Doctoral Thesis

Fatigue behaviour of AISI 316L
Mechanical response, microstructural evolution, fatigue crack propagation, & physically-based constitutive modelling

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FATIGUE BEHAVIOUR OF AISI 316L:
Mechanical response, Microstructural evolution, Fatigue crack propagation, & Physically-based constitutive modelling

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presented by
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Abstract

Demands to improve the efficiency, performance and reliability of mechanical components are driven by economic and environmental aspects, i.e. reducing costs and CO₂ emissions. Efforts to achieve these requirements involve: 1) reducing the existing level of conservatism in mechanical design, and 2) improving the capability in the component lifetime assessment. There is therefore a keen interest to obtain a comprehensive understanding of mechanical behaviour of materials, and develop an effective and reliable constitutive model to accurately describe the history dependence of mechanical response under service condition. To meet the requirements, a collaboration between three Swiss institutions (Empa, ETHZ, and PSI) and three industrial partner (ABB, Alstom, and Swissnuclear) was established with the support of the Competence Centre for Materials Science and Technology (CCMX). The present study dealing with an engineering steel (AISI 316L) has been mainly carried out at High Temperature Integrity Group (HTIG) of Empa Dübendorf. This study systematically investigates the microstructural evolution, and quantitatively relates it to internal stresses which characterise the cyclic flow stress response of the material. Established relationships between microstructural evolution and internal stresses provide a foundation to successfully develop an evolutionary constitutive model which 1) accurately describe the cyclic elasto-plastic deformation response of the material, (2) is capable of predicting the material cyclic deformation behaviour for different boundary conditions. Finally, an effort was also made to understand the relationship between the fatigue damage development and microstructural condition in order explain why there is a reduction in fatigue life of AISI 316L at 300°C relative to equivalent conditions at 20°C.

This dissertation is organised into eight chapters. Chapter I briefly reviews the history of fatigue which directly leads to the motivation of this PhD study. Chapter II provides the fundamental knowledge of defects in crystal materials. A review of the existing knowledge of the development of dislocation condition of face-centred-cubic materials during cyclic deformation is briefly given in this chapter. In Chapter III, the mechanical response during cyclic loading of the material is comprehensively characterised. The change in maximum stress, effective stress and back stress are quantified in order to obtain deeper insights into
the material cyclic deformation response. Special attention on the analysis of the complex material response at 300°C is given and discussed to examine the change in the manifestation of dynamic strain ageing during cyclic loading of AISI 316L. The knowledge obtained from this chapter together with the understanding of the microstructural evolution (which is given in Chapter IV) contributes to an understanding of physical-underlying mechanisms responsible for cyclic deformation response at 20 and 300°C. Chapter IV focuses on the systematic characterisation of microstructural condition during cyclic loading at both 20 and 300°C. The strain amplitude/temperature dependences of microstructural evolution are also described. The microstructural characterisation shed light on the origin of the material cyclic deformation response, in particular the complex response which associates with a strong short-range interaction between point defects and dislocations at 300°C. The exhaustive investigations of mechanical response and microstructural evolution provide the basis to relate internal stresses to dislocation condition. The relationship between internal stresses and microstructural evolution is thereby established, and shown in Chapter V. An evolutionary constitutive model of internal stress variables representing the history dependence of material cyclic response is developed on the basis of the obtain knowledge. The model development is first given in Chapter VI. The descriptive and predictive capabilities of the model are then discussed to reveal the effectiveness and the limitation of the model. The role of microstructural condition on the fatigue damage, e.g. crack initiation and fatigue crack propagation, and the characteristics of fatigue crack propagation are finally discussed in Chapter VII in order to thoroughly understand the fatigue damage development of the material. Important remarks on the material cyclic deformation response and fatigue damage are summarised in the last chapter, Chapter VIII.
Zusammenfassung


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Terminology

\( a \)  
The lattice parameter of AISI 316L

\( a \)  
Fatigue crack length

AISI  
American Iron and Steel Institute (Material grading system)

\( b \)  
Magnitude of Burgers vector

BF  
Bright field

CBED  
The convergent-beam electron diffraction

\( C_{\text{inter}}/s_{\text{inter}} \) and \( C_{\text{intra}}/s_{\text{intra}} \)  
Magnitude of inter-granular and intra-granular back stress during a cycle

\( C_{s,0} \) and \( C_s \)  
The nominal concentration and the saturation concentration of a type \( s \) of solute atom along dislocation lines

\( da/\ dN \)  
The fatigue crack propagation rate

\( D_g, d_w, \) and \( d_c \)  
Grain size, wall thickness, and cell size (i.e. channel width), respectively

\( D_{v,i}^* \) and \( D_0^{i=p=0} \)  
Mobility of point defects under conditions of with and without pressure

DF  
Dark field

DSA  
Dynamic strain ageing


\( E, \Delta E_{c-t} \)

Apparent elastic modulus, and the absolute difference between the \( E \) of during reverse loading transients from compressive and tensile peak stresses.

**ECC**  
Electron channeling contrast

\( E^\ast_v, i, \Delta E^\ast_v, i \)

The activation energy for diffusion of vacancy (or interstitial atoms), and the change in \( E^\ast_v, i \) (J/mol)

\( F, \Delta F \)

Helmholtz free energy, and the change in \( F \) (J/mol)

**FCC**  
Face-centered cubic

\( f_b, f_w \)

The area fractions of boundary dislocations, and dislocation walls

\( f_j \)

Fraction factor

\( g \)

The reciprocal vector of reflexion

\( g_{\text{inter}}, g_{\text{intra}} \)

Proportional to the coefficient characterising the annihilation of dislocations relating to inter- and intra-granular back stresses during plastic deformation

\( G, \Delta G \)

Gibbs free energy, and the change in \( G \)

**HCF, and LCF**  
High cycle fatigue, and Low cycle fatigue

**Hysteresis loop**  
A closed loop of stress-strain response during one cycle

\( J \)

The J-integral

\( K, \Delta K \)

Stress intensity factor, Cyclic stress intensity factor

xxvi
\[ k = 1.38 \times 10^{-23} \]

Boltzmann constant [J/K]

\[ k_1, \text{ and } k_2 \]

Coefficients characterise the process of dislocation storage and dislocation annihilation during plastic deformation.

\[ \frac{k_{1-b}}{k_{2-b}} \text{ and } \frac{k_{1-i}}{k_{2-i}} \]

The square root densities of boundary dislocations and interior dislocations at the end of the cyclic hardening stage, respectively.

\[ \frac{k_{1-j}}{k_{2-j}} \]

The square root density of dislocation sessile junctions at stabilised condition.

\[ k_{2-b}, k_{2-i} \text{ and } k_{2-j} \]

The rates of annihilation of dislocations, boundary dislocations, interior dislocations and sessile junctions with respect to \( p \), respectively.

\[ k_{2-w} \]

A coefficient characterises the rate of the formation of dislocation walls.

L

Dislocation mean free path

LCF

Low cycle fatigue

LD

Loading direction

M

Taylor factor

Max DSA transient

The maximum value of stress drop during the serration

\[ m, n \]

Representing the shape of obstacles

N, and \( N_{2\%} \)

Number of cycles, and number of cycles to 2\% of maximum stress drop, respectively
\( p \)  
Hydrostatic pressure

PSBs  
Persistent Slip Bands

\( p, p_{hi}, p_s, \) and \( p_c \)  
The accumulated plastic strain, plastic strain accumulated during the primary cyclic hardening and softening stages, and the plastic strain accumulated to form the well-developed corduroy structure, respectively

SADP  
Selected area diffraction pattern

Serration length  
A period during which the serration of flow stress happens

SFE  
Stacking fault energy

SODS  
A self-organised dislocation structure

\( S_{r,i}^*, \) and \( \Delta S_{r,i}^* \)  
Entropy of point defects, and the change in \( S_{r,i}^* \)

\( T \)  
Temperature \([\degree C, \text{ or } K]\)

TEM  
Transmission electron microscope

\( t_r, \) and \( t_a \)  
Arrest time, and ageing time

\( \bar{v} \)  
The average speed of mobile dislocations

\( V_H \)  
Loop shape parameter

\( V_{r,i}^*, \) and \( \Delta V_{r,i}^* \)  
Activation volume for the formation and migration of point defects, and the change in \( V_{r,i}^* \)
$X, \, X_{\text{inter}}, \text{ and } X_{\text{intra}} \quad \text{Back stress, Inter-granular back stress, and Intra-granular back stress}$

$Y \quad \text{Crack geometrical factor}$

$\text{WB} \quad \text{Weak beam}$

$W \quad \text{is elastic strain energy density field}$

$\alpha \quad \text{A geometric constant of the elastic interaction of dislocations}$

$\varepsilon_e, \varepsilon_p, \varepsilon_p^a \text{ and } \varepsilon^a \quad \text{Elastic strain, plastic strain, plastic strain amplitude, and total strain amplitude, respectively}$

$\varepsilon, \gamma, \text{and } \gamma_0 \quad \text{Strain rate, shear strain rate, and reference shear strain rate}$

$\eta \quad \text{is pre-factor of } \hat{\tau}, \text{ which relates to the strength of obstacles}$

$\gamma_p, \gamma_p^a \quad \text{Shear plastic strain, shear plastic strain amplitude}$

$\kappa \quad \text{Geometrical factor of dislocation cell structures}$

$\mu \quad \text{Shear modulus}$

$\nu \quad \text{Debye frequency, taken as } 10^{12} \text{s}^{-1} \quad [1]$ 

$\Omega \quad \text{Atomic volume}$

$\rho_t, \rho_b, \rho_{I}, \rho_{j}, \rho_{w}, \quad \text{The density of total dislocations, boundary dislocations, interior dislocations, dislocation sessile junctions, wall dislocations, and channel dislocations, respectively}$

$\rho_c$
\[ \rho_{\text{GNDs-inter}}, \text{and} \ \rho_{\text{GNDs-intra}} \]

GNDs are required to accommodate the plastic strain incompatibility between grains, and between dislocation high/low density regions

\[ \sigma^{\text{ext}}, \sigma_0, \sigma_{\text{max}}, \text{and} \ \Delta \sigma \]

External stress, yield strength (limit of proportionality), maximum stress, and stress range

\[ \sigma_E, \sigma_E^0, \text{and} \ \sigma_E^R \]

Effective stress, initial effective stress, and the increment of effective stress

\[ \tau, \hat{\tau} \]

Shear stress, and shear yield strength of materials at 0K

\[ \theta \]

Cell-to-cell misorientation

Superscripts or subscripts

\textit{ext}: external

\textit{gen}, and \textit{ann}: generation, and annihilation

\(i\) and \(v\) stand for interstitial atoms and vacancy, respectively

\(h1, h2,\) and \textit{soft}: the primary, secondary cyclic hardening, and the cyclic softening, respectively.

\textit{hard, soft,} and \textit{midlife}: hardening, softening, the mid-life cycle.

\textit{mo,} and \textit{im}: mobile and immobile.

\textit{peak, hom,} and \textit{sat}: the values of parameters at conditions of: the end of cyclic hardening stage, quite homogeneous distribution of dislocations over grains, the stabilised response condition.

\textit{s}: Solute atoms (Carbon or Nitrogen).

