measuring absolute values of surface height and finer details in the microstructure. In all the measurements presented here only changes in surface height were of interest and the features studied are on a much larger scale (hundreds of nanometres) than the limitation of the technique (a few nanometres). In the case of EBSD, there is an uncertainty of

± 0.5°, at each measured point for the absolute orientation value and misorientation values below 1.5° are not reliable without proper filtering of the data points. As in the case of AFM, the results presented here consist mainly of relative change in orientation and not absolute values and therefore the possible errors in the measurements should be negligible.

In conclusion the experimental work in this thesis has been concentrated on the behaviour of grain boundaries during plastic deformation. The two methods employed here, AFM and EBSD, correspond well with each other, both on a local scale and on a global scale. The results indicate the occurrence of significant out-of-plane rotation. Both the surface roughness and the average intra grain misorientation showed a linear increase with strain. The surface behaviour should thus reflect the overall deformation behaviour. The results also generally indicate more inhomogeneous deformation behaviour in large grains, although there are more grains in the fine-grained sample with large intra-grain misorientations. This latter effect is perhaps due to the greater strain hardening observed experimentally, when the grain size is smaller.

Chapter 4

Summary of appended papers

Paper I

Solid Solution hardening - a comparison of two models

In this paper, two different approaches to model solid solution strengthening, namely a discrete-obstacle model and a collective model, are compared. In the former model, interaction between a dislocation and a single solute is considered, while in the latter, the dislocation line interacts collectively with a row of solutes. Model predictions are compared with experimental data for Cu-Mn and Nb-Mo single crystal systems and a Ni-C polycrystal system. The collective model gives better results for the two fcc systems while the bcc system cannot be explained well by either model. The limitations and modelling capability of the two approaches are discussed with respect to the experimental information.

Paper II

Grain Size Strengthening in Polycrystals

This paper deals with grain size strengthening and focuses on the different models proposed in the literature to describe the experimental information for different alloys. The strengthening effect of grain boundaries is well established and observed experimentally as the Hall-Petch relationship. In this paper different mechanisms proposed in the literature to explain the observed Hall-Petch effect are reviewed critically. The fundamental implications of the different approaches are discussed with reference to experimental data for two different classes of materials;

- Materials with locked dislocations, i.e. with a sharp yield point behaviour.
- Materials without locked dislocations, i.e. with a smooth yielding behaviour.

It is shown that a simple model (Bergström) can be used to understand the grain size strengthening in the latter class of materials while more work is needed to quantitatively understand the behaviour of materials showing a sharp yield point.

The present author performed the work and wrote the paper, supervised by Kjell Pettersson.

Paper III

Micromechanical Modelling of Two-Phase Steels

An important issue, when modelling the flow stress of commercial materials, is how to include an appropriate microstructural length scale within a continuum framework. An attempt is made in this paper to address this question for the case of a ferritic/pearlitic steel. A two-dimensional micromechanical model based on the finite element method is presented to model two-phase ferritic/pearlitic steels, with the aid of generalised plane strain elements. A periodic representative cell containing 100 ferrite grains, and the desired pearlite fraction is used. Simulation of the loading, by an average stress or strain state, is possible by applying periodic boundary conditions. Uniaxial tensile tests were performed on specimens containing the ferrite and pearlite microstructures and on two-phase materials containing 25% and 58% pearlite respectively. The stress-strain data of the pearlite material is used to fit a lamellae dependent Taylor relation to represent the work hardening. Thereafter, lamellar spacings in the two-phase materials were measured and the total stress-strain response of the materials was modelled. Comparisons between generated data and experiments show good agreement up to a strain of 2%.

The present author performed the experimental work and contributed to the discussion.

Paper IV

Comparison of Surface Displacement Measurements in a Ferritic Steel using AFM and Non Local Crystal Plasticity

This paper deals with another important aspect in the development of continuum models with a length scale namely, the lack of reliable experimental information on a microstructural level. An attempt to experimentally study the deformation characteristics around grain boundaries and to analyse the presence of strain gradients is presented. The evolution of surface profiles is studied by atomic force microscopy (AFM) at relatively small strains. The results indicate that this method can be used to draw conclusions about the deformation characteristics. For example, in large grains the surface profile seems to vary within a grain. This latter effect can be seen as an indication of the inhomogeneous deformation occurring within large grains. The results are also compared with FEM calculations using a non-local crystal plasticity theory that incorporates strain gradients in the hardening moduli.

The present author performed the experimental work and wrote the paper together with Mikael Nygårds.

Paper V

A Study of the Surface Deformation Behaviour at Grain Boundaries in an Ultra Low-Carbon Steel

In light of the results from papers 3 and 4 there was a need to further investigate the evolving deformation behaviour at grain boundaries, especially at relatively small strains An important question was if out-of-plane rotation is associated with changes in surface roughness.

In this paper, tensile specimens of ultra low-carbon ferritic steel with two different grain sizes are studied by AFM and EBSD after different plastic strains up to 10%. Different parameters such as the change in surface roughness and the change in misorientation, with strain are evaluated. There is good agreement between the AFM and EBSD results. Both the surface roughness and the misorientation measurements on the surface, show a linear increase with the overall strain. An obvious conclusion is that both AFM and EBSD are suitable for the characterising the surface deformation behaviour. Inhomogeneous features are more predominant in large grains although there are more grains in the fine-grained sample with large intra-grain misorientations. It can also be concluded that significant out-of-plane rotation is consistent with plastic deformation at small strains. The results are discussed with respect to the difference in grain size in the samples and the implications on the strain hardening behaviour.

The present author performed the experimental work and wrote the paper together with Mikael Nygårds.