\(t,\) and \(c\): Reverse loading from tension and compression peak stresses.
Chapter I: Introduction
I.1. Brief historical review of fatigue

In this part, a very brief historical review of the fatigue of materials is given. This review does not aim to cover every breakthrough work of many distinguished engineers or scientists. It is made to give straightforward information about the significance of the fatigue on civil applications, as well as to lead the motivation of this PhD work. To get detailed informative description of the history of fatigue, the reader can refer to comprehensive reviews of Schütz, Schijive and Suresh [2-4].

Fatigue failure is one of the most common sources of failure of engineering structures. It is said that about 90% of the catastrophic failures of mechanical/structural systems of civil applications are associated with fatigue damage. Since the first study of metal fatigue which is attributed to a German engineer, W. A. J. Albert in 1829, the study of fatigue behaviour of metallic materials has been increasingly expanding over more than 180 years. The significance of fatigue failure has been greatly appreciated, in particular in fatigue-critical applications where catastrophic failures cause the loss of lives, e.g. aircraft, and nuclear power plant industries. Some well-known examples highlight the importance of the study of fatigue damage on the deterioration of structural integrity of mechanical/structural systems:

(1) The railway accident in Versailles, France in 1842 was the first acknowledged fatigue failure of civil application which cost the loss of human life. The fracture of a railway axle during repeated stress loading was the cause of the accident (a detail description is given by Smith and Hillmansen [5]).

(2) In the incidence of a series of accidents of the Comet airplanes in 1950s, there was an exhaustive examination done by the Royal Aircraft Establishment in England to find out the cause of those accidents. The investigation drew the attention of the public and scientific community to the significance of fatigue failure on the mechanical design of airplanes. Repeated stress cycles due to the pressurisation and depressurisation of the passenger cabin during taking off and landing stages was responsible for cracking of the Comet airplanes, leading to the several accidents of the airplane.

The word “fatigue” was introduced during the 1840s and 1850s to describe the failure occurring from the repeated stress loading. A great effort to investigate the fatigue failure of
railway axles under repeated stressing far below the static strength was systematically made by August Wöhler in the period of 1850-1870 in Germany. Wöhler in his lengthy paper presented the relationship between stress and life to indicate that: (1) higher stress amplitudes resulted in lower fatigue lives, and (2) there was a no fatigue fracture below a certain stress amplitude, i.e. fatigue endurance limit (The concept of fatigue endurance limit was accepted until the middle of the 20th century. Significant advances in the study of ultra-high cycle fatigue however indicates that there is no such a limit existing [6]). The Wöhler study is however still of great importance to successive studies of fatigue, and to modern mechanical design against fatigue damage. Amongst many excellent studies which aim to empirically establish the relationship between stress range (or strain range) and fatigue life, there are two well-known relationships: one is the log-log stress range (Δσ) versus life (Nf) relationship proposed by Basquin in 1910 [7],

\[
\frac{\Delta \sigma}{2} = \sigma_f (2N_f)^b
\]  

(1.1)

and the other one is an empirical relationship between plastic strain amplitude and fatigue life, which was independently suggested by Coffin and Mason in 1954 [8, 9].

\[
\frac{\Delta \varepsilon_p}{2} = \varepsilon_f (2N_f)^c
\]  

(1.2)

Where: \( \sigma_f \), \( b \), and \( \varepsilon_f \), \( c \) are the coefficient, and the exponent of fatigue strength; and the ductility coefficient, and the exponent of fatigue ductility. The four parameters are defined from the total strain – fatigue life diagram. The two empirical relationships laid down the basis for the stress-life approach (the former relationship), and for the strain-life approach (the latter relationship) of fatigue design in the 20th century.

The mechanism of fatigue failure was still not understood until a noteworthy study of the metallurgical change during rotating-bending fatigue by Ewing and Humfrey in 1902 [10]. They impressively showed that localised slip bands developed in many grains during fatigue, resulting in the formation of microcracks. A dominant crack then grows, propagates, and causes to the final fatigue failure of materials. They also demonstrated that localised slip bands intersect with polished surfaces to form slip steps in the form of the extrusion and intrusion. Their study provided an insight into fatigue failure that fatigue cracking is formed as
a consequence of microplasticity taking place during cyclic loading. The cyclic plasticity results in the formation of extrusions/intrusions at free surfaces, leading to the formation of stress raisers. Such stress raisers can then induce the formation of fatigue cracks at free surfaces (Fatigue cracks are often found to initiate from free surfaces due to low/high cycle fatigue in smooth samples, but can also initiate from sub-surface locations for very high cycle fatigue loading). In other words, the formation of fatigue cracks is a surface phenomenon. The study of Ewing and Humfrey laid a cornerstone for the modern understanding of the mechanism of fatigue. Since then, there have been numerous researches striving to reveal the nature of the early stage of fatigue failure which is of great interest for applications in aircraft, nuclear and railway industries. In 1956 Thompson et al. showed that localised slip bands persistently reappeared at the same locations during cyclic loading even after removing the material and re-cyclic loading again [11]. The term Persistent slip bands (PSBs) is then named. PSBs are found to be a very important microstructural feature since it is one of the main microstructural sources to initiate fatigue crack, and get considerable attention over the last 50 years [12-18]. PSBs are sometimes abusively and loosely used to claim that any cyclic slip steps on free surface were due to PSBs. Mughrabi however pointed that PSBs only form for a condition where the loading amplitude exceeds a critical threshold [19]. The investigation of the origin of fatigue cracks clearly shows that the fatigue failure originates from cyclic plasticity which involves the movement and the rearrangement of dislocations. The evolution of microstructure during cyclic deformation leads to the initiation of fatigue cracks, and finally to fatigue fracture is summed up in Figure 1.1. The microstructural description during cyclic loading has been therefore intensively studied since the second half of the 20 century. In particular, Transmission Electron Microscopy (TEM) is intensively used to understand the fatigue mechanism after the first direct image of dislocations was recorded by TEM in 1956 [20, 21].
A great advance in the quantitative characterisation of fatigue failure has been achieved with the advent of modern fracture mechanics. The first is the development of linear elastic fracture mechanics (LEFM). It started in 1913 when Inglis [23] introduced the mathematical calculation of stress of brittle solids in the presence of cracks. Later, Griffith in 1920 laid the basis of the energy-based approach to treat crack growth [24]. A great step of the application of fracture mechanics on the fatigue failure was made by G. R. Irwin, who is regarded as “The father of fracture mechanics” [25]. Irwin first introduced a scalar quantity called ‘Stress intensity factor (K)’ to characterise the stress field ahead of crack tip. K is a function of applied stress (σ), crack length (a) and the geometrical factor (Y which is dependent on crack shape)

\[ K = Y\sigma\sqrt{\pi a} \]  (I.3)

Paris and Erdogan in 1963 proposed that the growth rate of intermediate fatigue cracks is driven by cyclic stress intensity factor during constant stress amplitude loading [26].

\[ \frac{da}{dN} = C(\Delta K)^n \]  (I.4)

With \( \Delta K = Y\Delta\sigma\sqrt{\pi a} \)  (I.5)
The Paris’s law is well accepted and widely applied in the defect-tolerant mechanical design against fatigue.

Because engineering materials inherently contain defects inside, the defect-tolerant approach has been commonly used in many practical applications, in particular in fatigue-critical applications, e.g. nuclear, automotive and aircraft applications. The defect-tolerant approach accepts that flaws are always inherently present in every engineering structural component. The defect-tolerant approach of modern mechanical design requires the quantitative knowledge of fatigue crack growth which fosters the increasing development of a research topic “Fractography” which was first promoted by three metallurgists Zappfe, Langraf and Worden in 1948 [27]. In 1954, Zappfe & Worden first described characteristic ripple markings (i.e. fatigue striations) on the surface of fatigue fracture [28]. Ryder in 1958 [29] and later in 1960 Forsyth and Ryder [30] successfully revealed the relation of fatigue crack propagation rate to the striation spacing. These pioneer studies introduced an important method to understand fatigue crack growth and the analysis of fatigue failures. The fractographic examination of fatigue fracture surface provides invaluable information of fatigue crack initiation and fatigue crack propagation. The advancement of electron microscopic techniques opens a door to get deeper insight into the initiation stage of fatigue crack, and the interaction between microstructural features and fatigue short cracks. The deeper knowledge of these events is of great interest over the last 50 years ago due to broad applications of mechanical/structural components whose lives are mainly constituted of the fatigue crack initiation and short fatigue crack growth stages.

The interest in crack initiation and short fatigue cracks also demands a more thorough treatment of fracture mechanics to deal with the plastic deformation process taking place ahead of crack tips because the dimension of the cyclic plastic deformation zone is comparable to the physical dimensions of fatigue cracks, and $\Delta K$ is no longer an applicable parameter to characterise the deformation field in front of crack tip. Efforts to solve this challenge led to the development of elastic-plastic fracture mechanics (EPFM) for which the contribution of Irwin is greatly appreciated, in particular his idea of introducing a crack-tip characterising parameter [31]. In 1972, Begley and Landes [32] proposed to use the path independent J-integral (which is defined by Rice [33] for crack condition after adopting the similar concept which was used for dislocation problem proposed by Eshelby [34]) as a
fracture criterion since the J-integral can averagely characterise the crack tip elastic-plastic field.

\[ J = \oint_{\Gamma} (Wdy - T_i \frac{\partial u_i}{\partial x} ds) \]  

(1.6)

Where:

- \( \Gamma \): is a closed contour bounding the crack tip.
- \( ds \): is an infinitesimal segment of \( \Gamma \).
- \((x, y)\): is the Cartesian coordinate system.
- \( u \): is displacement vector of \( ds \) under the traction force \( T \) \((T = \sigma_i n, \) with \( n \) is the outward normal to \( \Gamma \) )
- \( W \): is elastic strain energy density field, with \( W = \int_0^{\varepsilon_i} \sigma_i \varepsilon_i \)

Since the J-integral is independent of the contour path, it can be calculated based on the far-field stress state, e.g. applied stress. The J-integral therefore appears to be a very effective tool to quantitatively characterise the driving force of crack growth for both LEFM and EPFM. The extension of the application of the J-integral to the fatigue crack problem was first made by Dowling [35]. The introduction of the J-integral makes EPFM very capable to quantitatively characterise fatigue crack propagation which is important for the defect-tolerant mechanical design.

The above briefly historical overview of the study of fatigue definitely cannot mention every great contribution of a number of masterminds in this field. It however leads to a necessity to understand physical-underlying mechanisms responsible for cyclic deformation response of materials to provide a foundation to develop effectively constitutive model equations to accurately describe (and predict) the stress-strain response during cyclic loading, which is essential for mechanical design. Such constitutive model equations will also help to more accurately calculate \( \Delta K \) or the J-integral (Eqns. I.5 or I.6) to better quantitatively characterise fatigue crack propagation. Moreover, deep understanding of physical-underlying mechanisms of cyclic deformation response can shed light on the fatigue damage development of materials as referred to the above (Figure I.1). In particular, currently
increasing demands to reduce the prevailing conservatism in both mechanical design and lifetime assessment procedures to improve thermodynamic efficiency without compromising performance and reliability urge the development of effectively evolutionary constitutive model equations and a better damage assessment procedure for cyclic loading condition.
I.2. Motivation

The material of interest is an austenitic stainless steel (AISI 316L) which is a face-centered-cubic material. The material is widely used in industrial applications (e.g. petro-chemical and power generation industries) due to its outstanding corrosion resistance at low temperatures, and high oxidation resistance and superior creep resistance at high temperatures. The composition of the steel is shown in Table I.1.

Table I.1. Material composition

<table>
<thead>
<tr>
<th>C (%)</th>
<th>Mn (%)</th>
<th>P (%)</th>
<th>S (%)</th>
<th>Cr (%)</th>
<th>Mo (%)</th>
<th>Ni (%)</th>
<th>Cu (%)</th>
<th>B (%)</th>
<th>Co (%)</th>
<th>N (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.014</td>
<td>1.630</td>
<td>0.030</td>
<td>0.002</td>
<td>16.730</td>
<td>2.120</td>
<td>11.090</td>
<td>0.600</td>
<td>0.0011</td>
<td>0.090</td>
<td>0.060</td>
</tr>
</tbody>
</table>

The project material originated from a hot finished pipe which had been quenched in water from 1050/1080°C. This solution treatment was responsible for an austenite grain size of 60 µm (Figure I.2).

Figure I.2: Austenitic grains in the as-received condition
The material is utilised in nuclear power plants in Switzerland as a primary cooling circuit pipe material at temperatures up to about 300°C (Figure I.3). The performance and the mechanical integrity of the material in service are of the utmost important to improve the thermal efficiency as well as to assure the reliability of the whole nuclear power plant system. To improve the performance without compromising the reliability of structural components, necessary considerations must be taken both in the designing phase and in the lifetime condition assessment. In the design stage, reducing the conservatism can save a lot of the capital investment, and also makes structures lighter which can result in less energy consumption. Moreover, extending the lifetime of existing structural systems is desirable nowadays, which definitely requires a better lifetime prediction procedure. These demands can be achieved by: 1) developing accurately constitutive model equation describing well long-term mechanical behaviour response of the material, in particular cyclic deformation behaviour, on the basis of understanding underlying-physical mechanisms; and 2) obtaining deeper insights into the fatigue damage development of the material.
I.3. Objectives

This study aims to understand both cyclic deformation response and fatigue damage of the material at 20 and 300°C. The first objective is to systematically characterise the long-term mechanical response of AISI 316L steel at room temperature (20°C (293K)) and at an elevated temperature (300°C (573K)). The variation in microstructure during cyclic loading is exhaustively investigated (i.e. the characteristic of the dislocation motion, dislocation density, dislocation structures). The quantification of both mechanical response and microstructural evolution during cyclic loading is made in order to establish their relationships. Subsequently, the study aims to use the obtained knowledge of the relationship of microstructure-mechanical behaviour to formulate evolutionary constitutive model equation representations of internal variables which can accurately represent the history dependence of material cyclic response. The last objective is to investigate the influence of microstructural condition on the initiation and the propagation of microstructurally short fatigue cracks in order to understand the mechanism responsible for a reduction in fatigue life at 300°C relative to 20°C.
I.4. Structure of Thesis

A number of strain-controlled low cycle fatigue (LCF) tests at temperatures of 20 and 300°C, at different strain rates ($10^{-4}$ and $10^{-3}\text{s}^{-1}$) and strain amplitudes ($\pm0.25$, $\pm0.4$, and $\pm0.7\%$) were successfully performed to comprehensively characterise the cyclic deformation response of AISI316L (Chapter III). These tests were taken to failure or interrupted at different life fractions to provide samples for detailed microstructural evaluation. The microstructural investigation of the material during cyclic loading was exhaustively done by electron microscopy (EM), in particular by transmission electron microscopy (TEM) (Chapter IV). The quantification of microstructural change was also made. The physical interpretation of internal stresses is discussed in order to establish the relationship between microstructural evolution and cyclic deformation response (Chapter V). The relationship provides the basis to develop a physically-based constitutive model which is able to (1) describe the history dependence of the material cyclic response and (2) predict the material long-term response based on the data of short-term mechanical testing (Chapter VI). The model parameters were identified at a specific testing condition, and verified at other testing conditions. The model was also benchmark tested under considerably different testing conditions in order to independently check its effectiveness (Chapter VI). An effort to investigate the influence of microstructural condition on fatigue crack initiation and propagation during cyclic loading at 20 and 300°C was also made in order to understand the fatigue damage development of the material (Chapter VII).
Chapter II: Crystal defects and Role of dislocations in fatigue

Abstract:

This chapter first reviews the fundamental knowledge of crystal structure of metallic materials, in particular those with a face-centred-cubic (FCC) structure. Defects in crystal materials are also introduced in this chapter. Special attention is given to dislocations, e.g. their movement and their structural evolution during loading, and their observations by electron microscope. Some other fundamental knowledge of crystallography, to help readers more easily to follow microstructural investigations in this dissertation, is also introduced here. Finally, the microstructural evolution of FCC materials during cyclic loading is briefly reviewed.
II.1. Crystal structure

In a perfect crystal material, atoms arrange in a pattern (the basis) which periodically repeats in space. The way the basis repeats is called the lattice. The structure is then defined by: the basis + the lattice. A lattice is a set of points in the three dimensional space. All points of a lattice possess the identical surroundings. They are also identical in position relative to the repetition of the basis. Connecting lattice points will form parallelepipeds in the 3-D space. The smallest parallelepiped, which contains only one lattice point and fully represents a lattice by translation of the smallest parallelepiped from one lattice point to another, is called primitive cell. Another type of parallelepiped is called the unit cell which is a non-primitive cell and often used to better represent the symmetry of structure. A French physicist A. Bravais proved that the possible maximum number of space lattice is 14 (consisting of 7 primitive lattices and 7 non-primitive lattices). The 14 lattices are then named Bravais lattices.

The planes, directions and points in a lattice are defined by reference to the unit cell and the three principle axes, x, y, z (Figure II.1a). Indices of directions and planes are conventionally given in square ([hkl]) and round ((hkl)) brackets, respectively. Directions and planes of the same type are given in angle (<hkl>) and curly ({hkl}) brackets. The Miller indices are the
most common to index any plane and direction in lattice space. To index planes, the
intercepts of a representative plane with the three principle axes are first defined in relative to
\( \mathbf{a}, \mathbf{b}, \mathbf{c} \). Planes parallel to an axis are considered to intercept the axis at infinity. The
reciprocal of the three intercepts are taken, and then reduced to the three smallest integers.
For example, planes ABC and ODD’C are \((111)\) and \((\bar{1}0\bar{1})\). Any direction in a lattice space is
described by constructing a line which goes through the origin, parallel to the described
direction. The ratios of the projection of a segment of the constructed line starting from \( \mathbf{O} \) to
the closest lattice point to the corresponding lattice parameters are then defined. The
described direction is then given by the three smallest integers in the ratios. For instance,
directions OD’ and DB’ are \([111]\) and \([\bar{1}01]\).

The material of interest in this study is an austenitic stainless steel which has the face-
centered-cubic (FCC) structure. This crystal structure is one of the most common structures
of metallic materials. The FCC structure is represented by a unit cell. For FCC structure,
atoms locate at the eight corners and at six faces of its unit cell (Figure II.1b). If an atom is
considered as a hard sphere, atoms touch along the \(<110>\) directions. The closest-packed
directions and planes are \(<110>\) and \{111\}, respectively. The atomic parameter of FCC
structure is \( 2r\sqrt{2} \) with \( r \) is atomic radius. The stacking sequences of \{111\} planes are
ABCABC (Figure II.1c).
II.2. Defects in crystal materials

Imperfections of real crystal materials can be point, line, surface (or planar), or volume defects. The presence of these defects significantly governs the behaviour of crystal materials. Amongst these types of defects, line defects called dislocations are of great interest since they are the most common to accommodate the plastic deformation of crystal materials. The presence of dislocations and the way they interact with other types of defects intimately control the response of materials.

II.2.1. Point defects

Point defects are imperfections at specific atomic sites which can be formed by: (1) removing atoms from their sites to form intrinsic point defects (i.e. a vacant atomic site), or (2) introducing atoms into free space between host atoms to form a type of extrinsic point defects (i.e. interstitial atoms), or (3) replacing host atoms by impurity atoms to form other types of extrinsic point defects (i.e. substitutional atoms). The formation of point defects can be made either by thermal force or mechanical force or irradiating force.

The introduction of point defects into crystal materials induces the distortion around point defects. The stress field around point defects interacts with stress fields of other types, resulting in the alternation of material response. The mobility of point defects strongly depends on temperatures and the state of internal stresses which results from the presence of other types of defect, in particular dislocations. The interaction of point defects with dislocations is of great interest from both scientific and economic points of view. For example, the formation of numerous small clusters of vacancies due to irradiation significantly influences the mechanical integrity of structural components in nuclear power plants. Moreover, the strong interaction of point defects with dislocation cores over certain conditions results in some peculiar behaviour (e.g. flow stress serration, negative strain rate sensitivity etc.) of a number of metallic materials (Al, Al-Mg alloys, Low carbon alloys, Au-Cu alloys, Fe-Cr alloys etc.). This kind of interaction is examined in detail in this study (Chapter IV) in order to explain the dynamic strain ageing phenomenon of AISI 316L at 300C.
II.2.2. Dislocations

All crystal materials contain numerous lines of structural discontinuities which are called dislocations. They were first introduced by an Italian mathematician Vito Volterra in the late 19th century as an abstract mathematical concept. The extremely important recognition of the dislocation concept was identified in 1934 when Taylor [36, 37], Polanyi [38] and Orowan [39] independently proposed that dislocations could be responsible for the plastic deformation of crystals. The concept of dislocations was then often invoked to explain most of the phenomena in crystal plasticity and crystal growth in the first half of the 20th century. In the 1940s and 1950s, there were major advances in the development of the dislocation theory in crystal materials. There were great efforts to reveal the implication of the theory of dislocations on the crystal growth and mechanical behaviour of crystals. The two most comprehensive reviewing studies on this field during the period were made by Cottrell (“Dislocations and Plastic Flow in Crystals” [40]) and by Read (“Dislocations in Crystals” [41]). The major advance in dislocation theory urged the search of dislocation observation to underpin the proposed theory. In 1956, Menter [21] and then Hirsch et al. [20] first observed the direct image of dislocations by TEM. The theory of dislocations and its great importance to the study of the controlling mechanism of mechanical response were then strongly corroborated, and further developed significantly. In the second part of this chapter, and in Chapter IV and V, we will see the importance of the role of dislocations on cyclic deformation response. In addition, an evolutionary constitutive model based on dislocation theory is successfully developed to describe the cyclic deformation response of the material (Chapter VI). Moreover, it will be also shown (Chapter VII) that the activity of dislocation slip bands is the main source of the initiation of fatigue cracks.

The disruption of a crystal due to the introduction of a dislocation is characterised by the Burgers vector, \( \mathbf{b} \). In other words, the Burgers vector determines the nature of the dislocation and its characteristic feature. The Burgers vector is the most invariant characteristic of a dislocation. In order to determine the sense of Burgers vector, it is necessary to adopt a sign of convention. The RH/FS convention is adopted and explained as follows: A path is made from atom-to-atom in a crystal to enclose a dislocation line according to right-handed (RH) screw relationship to the dislocation line. The path is called a closed Burgers circuit. The similar path is mapped in an imaginary dislocation-free crystal at the same region. In order to
make the latter circuit to be closed, a Burgers vector is taken from the finish to the start of the path (the FS of the notation).