Paper VI

Grain Size Strengthening at Small Strains – Analysis of Experimental data and Modelling Implications

This paper again deals with grain size strengthening, specifically focusing on materials showing a homogenous yielding behaviour. As was shown in paper 2, a relatively simple model can be used to describe the grain size strengthening at the yield stress, for this class of materials. An interesting observation from the experimental information is the grain size strengthening at higher strains. This feature has not been captured satisfactorily in previous models, as discussed in paper 2.

In this paper an attempt is made to understand the grain size strengthening observed experimentally, in two different materials exhibiting a homogenous yielding behaviour. A critical analysis of the experimental information, using a classical single parameter work hardening model, is presented.

The results from this analysis show, that the strain hardening at small strains, is controlled by the grain size. At larger strains the hardening behaviour is controlled by the inherent dislocation structure.In order to capture local features at grain boundaries, a simple, numerical model was developed. The numerical model gives satisfactory results in a qualitative comparison with results from atomic force microscopy (AFM) and electron backscattered diffraction (EBSD) measurements.

The present author performed the work and wrote the paper, supervised by Göran Engberg.

Chapter 5

Conclusions and Future work

Understanding the mechanisms behind the strengthening in metals is crucial in the development of new materials with better mechanical properties. A systematic analysis of different mechanisms and how they depend on external variables like temperature and strain rate combined with experimental work on the evolution of plastic deformation at small strains has been the focus of this work. Two strengthening mechanisms namely, solid solution strengthening and grain size strengthening have been dealt with in detail. In both these cases existing models have been reviewed with aspect to their predicting capability and physical meaning. In the case of grain size strengthening the experimental work has been focused on the behaviour of grain boundaries during plastic deformation, at relatively small strains. A first attempt has also been made to include microstructural features in continuum plasticity models.

5.1 Solid solution strengthening

- Collective models give a better description of the experimental data compared with the discrete-obstacle approach, especially for the fcc-alloys studied.

- A complete description of solid solution strengthening requires a model that can incorporate size/modulus effects with the collective overcoming of solutes.

5.2 Grain size strengthening

- The models reviewed in this thesis do not give a good explanation of the grain size dependency in materials showing a sharp yield point behaviour.

- A simple model with an easy physical interpretation can be used to model the grain size strengthening in yield stress of pure fcc-metals.

- A classical single parameter work hardening model gives a reasonable description of the grain size strengthening observed at small strains, in materials showing a homogenous yielding behaviour. The results further indicate that the strain hardening is controlled by grain boundaries at relatively small strains.

- Experimental results from AFM and EBSD measurements show a good correlation with each other. In both cases the surface roughness and the average intra-grain misorientation respectively, increase linearly with overall strain.

- The experimental results also show that inhomogeneous features are more predominant in large grains although there are more grains in the fine-grained sample with large intra-grain misorientations. This last fact emphasises the stronger strain hardening observed in fine-grained samples.

5.3 Flow Stress modelling

The combination of theoretical analysis with experimental observations presented in this thesis is valuable for the understanding of the mechanisms behind the strengthening in metals. The experimental techniques used here correlate well with each other and with theoretical and numerical results. The theoretical modelling was focused on analytical models and their limitations in comparison with experiments.

Another important part of this project has been focused on the development of continuum models that include relevant microstructural features. The main results from this work are:

- The inclusion of pearlite lamellae spacing in a micromechanically based FEM-model for the flow stress of ferritic-perlitic steels.

- A good qualitative agreement was obtained between experimental results from AFM and FEM calculations using a non-local crystal plasticity theory that incorporates strain gradients in the hardening moduli.

5.4 Future work

As in all scientific research this thesis has raised more questions than answers. However, one aim of good research should be to pose relevant questions in such a way, that answers can be found. For the future the following tasks are suggested.

Solid solution strengthening has been reviewed in this thesis and existing models were found unable to explain the experimentally observed plateau in yield stress-temperature curves. Moreover, further work is needed to understand the strengthening in bcc-metals.

1. Development of a model for solid solution strengthening that can explain the concentration dependant plateau in the yield stress at higher temperatures.

2. Use of ab-initio methods to model the interaction of a dislocation with solutes. This is the only way to get realistic expressions for the activation energy in solid solution strengthening

3. Understand the interaction of solid solution strengthening with other strengthening mechanisms like Peierls-Nabarro and grain size strengthening in bcc-metals by modelling the interaction of a dislocation line interacting with different kinds of short-range and long- range obstacles.

As concluded in this thesis more work is needed to fully understand the grain size strengthening in materials having a sharp yield point i.e. inhomogeneous yielding due to the propagation of Lüders bands. In the future it would be most interesting to experimentally study the propagation of Lüders bands in low-carbon steels, with techniques such as AFM, EBSD and TEM, in order to answer the following questions:

1. Is there a crystal orientation effect in the propagation of the bands?

2. What is the experimental evidence of dislocation pile-ups and other more complicated dislocation configurations?

3. What are the exact mechanisms by which the grain size and carbon content influence the nucleation and propagation of Lüders bands?

A big part of this thesis has been concerned in understanding and quantifying the deformation behaviour at small strains. In this respect AFM and EBSD have been used with good results. For the future there are possibilities to develop this still further and use other novel experimental techniques. A few interesting ideas are:

1. Use of EBSD, with a very small step size, to characterise the evolution of the deformation structure at even smaller strains. The higher resolution should enable a more detailed study of the evolving substructure in very fine-grained materials.

2. Further explore the possibilities of AFM perhaps combined with magnetic force measurements (MFM) to map the local strain on a microstructural scale.

3. Use new techniques like in-situ 3DXRD to follow the deformation characteristics in 3D.

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