In every crystal material, the formation of dislocations results from many different ways, e.g. by shearing along a crystal plane which makes atoms slip over a part of crystal along the crystal plane (as a consequence, the boundary between slipped and unslipped parts of the sheared plane is a dislocation), or by the condensation of point defects (interstitials or vacancies). If a dislocation can form by this process and the shearing direction is perpendicular to the dislocation line, the dislocation is of the edge type. In contrast, if the shearing direction is parallel to the dislocations line, the dislocation is of the screw type. Consequently, the first type of dislocation is called an edge dislocation; the latter is called a screw dislocation. One can easily realise that $b_e$ is always perpendicular to the edge dislocation line, whereas $b_s$ is always parallel to the screw dislocation line. In reality, a large quantity of dislocations can form during plastic deformation due to applying shearing force. Most dislocations are of the mixed type. Dislocations also often form during crystal growth. For example, the misfit strain due to the thermal gradients during cooling the melt of a crystal results in the formation of dislocations. This explains why engineering materials always inherently contains a lot of dislocations in the as-cast condition.

If the passage of a dislocation leaves a slip step on the free surface of the crystal and the crystal structure is perfect again, the dislocation is called a perfect dislocation. The Burgers vector of a perfect dislocation is a lattice vector. In some circumstances, a perfect dislocation can dissociate into partial dislocations in order to reduce its energy. The Burgers vectors of partial dislocations are less than the lattice vector. The area between two partial dislocations is a faulted area, called a stacking fault.

## II.2.3. Surface defects

Typical examples of surface (planar) defects are stacking faults, grain boundaries, twin boundaries. Surface defects play an important role on the plastic deformation response of crystals. Hall [42] and Petch [43] independently proposed that the flow stress of polycrystals is proportional to the grain dimension by: $\sigma_f = \sigma_0 + kd^{-1/2}$. This finding is known as the
Hall-Petch relationship. The famous Hall-Petch relationship is due to the impedance capability of grain boundaries on dislocation movement, and fully accounts for the importance of grain boundaries on the strength of crystal materials. Moreover, stacking faults strongly influence the character of dislocation movement, which in turn affects material response during plastic deformation (Sect. II.6). The role of the slip character on the evolution of microstructure is thoroughly discussed in Chapter IV.

**Grain boundaries**

Crystal materials usually consist of many grains which orientate differently and are separated by grain boundaries. Grain boundaries normally contain a lot of defects, in particular dislocations. Depending on the characteristics of dislocations, grain boundaries can be the tilt (for edge dislocations) or twist (for screw dislocations) types. If the misorientation of two grains is small, their boundary is of the low-angle type.

**Stacking faults**

A stacking fault is a planar defect at which the stacking sequence of the crystal is interrupted. For example, for face centered cubic materials, the regular stacking consequence of (111) plane is ABCABCA… which is a most energetic stacking consequence of three close-packed planes A, B and C (Figure II.1c). However, at certain positions, the presence of a stacking plane is missing which results in a break in the stacking sequence, i.e. a stacking fault is formed. In addition, if there is a plane which is inserted between layer A and B, a faulty sequence occurs. A stacking fault formed by a removal of a plane is called an intrinsic stacking fault, whereas a stacking fault formed by inserting a new plane is called an extrinsic stacking fault.

A stacking fault is bound by partial dislocations. The breadth of a stacking fault (or the separation of partial dislocations) is governed by stacking fault energy (SFE). The lower the stacking energy, the wider is the spacing of the partial dislocations. The chemical/stress interactions between stacking faults and solute atoms attracts the segregation of small solute atoms into faulted areas, resulting in lowering the stacking fault energy [44].
Twin boundaries

Twinning deformation is another mechanism of plastic deformation, in particular at low temperatures. During twinning deformation, a part of a crystal is homogenously sheared to produce the same crystal structure to the parent structure but in another direction. Twinning deformation is of particular importance for face-centered-cubic, body-centered-cubic, close-packed hexagonal metallic materials and non-metallic materials. It is also another source of plastic deformation for low stacking fault energy materials at low temperatures. The boundary between the twinned part and the untwined part is called the twin boundary.

II.2.4. Volume defects

Volume defects are precipitates, voids, bubbles which can form during thermal-mechanical processing, or during radiation (e.g. the swelling of nuclear materials). Volume defects can very effectively control the movement of dislocations and the growth of grain boundaries, resulting in the alternation of material response. They are particularly of great interest in the study of long term creep behaviour.
II.3. Dislocation movement

Plastic deformation of crystal materials is effectively accommodated by the movement of dislocations. The way dislocations move under imposed loading governs the response of crystals. *Conservative motion* of dislocations (or dislocation glide) occurs when dislocations move in the plane which consists of both its line and the Burger vector. Whereas, *non-conservative motion* of dislocations (dislocation climb) happens once dislocations move out of the glide plane. Depending on the mobility of a dislocation on the slip plane under shearing, there is another way to classify dislocations. A *sessile dislocation* is not capable of moving, whereas, a *glissile dislocation* can move on the slip plane [45].

At low temperature (less than 0.3-0.4Tm), plastic deformation of crystals mainly occurs by dislocation glide. Dislocation glide depends on lattice resistance, discrete obstacles and phonon and other drags. For FCC and HCP crystals, the lattice resistance does not play an important role on dislocation glide. The flow stress is much more dependent on the distribution manner of discrete obstacles and their strength. Depending on the stress field induced by barriers to dislocation glide, thermal factors can play an influential role on the movement of dislocations, in particular the short-range movement of dislocations confined to the atomic dimensions. For instance, with the aid of thermal activation, dislocations can easily overcome the resistance of lattice and solute atoms. In contrast, thermal activation plays a much less important role on the way dislocations overcome barriers formed by a group of dislocations or large incoherent precipitates.

Before discussing the glide of dislocations in detail, some knowledge of crystallography of FCC materials is given to better describe the crystallographic orientation in the materials.

II.3.1. Stereographic projection of FCC structure

The *stereographic projection* is a visual aid which is able to represent crystal plane and directions on a diagram in two dimensions so that angular relationships and the symmetrical arrangement of crystals are preserved. Stereographic projection provides an excellent aid to investigate the change of crystal orientation with respect to the loading direction, i.e. rotation of crystal axes with the loading direction, and the movement of dislocations on different slip
systems during plastic deformation. The stereographic projection is also a good tool to understand electron diffraction pattern under certain beam direction.

Construction of stereographic projection (Figure II.2a): Imagine positioning a crystal with its centre at the centre of a sphere which is called the sphere of projection. Draw normal lines to crystal planes from the centre (O) of the sphere to intersect the sphere surface, say at P in the northern hemisphere. P is called the pole of the plane of which OP is the normal. A crystal direction is similarly represented by a point on the surface of the sphere which is an intersection of a line going through the centre and being parallel to the given direction with the sphere surface. A crystal plane can be represented by a line which is intersection of the parallel plane through the centre with the sphere surface. The intersection line is called a great circle. For the point P, draw a line from the south pole to the P which cuts the equatorial plane at P’. A line of intersection of the equatorial plane with the sphere is a great circle which is called the primitive circle. The primitive circle is the stereographic projection and the point P’ uniquely represents a plane whose radial normal goes through P. If P is in the southern hemisphere, a line is drawn from the northern pole. In this case, P’ is marked by a ring instead of a dot. In the standard projection, z axis points to the northern pole, the x and y axes are in the stereographic projection plane. In order to get to know how to construct a detail standard stereographic projection, one can refer to any fundamental book on crystallography (e.g. Crystallography and crystal defects by A. Kelly, G. W. Groves and P. Kidd [46]). There are some important rules of standard stereographic projection:

- A great cycle always passes through opposite ends of a diameter of the primitive cycle
- Poles of set planes sharing the same zone axis lie in a great cycle which is apart 90° from the pole of the zone axis.
- The angle between two poles on an inclined great cycle is the angle subtended by arc which constructed by projecting two poles to the primitive cycle from the pole of the inclined great cycle.

The Wulff net provides a useful stereographic projection for angular measurement. The Wulff net is constructed with the angle interval of 2° of 90 great cycles all passing from the north pole to the south pole and another is the equator (Figure II.2b). All other latitude circles except the equator are called small circles.
Figure II.2: (a) Construction of the standard stereographic projection, (b) Wulff net.

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The stereographic projection for the FCC materials (Figure II.3) is divided by 24 triangles, each bounded by a set of adjacent points of the forms \{100\}, \{110\} and \{111\}. If a single crystal is loaded, the loading direction is represented by a unique point in the projection.

Figure II.3: A stereographic projection of face-centred cubic structure with the [011] normal.

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II.3.2. Thompson tetrahedron [48]

The best way to express gliding planes and directions in FCC materials is to use the Thompson tetrahedron which was suggested by Thompson in 1953. The Thompson tetrahedron is made up by four lattice points: one lattice point (D) locates at a corner (the origin of an arbitrary co-ordination) and three locates (A, B, and C) at the centre of three adjoining faces which are correspondent to planes of x=0, y=0, and z=0, respectively (Figure II.4). The faces of the Thompson tetrahedron represent the four close-packed planes of the FCC structure while the edges represent the close packed slip directions. Vectors connecting from one vertex to another of the tetrahedron are perfect translation. Vectors connecting from one vertex to the centre of the tetrahedron faces are imperfect translation.

![Thompson tetrahedron construction](image)

Figure II.4: The construction of the Thompson tetrahedron

II.3.3. Glide systems

Under imposed plastic deformation, dislocations can move on certain defined crystallographic planes and directions which are both close-packed planes and close-packed direction in crystal structure.

Glide of many dislocations on a defined crystallographic plane and direction result in slip plane and slip direction. A slip plane and a slip direction in that plane constitute a slip system.

In FCC materials, the most close-packed planes are the four \{111\} planes, and the shortest translation lattice vectors are the three \(\frac{1}{2}<110>\) vectors. The FCC materials therefore have 12 slip systems. In order to move dislocations along a slip plane, a required shear stress is required. If a specimen of cross section area of A is subjected to a force F which forms an
angle \( \lambda \) with the slip direction and an angle \( \theta \) with the normal slip plane (Figure II.5), then the resolved shear stress of \( F \) on the slip plane in the slip direction is

\[
\tau = \frac{F \cos \theta \cos \lambda}{A}
\]  

(II.1)

The resolved shear stress should be higher than a critical value of shear stress in a given plane in order to move the dislocation. The threshold of the resolve shear stress is called critical shear stress.

Figure II.5: Slip plane and slip direction under tensile load

Under loading, a slip plane of the highest resolved shear stress is the most favourable for dislocation movement. This slip plane is called the primary slip plane. Upon further loading, crystal axes rotate so as the loading axis approaches the slip direction in case of tensile loading and towards the normal of the slip plane in case of compressive loading [49]. The crystal rotation during loading results in the equal resolved shear stress on two slip systems, which result in double slip. The second slip system is the conjugate system. The double gliding induces the crystal rotation until loading axis is symmetrically related to the two slip
systems. The loading axis therefore towards $<112>$ directions in case of tensile loading, and $<011>$ directions in case of compressive loading.

![Diagram](image1.png)

**Figure II.6**: Primary slip, conjugate slip, critical slip and cross slip planes in case of tensile force axis is presented by F point in the stereographic projection.

Screw dislocations can change their slip plane during loading, which greatly influences the plastic deformation behaviour of crystal materials. *Cross slip* is a term to describe the process whereby trapped screw dislocations in primary slip plane can escape by change their slip plane into cross slip plane but still keep moving in the same direction. The cross slip activity increases a chance of the annihilation of dislocations. Cross slip is therefore responsible for stress relaxing and dynamic recovery process, resulting in the softening. The inception of cross slip explains the decrease in strain hardening rate in the transition from stage II to stage III of work hardening in monotonic loading condition (Sect. II.4). Cross slip plays an important role in the formation of dislocation-rich and dislocation-poor regions upon further loading. Every slip systems except the primary slip one is called secondary slip. For example, primary, conjugate, critical and cross slip systems for FCC materials under a given tensile loading direction (denoted by F) are $\langle 101 \rangle \langle 111 \rangle$, $\langle 101 \rangle \langle 111 \rangle$, $\langle 101 \rangle \langle 111 \rangle$, and $\langle 101 \rangle \langle 111 \rangle$, which are demonstrated in **Figure II.6**.
II.3.4. Dislocation-dislocation interactions

Lomer-Cottrell interactions [50, 51]: The interaction of dislocations on the primary slip plane with ones on the conjugate slip plane result in immobile dislocations (Lomer-Cottrell dislocation junctions) which in turn restrict dislocation movement during loading. The Lomer-Cottrell dislocation interaction is the most common interaction of dislocations in FCC materials. The locking mechanism due to the formation of Lomer-Cottrell sessile dislocations is called the Lomer-Cottrell locking mechanism. This locking mechanism plays an important role on work hardening and stabilising of dislocation substructures. For example, if we assume the primary slip plane is (111), then the conjugate, critical, and cross slip planes are (-1-11), (-111), and (1-11), respectively. Slip directions on the faces d, (111) and c, (-1-11) are \( \frac{a}{2} [1-10] \) (AB), \( \frac{a}{2} [01-1] \) (BC), \( \frac{a}{2} [-101] \) (CA), and \( \frac{a}{2} [1-10] \) (AB), \( \frac{a}{2} [-10-1] \) (BD), \( \frac{a}{2} [011] \) (DA), respectively (where d and c are Thompson tetrahedron faces corresponding to vertex D and C of the tetrahedron (Figure II.4)). Lomer showed that a dislocation whose Burgers vector is BC on the primary slip plane can interact with another one whose Burgers vector is DB on the conjugate slip plane to form a dislocation with Burgers vector of DC. The resulting dislocation is immobile due to its line and its Burgers vector not lying on the same close-packed plane. Moveover, Cottrell emphasised that the Lomer product dislocation can become very immobile if it further dissociates into imperfect dislocations in order to reduce its energy as in the following way:

\[
DC, \frac{a}{2}[110] = D\gamma, \frac{a}{6}[112] + \gamma\delta, \frac{a}{6}[110] + \delta C, \frac{a}{6}[11-2]
\]

Hirth lock [52]: Interactions of dislocations on the primary slip plane with ones on the critical slip plane can also produce immobile dislocations which also play as locks for dislocation movement.
II.4. Work hardening stage in monotonic loading

Stage I: Dislocations easily move on single slip mode. The hardening rate of this stage is small. The occurrence of stage I greatly depends on the orientation of crystals with respect to the loading direction. If the interested crystal orients to the loading direction in such a way that it prefers the multiple slip, stage I will not occur (Figure II.7).

Stage II: The hardening rate of this stage is highest due to strong activity of secondary slip systems which induces increase in dislocation density and sessile dislocations, resulting in a significant increase in the hardening rate. The work hardening rate in this stage is rather constant \( \frac{d\tau}{d\gamma} = \frac{\mu}{200} \) and being weakly dependent on temperature and strain rate.

Stage III: The work hardening rate linear decreases in this stage due to dynamic recovery which is sensitive to temperature, strain rate and the activity of cross slip. This stage takes place earlier for crystal materials of high SFE, which causes a decrease in the duration of stage II. At high temperature working conditions, the stage II even does not occur for high SFE materials.

Stage IV: After the decrease in the work hardening rate during the stage III, if the material still endures higher plastic strain (the strain amplitudes of the order of 5-8%), the rate reaches to a very low value which is almost in the same order of that of stage I.

Stage V: The work hardening of materials terminates by the true saturation stage (stage V) at a very larger strain.
Figure II.7: Ideal work hardening stages of single slip and polyslip in the hardening coefficient $(\frac{d\tau}{d\gamma})$ versus flow stress $(\tau)$ co-ordination (after [53]).

Polycrystal materials normally do not show stage I work hardening after imposed shear stress reaches the critical resolved shear stress. They often start to behave in the same manner of stage II and then followed by stage III (Figure II.7).
II.5. Observation of dislocations

II.5.1. Transmission electron microscopy

Transmission electron microscopy (TEM) is the most powerful and versatile technique to directly characterise dislocations in multi-scale levels. After the first direct study of TEM on dislocations [20, 21], several useful techniques based on diffraction contrast (Bright field, Dark field, Weak-beam, Convergent beam techniques) have been developed to observe and characterise the nature of dislocations (e.g. their Burger vectors, their interactions, configurations, distributions) in many crystal materials under various loading conditions [47].

The structure of a typical TEM is shown in Figure II. 8. A typical TEM consists of an electron gun which provides the incident electrons. Electrons emitted from the electron gun will be collimated when they go through two condenser lenses, and straight travel onto the very thin specimen. The electron beam is diffracted along certain orientation according to Bragg’s law when it goes through the crystal structure. The image of dislocations is essentially a diffraction phenomenon. For a thin foil, the Bragg condition is relaxed, leading to the reciprocal lattice points becoming ‘relrods’. Moreover, the presence of dislocations induces the crystal distortion which changes the diffraction condition of the interested location. That makes the diffracted beam of distorted area different to that for the perfect parts of the foil. Consequently, dislocations can be imaged.

An objective lens which is immediately below the specimen brings diffracted beams into focus in the back focal plane of the objective lens (Figure II. 9). At the back focal plane of the objective lens, electron beams which leave the thin specimen at the same angle will converge at the same point. The back focal plane consists of the diffraction pattern of the thin specimen. The diffraction of the specimen can be imaged in the diffraction mode of TEM by the appropriate operation of subsequent lenses.
II.5.1. Bright field and Dark field imaging

In the imaging mode of TEM, an image of illuminated area of the thin specimen is obtained in the image plane of the objective lens. The image is then magnified by the intermediate lenses. In a simple way, an image of the interested location is formed from all transmitted and diffracted beams. The contrast of this imaging mode is very low. There is a small aperture (Objective aperture) placed close to the back focal plane of the objective lens to allow us to select either transmitted beam or some specific diffracted beams, which then
gives much better contrast. There are consequently two ways to obtain images in TEM, which are:

1. Bright field (BF) imaging if the transmitted electron beam is selected
2. Dark field (DF) imaging if a specific diffracted beam is selected.

Figure II. 9: Ray diagram of electron beam in TEM, image mode.

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While the electron beam is still along the optical axis in the BF mode (Figure II.10a), the beam is deflected from the optical axis by the diffraction angle in the DF mode (Figure II.10b). This makes the TEM image in DF mode blurred due to the spherical aberration. To improve the quality of the DF image, it is necessary to tilt the incident illumination so as the diffracted beam is now parallel to the optical axis (Figure II.10c). This technique is called ‘Axial dark field’. In this study, dark field image is indeed used to represent the image
obtained by the ‘Axial dark field’ technique. **Figure II.11** gives an example of the diffraction under two-beam condition and its corresponding BF and Axial DF images.

![Diagram of microscope optics](image1)

**Figure II.10:** (a) Bright-field mode, (b) Dark-field mode, (c) Axial Dark-field mode.

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![Images of diffraction patterns](image2)

**Figure II.11:** (a) The two beam diffraction pattern (the most intensive diffracting plane is (200)) in the [011] beam direction, and its corresponding (b) BF image and (c) DF image.
II.5.1.2. Weak-beam Dark-field (WB) technique

It is necessary to get more distinct contrast of dislocation lines in order to get a better evaluation of microstructural condition, e.g. dislocation density measurement. The weak-beam dark field technique is a powerful way to make a very sharp image of dislocation lines (Figure II.12). There are three steps to make a weak beam image:

1. In the diffraction mode, rotating the specimen into a two-beam condition. In other words, there are only two strong spots in the diffraction pattern. One is the transmitted and the other is the excited diffraction spot.
2. Tilting the incident beam until the diffracted spot moves into the position of the transmitted spot. The diffracted spot now becomes weak.
3. Inserting the objective aperture to let only the interested diffracted spot go through to obtain the WB image.

Figure II.12: (a) BF image under two-beam condition with excited (220) diffraction. (b) Weak beam image of dislocations with weak (220) diffraction.

(Reprinted from [54] with permission of Springer Verlag)
II.5.1.3. Selected area electron diffraction

To image the diffraction pattern of the specimen, the intermediate lenses are weakened, and are focused on the back focal plane of the objective lens. A second aperture is placed in the image plane of the objective lens to obtain the diffraction pattern of a selected area. The selected area electron diffraction technique (SAED) is performed as follows. The examined specimen is first observed to find an interesting area (Figure II. 13a). The intermediate aperture is then inserted and positioned to illuminate only the selected area. The microscope is then switched into the diffraction mode to get the diffraction pattern of the selected area (Figure II. 13b). The diffraction pattern of a selected area will provide the information of crystallographic orientation of the selected area with respect to a specific orientation, e.g. loading direction (LD). Especially, the SAED can also shed light on the interaction of solute atoms with dislocations. Since solute atoms, e.g. C and N, are light elements, TEM imaging cannot reveal the clear configuration of C and N around dislocation lines. The structure of solute atom atmosphere of C and N however can be realised from the analysis of the selected area electron diffraction pattern [47]. This is of great importance to this study since the interaction of solute atoms with dislocations at 300°C causes an interesting phenomenon called Dynamic Strain Ageing which significantly changes the material deformation response.

Figure II. 13: (a) BF image of dislocations and (b) its corresponding SAED (the diffraction pattern of the [001] beam direction)
II.5.1.4. Convergent-beam electron diffraction

Modern TEMs provide a great versatility to focus incident electron beam by two condenser lenses to form a cone of incident beam impinging on the specimen over a range of angles. The convergent-beam electron diffraction (CBED) is performed by controlling the angular angle of the cone and its size. Unlike the SAED technique where electron rays are parallel, the electron beam is convergent in CBED which makes the examined region much smaller than that of SAED. This technique becomes increasingly important for nanotechnology since it can make diffraction from a region which is only nanometers in its size. Some very useful information of crystal materials can be obtained by CBED. For instance, the sample thickness can be measured by parallel fringes (the Kossel-Möllenstedt pattern) in the CBED pattern under two-beam conditions (Figure II.14). In addition, a wealth of crystallographic information can also be obtained by CBED, in particular from the higher-order Laue-zone pattern.

Figure II.14: Parallel fringes in a zeroth order Laue-zone in a CBED pattern of an AISI 316L specimen taken under two-beam conditions. The beam direction is [011].
II.5.1.5. Kikuchi lines

![Image of diffraction pattern and Kikuchi lines](image)

Figure II.15: Diffraction pattern and pairs of bright and dark Kikuchi lines.

In diffraction mode, if the examined region of crystal specimen is thick enough (but is still relatively transparent to electrons), there is set of diffuse straight lines (which are called Kikuchi lines) and the diffraction spots (Figure II.15). Kikuchi lines provide important information about crystallographic orientation. Kikuchi lines of a selected area are applied in this study to measure the mis-orientation from location to location. Kikuchi lines are formed due to two electron scattering processes: first an incoherent electron scattering induces excess (bright) lines, some incoherently scattered electrons then undergo a coherent Bragg diffraction to form deficient (dark) lines. To understand more about the Kikuchi lines (e.g. the origin, how to index Kikuchi lines, and their applications), please refer to some text books of TEM, e.g. [54] or [47].
II.5.2. Scanning electron microscopy (SEM)

II.5.2.1. General

Scanning electron microscopy (SEM) is also a powerful technique for microstructural characterisation and for fracture investigation. SEM offers a wide range of observation, from nanometer to micrometer scales. The requirements of samples for SEM observation are not as critical as those for TEM. In addition, their efficiency in recording images and their large depth of field make SEMs more effective for microstructural characterisation and fractographic investigation than TEM to some extent.

A scanning electron microscope consists of three basic component blocks, which are:

1. Electron gun and lenses to produce a small electron beam
2. Deflection control systems to move the electron beam to a series of discrete points along lines
3. Electron detector to collect the signal. When electrons impinge on the specimen, they interact with the specimen, which results in many types of generated signal. There are two main types of signal often collected to image the specimen, which are secondary electrons (SE) and backscattered electrons (BSE).

The two principal signals, e.g. SE and BSE, are generated within the interaction volume, and carry a lot of information regarding the specimen composition, shape, crystallographic orientation. While SE can mainly provide the information of the morphology of the specimen which is very useful for fractographic observation, BSE can provide a wealth of information relating to atomic number and local crystallographic orientation. The orientation dependence of BSE signal is of great importance to quantify the crystallographic orientation of the specimen by Electron backscattered diffraction technique (EBSD), and also to image dislocation structures (even individual dislocations) by Electron channeling contrast technique. Recently, there is a great effort put into crystallographic-based contrast SEM technique (Electron channeling contrast technique) to improve the capability of SEM on the observation of dislocations [55-59].
II.5.2.2. Electron channeling contrast imaging

TEM observation of dislocations requires a very thin and shiny specimen, which makes the TEM sample preparation to be difficult and time-consuming. In addition, it takes considerably time to train a new user who can operate TEM in a properly manner to observe dislocations. Moreover, the examined area by TEM is relative small. An alternatively powerful technique to overcome these difficulties is to perform electron channel contrast imaging (ECCI). Although TEM is definitely the right choice to get comprehensive analysis of dislocations, ECCI offers great advantages, which are:

1. The ease of sample preparation
2. Large examination area
3. Much easier to perform in-situ mechanical testing with observing a great detail of microstructure.

The periodic arrangement of atoms in crystalline materials can influence the backscattered electrons. If parallel electron rays impinge on a crystalline sample, electrons will encounter a number of atoms. Electrons can enter deeper into the crystalline sample along certain directions along which the number of atoms is low, before they are scattered back onto the free surface. The intensity of BSE signal in these directions is therefore lower compared to the directions in which there are higher numbers of atoms. The BSE signal is therefore dependent on the crystallographic orientation of the examined region of the crystalline material relative to the direction of incident beam. The presence of dislocations introduces the distortion which alters the scattering condition of the electron beam. The mechanism of forming dislocation contrast using the ECCI technique is therefore similar to the diffraction-based contrast in TEM. The electron channelling contrast obtained by a dedicated BSE detector is relative low compared to diffraction-based contrast in TEM. It is necessary to have:

1. high brightness SEM (a field emission gun SEM),
2. high current intensity to increase the signal to noise ratio,
3. small probe size to get high resolution ECCI,
4. small beam convergence angle.
In this study ECCI is applied to investigate dislocation structures, in particular the activity of PSBs, in large areas which complements the detailed work of TEM (Figure II.16 and Figure II.17). Especially, since fatigue cracks (both long and short fatigue cracks) can be much easier to be preserved during SEM sample preparation compared to that for TEM observation. ECCI exclusively offers a great opportunity to investigate the interaction of dislocations structures with short fatigue cracks, or with the crack tip of intermediate fatigue cracks.

Figure II.16: Labyrinth structure of dislocations observed by ECCI technique.
Figure II.17: ECC image of Persistent slip bands in fatigued AISI 316L sample at 300°C
II.6. Microstructural evolution during cyclic loading

The microstructural condition strongly depends on the slip character of dislocations during cyclic loading. The wavy slip character, which are often exhibited in Aluminum, Nickel and Copper, indicates dislocations easily cross slip. The planar slip character means that dislocations are impeded to cross slip during movement. AISI 316L and α-brass show a strong tendency of planar slip character during loading [60]. The significance of wavy and planar slip characters on deformation response of crystals was first discussed by Feltner and Laird [61, 62]. It was then realised that the slip character of dislocation motion strongly influenced the cyclic deformation response of FCC materials. While the cyclic deformation response of wavy slip materials are not sensitive to the loading history, that of planar slip materials are greatly dependent on the history of loading which makes the material response of this type of materials rather complex. The reason for the complex change of material response is due to the fact that the slip mode character causes the different development of microstructure during cyclic loading. The dependence of dislocation structures on the slip character is well described by the diagram of the dependence of fatigue-induced dislocation patterns on cyclic slip mode and fatigue life (Figure II.18) [6].

![Diagram of the dependence of fatigue-induced dislocation patterns on cyclic slip mode and fatigue life.](Reprinted from [6] with permission from Elsevier)
The origin of planar or wavy slip characters of dislocations is first believed due to the stacking fault energy. The lower stacking fault energy means the wider distance between two partial dislocations, resulting in more planar slip character of dislocation motion. It is however recognised that the planar slip of dislocation movement even exists for intermediate and high stacking fault energy materials, e.g. Ni-Al, Al-Li and Ni3Fe. The origin of planar slip mode of dislocations is then revealed that the planarity of dislocation slip also strongly depends on the presence of well-developed short range ordered (SRO) structures [63]. For austenitic stainless steels with a high concentration of Nitrogen (N), N can form a SRO structure with other metal atoms such as Cr [64]. It partially explains why AISI steels increase the planar slip tendency with the increase in the N concentration.

For low stacking fault energy materials, in the as-annealed condition and during a small degree of plastic deformation, dislocations dissociate into partial dislocations, and are present in the planar character of dislocation configuration (e.g. regular dislocation arrays). The activation barrier for cross slip can be overcome or not which depends on the plastic strain amplitude, temperature, and the accumulated plastic strain. At higher plastic strain amplitudes or at elevated temperatures, the stage of planar character of dislocation movement usually takes place in shorter periods. The higher the number of cycle is, the more wavy dislocations slip. Upon further loading, dislocations increase in their number and interact with other dislocations in other slip systems and with other defects (secondary phases, grain boundary etc.) to form more tangled structures, such as bundles, pile-ups in regions close to obstacles. Dislocation tangled structures induce local stress field which can promote the cross slip of dislocations. The planar slip character becomes more wavy slip character during cyclic loading. For intermediate and high plastic strain amplitude condition, dislocation structure at the end of fatigue life of planar slip materials is consequently more or less similar to that of wavy slip materials. For instance, there are some similarities between the dislocation structures at the end of fatigue life of AISI 316L and pure Cu crystal (which exhibits very wavy slip character): dislocation loop patches, PSBs which are characteristic consequence of wavy slip activity of dislocations [65-67].

For planar slip materials, in the beginning, plastic deformation is mainly carried out thanks to the movement of dislocations (primary dislocations) in the primary slip plane. More primary slip activates during cyclic loading, leading to the reduction of inter-distance of primary slip
planes. The activity of secondary slip (including cross slip) results in interactions between dislocations. In FCC materials, the Lomer-Cottrell interaction is the main interacting activity between dislocations themselves, causing a rise in the number of dislocation sessile junctions. These sessile junctions are immobile, which initiate and stabilise dislocation tangled structures (dislocation veins, walls). The cross slip activity is of great importance. It increases the mobility of dislocations and mainly contributes to the dynamic recovery, resulting in a lower rate of work hardening. Increasing the imposed plastic strain amplitude or loading temperature can make cross slip activate earlier. In particular, screw dislocations can cross slip and travel between the primary slip planes. The cross slip activity of screw dislocation can cause:

(1) The reduction of inter-plane distance between primary slip planes;

(2) The shortening of edge dislocation trailing. In particular, screw dislocations can cut dislocations in primary slip planes into short segments, which promotes the formation of dislocation veins [65, 66, 68].

The interaction between screw dislocations and between edge dislocations on secondary slip planes and edge dislocations on primary slip plane cause the development of dislocation tangled structures. In the early stage of the formation of dislocation tangled structures, dislocation loop patches which are often observed in pure copper consist of dislocation veins of high dislocation density which almost encircle regions of low dislocation density [69]. Dislocation veins are observed to lie almost parallel \([12\bar{1}]\) (which is the direction of the kink band plane) or along the intersections of the critical and conjugate planes, \(<110>\). Dislocations within veins are mainly of primary edge dislocations. About an order of magnitude lower is of secondary dislocations [69]. Regions of low dislocation density (dislocation channels) mainly contain screw character dislocations straddling dislocation veins. The dislocation density within channels is about two or three orders of magnitude smaller than that within veins.

Upon further loading, dislocation veins and channels structure develop into more well-organised structures, i.e. dislocation walls and channels. Dislocation walls consist of denser dislocations than those within veins, whereas channels become clearer of dislocations. In
addition, depending on the strain amplitude condition, a very striking and important feature of dislocation arrangement emerges. This striking feature is called Persistent Slip Bands (PSBs). As pointed by Mughrabi for pure Copper single crystals, PSBs form in early saturation response stage if shear plastic strain amplitude higher than about $6 \times 10^{-5}$ (the regime B in Figure II.19). The motivation of the formation of PSBs is to accommodate higher plastic deformation. It predicts that PSBs can accommodate about $7.5 \times 10^{-3}$ which is much larger than that by ordinary vein/channel structure (about $10^{-4}$). At a constant strain-controlled loading condition, dislocations rearrange to form loop patches of veins (or walls) which quite effectively restrict the movement of mobile dislocations, leading to less medium to carry plastic deformation. Dislocation loop patches become unstable after a large number of cycles, and break into a structure of ladders of dislocation veins (or walls) in some locations. The collective movement of dislocation veins (or walls) can accommodate higher plastic strain. The volume fraction of ladder walls is about 10% of the PSBs volume. Upon further loading (or when plastic strain amplitude increases), the volume fraction occupied by PSBs increases to reach a value of 100%. Finally, when the volume fraction of PSBs is about 100%, PSBs transform into a more stable structure, a cellular structure which indicates more homogeneous plastic deformation [19, 70]. Another mechanism to form the cellular structure is due to the activity of secondary slip [71-73]. According to this mechanism, the activity of secondary slip makes connections between veins/walls, which finally transform the vein/channel structure into cellular structure. As a consequence, the dislocation cellular structure is shortened upon further loading due to more activity of secondary slip, finally becoming more equiaxed.
II.7. Plastic amplitude dependence of microstructure condition

During plastic deformation, dislocations are required to generate to accommodate the imposed plastic strain. The higher the imposed plastic strain amplitude, the higher the number of dislocations which need to be generated. The dislocation density plays an important role for dislocation evolution upon further loading. A high dislocation density promotes the interaction of dislocations, resulting in the formation of dislocation junctions, i.e. the part of partial dislocations is recombined. This induces partial dislocations to cross slip easier [74, 75]. Spatial dislocation structures, e.g. veins/walls, cells, consequently form earlier for higher plastic strain amplitude conditions.

![Graph](image)

Figure II.19: The cyclic stress-strain curve (CSSC) of single copper crystals.

(Reprinted from [19] with permission from Elsevier)

In 1978, Mughrabi studied the dependence of the stress on the saturation response stage (which is often attained after a certain number of cycles) on the imposed plastic strain of pure Cu [19]. There are three distinct regimes of the curve of saturation stress with respect to the shear plastic strain amplitude (the cyclic stress-strain curve (CSSC), Figure II.19). There are
also 3 microstructural regimes corresponding to the three regimes of plastic strain amplitude dependence of saturated stress.

**Regime A:** Single Cu crystals experience very low shear plastic strain amplitudes, $\gamma_p^{\alpha} \leq 6 \times 10^{-5}$. In this regime, the saturation stress increases with the increase in the plastic strain amplitude ($\gamma_p^{\alpha}$). Dislocations are present in the form of dense bundles (or veins) on the primary slip planes. Bundles (or veins) becomes denser for a higher $\gamma_p^{\alpha}$, which more effectively restricts the movement of mobile dislocations. Cyclic loading with a higher plastic strain amplitude consequently induces a higher saturation stress.

**Regime B,** $6 \times 10^{-5} \leq \gamma_p^{\alpha} \leq 7.5 \times 10^{-3}$: the saturation stress keeps constant over this range of shear plastic strain amplitude. The dislocation condition at the saturation stage consists of veins or walls, low dislocation density channels and the Persistent Slip Bands (PSBs). The PSBs can accommodate quite high plastic strain locally, resulting in the softening response preceding the saturation response stage. Over this range of strain amplitude, the saturation stress does not change due to the linear increase of the volume fraction (which is occupied by PSBs) with increasing in the $\gamma_p^{\alpha}$. This volume fraction is expected to reach 1 as $\gamma_p^{\alpha}$ increases to the end of the regime B.

**Regime C,** $\gamma_p^{\alpha} \geq 7.5 \times 10^{-3}$: The saturation stress increases again with the plastic strain amplitude. A dislocation cell structure is observed in the saturation stage. The cell structure is believed as a consequence of the transformation of PSBs once their volume fraction reaches 1, i.e. the homogeneous plastic deformation condition. With a higher plastic strain amplitude, the fraction of cell is higher and the shape of cells more equiaxed. In addition, the cell size decreases with increasing plastic strain amplitude.

The similar shape of the CSSC is also found for other FCC materials, e.g. Ni, Ag [76]; and even for polycrystal AISI 316L steel [77].
II.8. Grain boundary dependence

The analogous form of three regimes of plastic strain amplitude dependence of the saturation stress to the case of single crystal metals was also reported for polycrystal FCC materials [77-79]. The regime II of polycrystals was observed to occur in a short range of plastic strain amplitude for coarse grain materials [77, 79]. However, the analogy of the CSSC between single and poly-crystal materials was received a criticism which states that there is no plateau regime of CSSC of polycrystal FCC materials [76].

Nevertheless, the effect of grain boundaries on the dislocation evolution is quite evident because grain boundaries on the one hand play as a source of dislocation generation, on the other they are also effective obstacles to the movement of dislocations. The requirement to preserve the material continuity from grains to grains during plastic deformation asks the activity of multiple slip systems, leading to the formation of spatial dislocation structures earlier.
II.9. Temperature dependence

Temperature is an important factor on the dislocation condition during cyclic loading owing to its role on the mobility of defects. At an elevated temperature, the mobility of dislocation is higher, thus leading to the cross slip activation earlier. The spatial dislocation structures are observed in earlier time. Over a certain range of temperatures, solute atoms (e.g. C, N) however have a high mobility to aggregate around mobile dislocations, resulting in the formation atmospheres of solute atoms around dislocation cores [44, 80, 81]. Therefore, it requires a higher stress to move dislocations together with the dragging atom atmospheres. Once the applied stress increases, dislocations can escape the atmospheres. In other words the dislocations can move easier, resulting in a reduction in stress. However, the speed of dislocation motion will reduce with reducing stress which, in turn, could make dislocations be anchored again by solute atom atmospheres. This event repeatedly takes place during loading, causing the serration of the flow stress. This phenomena is called the dynamic strain ageing (DSA) [80].
II.10. Environmental dependence

Other environmental factors, e.g. oxidation, corrosion etc., strongly influence the fatigue life of materials because fatigue damage development is a surface phenomenon. The cyclic deformation response and microstructural evolution of AISI 316L during cyclic loading in vacuum were investigated by Gerland [67, 82]. The prolongation of fatigue life can lead dislocations to form a more well-organised cellular structure at room temperature, or to the formation of a corduroy structure in the regime of DSA.
II.11. Relationship between dislocation condition and cyclic deformation response

In the beginning of plastic deformation, the increase in dislocation density and the interactions between dislocations and obstacles cause the rise of stress, resulting in the primary cyclic hardening response. Depending on the plastic strain amplitude, the dislocation density required to accommodate imposed plastic strain is different. Higher imposed plastic strain amplitudes require higher numbers of dislocations, resulting in higher stress.

Once dislocations start re-arranging to form veins/walls and PSBs, there are two competing processes taking place simultaneously. One is the cyclic hardening caused by the formation of harder structures such as veins/walls because the veins (or walls) of dense dislocations effectively hinder the movement of dislocations. Another process is the cyclic softening caused by: (1) the ease in the cross slip of screw dislocations, (2) the formation of dislocation-low-density regions (i.e. channels) within which dislocations can move quite freely, and (3) the activation of PSBs which can accommodate much higher plastic strain than the matrix of veins and channels. Since the volume fraction of channels is much higher than that of veins (or walls), the cyclic softening effect takes over the hardening effect in the beginning of the rearrangement process of dislocations. Consequently, a cyclic softening response stage follows the primary cyclic hardening stage for almost poly-crystal FCC materials, in particular for more planar slip materials.

After a number of cycles, the formation of a cellular structure of dislocations leads to more restricted areas for dislocation movement compared to the wall/channel structure. This strengthens the material, which can compensate the softening effect. The cellular structure is also more stable than the wall/channel matrix or PSBs, leading to a saturation response stage after the softening phase.
Chapter III: Cyclic deformation response of AISI 316L

Abstract:
The material of interest in this study is the AISI 316L steel which is widely used in engineering industry applications due to its outstanding corrosion resistance at low temperatures, and high oxidation resistance and superior creep resistance at high temperatures. In particular the material is utilised in the primary cooling circuits of nuclear power plants in Switzerland for which the working temperature is about 300°C. Understanding the fatigue behaviour of the material under such conditions is required to verify the safety factors applied to the material in service duty. Cyclic deformation response of the material has been therefore studied at different strain amplitudes, different strain rates and different temperatures, which are close to the working conditions of the primary cooling circuit. This chapter provides detailed information of mechanical behaviour during cyclic loading at temperatures of 20 and 300°C, at strain rates of $10^{-3}$ and $10^{-4}$s$^{-1}$, and at strain amplitudes of ±0.25%, ±0.4% and ±0.7%.
III.1. Strain-controlled fatigue testing

Table III.1: Mechanical testing matrix

<table>
<thead>
<tr>
<th>Strain amplitude</th>
<th>±0.7%</th>
<th>±0.4%</th>
<th>±0.25%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strain rate (s⁻¹)</td>
<td>10⁻³</td>
<td>10⁻⁴</td>
<td>10⁻³</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[K]</td>
<td>20</td>
<td>300</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>[293]</td>
<td>[573]</td>
<td>[293]</td>
</tr>
<tr>
<td>No. of tests</td>
<td>7</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>(with/out interruption)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Cylindrical testpieces, with a parallel length of 20mm, a gauge diameter of 8mm and with threaded end-grips, were machined from the pipe (Table I.1 and Figure I.2) with an axial orientation, in accordance with the requirement of ISO 12106 (Figure III.1a). The testpieces were fatigue loaded with constant total strain amplitudes of 0.25, 0.4 and 0.7%, strain rates of 10⁻³ and 10⁻⁴ s⁻¹, at 20 and 300°C (i.e. 293 and 573K) (Table III.1) using a servo-controlled electromechanical 50kN machine manufactured by Walter+Bai AG, Switzerland. An extensometer with a gauge length of 15mm was attached to the testpiece parallel length to control and measure strain during cyclic loading (Figure III.1b). The acquisition program recorded: (1) the force and displacement of selected cycles from which the stress-strain hysteresis loop of every selected cycle could be plotted (Figure III.1c), and (2) the maximum (and minimum) stress of every cycle during cyclic loading until failure (Figure III.1d). For each testing condition, the endurance test was first conducted to identify the general cyclic deformation response, e.g. the curve of maximum stress versus number of cycles (Figure III.1d). Based on the analysis of this curve, a number of interrupted tests were proposed (Figure III.1d, and Table III.1).
Figure III.1: (a) Geometry of testpiece. (b) the representation of strain-controlled scheme. (c) The stress-strain hysteresis loop of a cycle. (d) The indication of interrupted tests.
III.2. Data recording

Data of the force-displacement (i.e. stress-strain hysteresis loop) of every cycle from 1 to 1000 was recorded. After the 1000th cycle and the 10000th cycle, the data of every 10 cycles and 50 cycles were respectively recorded.
III.3. Analysis procedure of mechanical testing data

Parameters representing the elasto-plastic behaviour of the material of every recorded cycle (e.g. elastic modulus \((E)\), yield stress \((\sigma_0)\), elastic strain \((\varepsilon_e)\), plastic strain \((\varepsilon_p)\), effective stress \((\sigma_E)\) and back stress \((X)\)) were calculated from the stress-strain hysteresis loops of recorded cycles (Figure III.2). Matlab-based subroutines were developed to calculate these parameters. Detailed procedures of how to calculate these parameters are described in the following.

![Stress-strain hysteresis loop](image)

Figure III.2: A scheme of quantitative measurements of parameters representing elasto-plastic behaviour.

III.3.1. Elastic modulus

Elastic modulus \((E)\) is an intrinsic property of a material. It is defined as the ratio of stress and corresponding strain in a region below the limit of proportionality \((\sigma_0)\). An
accurate determination of elastic modulus is extremely important for engineering
design and for providing reliable parameters for material constitutive equations.

In this study, the apparent elastic modulus of a hysteresis loop is determined by a least
squares fitting function built in to a Matlab program. However, the accuracy of the
elastic modulus measurement of a recorded data set depends on the fitting length and
the determination of stress range. A preliminary analysis of a stress-strain curve is
necessarily performed in order to identify an appropriate stress range over which data
is fitted to measure $E$ modulus. Minimum and maximum fitting lengths are also
determined in the preliminary analysis. Within the approximately identified stress range,
the first fitting procedure is done with the minimum fitting length but changing the fitting
region over the identified range. Afterwards, the fitting length increases, and the
procedure is repeated until the maximum fitting length is reached. The elastic modulus
is finally measured as the average of elastic modulus values obtained from every fitting
step.

### III.3.2. Yield stress

Yield stress ($\sigma_0$) is a stress level at which a material starts to plastically deform. Yield
stress is the stress required to let dislocations overcome the local resistance to the
dislocation motion. A poly-crystal material consists of many small volumes whose
plastic deformation commences at different stress levels; thereby it is difficult to define
the exact value of yield stress. In macroscopic scale, the yield stress is conventionally
defined as the yield strength which corresponds to a certain degree of plastic strain. In
this study, $\sigma_0$ is the offset yield stress corresponding to a plastic strain of 0.05% for
stress quantitative characterisation of internal stresses, and to a plastic strain of 0.01%
for modelling.
III.3.3. Elastic strain

Elastic strain ($\varepsilon_e$) corresponding to measured elastic modulus at zero-stress position equals to $\sigma/E$ below the limit of proportionality ($\sigma_0$) (Figure III.2).

III.3.4. Plastic strain range and accumulated plastic strain

The width of a hysteresis loop at the zero-stress position gives the value of the plastic strain range, $\Delta\varepsilon_p$. The accumulated plastic strain ($P = 2\Delta\varepsilon_p$) is also given.

III.3.5. Effective stress and back stress

Effective stress ($\sigma_E$) consists of the initial yield stress ($\sigma_E^0$) and the increment of yield stress ($\sigma_E^R$) during loading. The initial yield stress is required to make dislocations overcome the resistance to dislocation movement which includes (i) the intrinsic resistance, e.g. periodic lattice resistance, and (ii) the extrinsic resistance, e.g.:  

- the phonon drag: the resistance to the movement of dislocations due to their interaction with thermal vibrations of the lattice (Lattice phonons).
- the restriction of movement induced by the presence of other dislocations at the initial condition of the material.

$\sigma_E^R$ associates with a change in the motion resistance due to the variation of short-range interactions of dislocations. For FCC materials, the lattice-related resistances are negligible compared to other resistances, in particular resistances relating to: the short-range interaction between dislocations themselves (e.g. Lomer-Cottrell sessile
dislocation junctions [50, 51]) and other defects, e.g. solute atoms [40, 53, 83] (Figure III. 3).

![Image](image-url)

Figure III. 3: Short-range interactions of dislocations. (a) Straight Lomer-Cottrell junctions due to the dislocation-dislocation interactions. (b) Dislocations pinned due to their interactions with solute atoms.

![Image](image-url)

Figure III.4: Evolution of yield surface in principle stress space.

In the principal stress co-ordination, yield stress is therefore the radius of the yield surface. The change in effective stress is responsible for the expansion or contraction of the yield surface [84] (Figure III.4).
Back stress (X) associates long-range interactions of collective dislocations which arise due to inhomogeneous plastic deformation between microstructural heterogeneities of materials on different scales, e.g. grains, sub-grains, cell blocks, and families of dislocation wall/channel structure (Figure III.5) [40, 85-91]. Microstructural heterogeneities, which are either present before loading or formed during loading, possess different strength with respect to loading direction, resulting in inhomogeneous deformation.

Detailed relationships between internal stresses and microstructural condition of AISI 316L will be thoroughly discussed in Chapter V.

**Quantification of effective stress and back stress**

A simple method to quantitative values of back stress and effective stress from the hysteresis loop (as indicated in Figure III.2) was first introduced by Cottrell [40] and then is widely applied, e.g. [91-93]. At the start of the plastic deformation during a reverse loading of a cycle, the back stress (generated during the preceding half cycle)
acts in the same direction of the reverse stress. Therefore, the yield stress ($\sigma_0$) can be calculated as

$$\sigma_0 = \sigma_E - X$$  \hspace{1cm} (III.1)

However, after getting the maximum value of strain (back stress gets its maximum at the same time), back stress counteracts instead of aiding the deformation. The maximum stress is therefore equal to the sum of effective stress and back stress, i.e.:

$$\sigma_{\text{max}} = \sigma_E + X$$  \hspace{1cm} (III.2)

Eqns. (III.1) and (III.2) lead to:

$$\sigma_E = (\sigma_{\text{max}} + \sigma_0)/2$$  \hspace{1cm} (III.3)

and

$$X = (\sigma_{\text{max}} - \sigma_0)/2$$  \hspace{1cm} (III.4)

### III.3.6. Flow stress serration

Since flow stress serrations are the only visible manifestation of DSA in the stress-strain hysteresis loop, their characteristics provide the most suitable variables to study evolution of the phenomenon during cyclic loading. A Matlab subroutine was developed to analyse changes in stress-strain hysteresis in particular changes in flow stress serration characteristics. The subroutine is able to detect any consistent instability in flow stress based on the fact that the serration causes a consistent and sudden change in the strain hardening rate. The serrated flow stress of every stress-strain hysteresis loop was quantified by the maximum value of stress drop (called max DSA transient) and the period (called serration length) over which serrated flow stress happens (Figure III.6). Higher values of max DSA transient and serration length indicate stronger DSA activity.
III.3.7 Quantification of the cyclic deformation response

Rates of change for different cyclic deformation response stages were calculated to quantify the cyclic response of the material (Figure III.7). These rates were determined as slopes of the most linear parts during different stress response stages in the $\sigma_{\text{max}} \log N$ scale. Positive values of the rate represent cyclic hardening response, whereas negative values represent cyclic softening response. The rates of change of effective stress, back stress were calculated in the similar way.
Figure III.7: The definition of the quantification of cyclic deformation response.
(Note: h1 represents for the first cyclic hardening.)

**III.3.8. Loop shape parameter**

The loop shape parameter ($V_H$) is the ratio of the area of a hysteresis loop with respect to the area of the blue rectangle circumscribed by line pairs through the maximum stress and the minimum stress of the cycle (Figure III.8a). The loop shape parameter used in this study is slightly different to that used in other studies in which the denominator area is represented by a red rectangle circumscribed by a line pair going through the peak stresses and a line pair parallel to linear parts of the hysteresis loop (Figure III.8a) [19, 94]. The loop shape parameter used in the latter approach provides information concerning the hardening modulus during plastic deformation. A smaller value of $V_H$ indicates a more pointed shape of loop and a higher rate of work hardening. The change of loop shape parameter could also provide information concerning the activation of persistent slip bands (PSBs) which mainly cause crack
initiation during cyclic loading [19]. There are only insignificant changes in elastic moduli when fatigue cracks do not have a strong influence on the deformation response (Figure III.8b). The period of negligible influence of fatigue cracks on the deformation response contributes to almost 60 to 70% of fatigue life for smooth testpieces. The two types of loop shape parameter are therefore equivalent. When fatigue cracks become long and have an obvious influence on the material response, the former approach used in this study can provides more information concerning fatigue crack development.

Figure III.8: (a) Hysteresis loop shape parameters represented i) by a red parallelogram circumscribed by a line pair going through the peak stresses and a line pair parallel to linear parts of a hysteresis loop [19], and ii) by a blue rectangle bounded by two line pairs going through the peak stresses. (b) Elastic moduli of ±0.7%·10³s⁻¹·20°C.
III.4. Cyclic deformation response of AISI 316L at 20°C and 300°C:

III.4.1. General

Figure III.9: (a) Cyclic deformation response at a strain rate of $10^{-3}$ s$^{-1}$. The stress-strain hysteresis loops at (b) 20°C, and at (c) 300°C

The cyclic peak tensile (maximum) stress is plotted with respect to number of cycles for $\varepsilon^a$ of ±0.7, ±0.4, and ±0.25% at 20 and 300°C (Figure III.9 and Figure III.10). The material exhibits a complex behaviour during cyclic loading at both temperature conditions: firstly cyclic hardening, followed by cyclic softening, and finally an almost stabilised response stage (for all tests at 20°C, and for 300°C at ±0.7%) or secondary hardening (for ±0.25 and ±0.4% at 300°C) before failure. The main differences in the cyclic deformation response of the material at 300°C compared to that at 20°C are: firstly, flow stress is serrated at 300°C (Figure III.9c, and Figure III.10d); secondly the strain rate sensitivity of cyclic deformation response is positive at 20°C (Figure III.10a),
but negative at 300°C (Figure III.10b); thirdly the hardening rate of the first cyclic hardening response phase is higher than that at room temperature (Table III.4); and fourthly there is a secondary hardening response phase at 300°C which occurs for lower strain amplitude conditions (i.e. ±0.4, and ±0.25%) (Figure III.9a, Figure III.10c and Table III.3).

Figure III.10: (a) Positive strain rate sensitivity at 20°C. (b) Negative strain rate sensitivity at 300°C. (c) Secondary cyclic hardening. (d) Weak serration in the flow stress at ±0.25%·s⁻¹-300°C.

Table III.2, Table III.3 and Table III.4 give general quantitative information about the plastic strain amplitude; and about the three cyclic response stages, e.g. the duration of the three stages. In addition for each transient stage (cyclic hardening or softening stage), the maximum stress at selected cycles corresponding to the onset, the middle value of maximum stress range, and the end of a response stage are also given.
Table III.2: Detailed information of cyclic deformation response of the material at 20°C.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>$\varepsilon_p^a$</th>
<th>Duration</th>
<th>Cyclic hardening stage</th>
<th>Cyclic softening stage</th>
<th>Cyclic saturation $\sigma_{\text{midlife}}^{\text{max}}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(first cycle) (%)</td>
<td></td>
<td>Cycle</td>
<td>Max stress (MPa)</td>
<td>Cycle</td>
</tr>
<tr>
<td>±0.7%</td>
<td>10$^{-3}$ s$^{-1}$</td>
<td>0.5832</td>
<td>Start</td>
<td>1</td>
<td>225.7</td>
</tr>
<tr>
<td>20°C</td>
<td></td>
<td></td>
<td>Middle</td>
<td>4</td>
<td>270.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>End</td>
<td>31</td>
<td>313.2</td>
</tr>
<tr>
<td>±0.4%</td>
<td>10$^{-3}$ s$^{-1}$</td>
<td>0.2878</td>
<td>Start</td>
<td>1</td>
<td>214.8</td>
</tr>
<tr>
<td>20°C</td>
<td></td>
<td></td>
<td>Middle</td>
<td>5</td>
<td>238.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>End</td>
<td>39</td>
<td>259.8</td>
</tr>
<tr>
<td>±0.25%</td>
<td>10$^{-3}$ s$^{-1}$</td>
<td>0.1492</td>
<td>Start</td>
<td>1</td>
<td>194.2</td>
</tr>
<tr>
<td>20°C</td>
<td></td>
<td></td>
<td>Middle</td>
<td>3</td>
<td>205.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>End</td>
<td>21</td>
<td>214.2</td>
</tr>
</tbody>
</table>
Table III.3: Detailed information of cyclic deformation response of the material at 300°C

<table>
<thead>
<tr>
<th>Conditions</th>
<th>$\epsilon^0_p$</th>
<th>Duration</th>
<th>Cyclic hardening</th>
<th>Cyclic softening</th>
<th>Cyclic saturation</th>
<th>Cyclic secondary hardening</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cycle</td>
<td>Max stress (MPa)</td>
<td>Cycle</td>
<td>Max stress (MPa)</td>
</tr>
<tr>
<td>$\pm0.7%$</td>
<td>0.6260</td>
<td>Start</td>
<td>1</td>
<td>115.7</td>
<td>70</td>
<td>272.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Middle</td>
<td>8</td>
<td>197.9</td>
<td>314</td>
<td>250.2</td>
</tr>
<tr>
<td>$10^{-3}\text{s}^{-1}$</td>
<td></td>
<td>End</td>
<td>70</td>
<td>272.6</td>
<td>1500</td>
<td>227.8</td>
</tr>
<tr>
<td>$\pm0.4%$</td>
<td>0.3386</td>
<td>Start</td>
<td>1</td>
<td>103.8</td>
<td>91</td>
<td>192.0</td>
</tr>
<tr>
<td>$10^{-3}\text{s}^{-1}$</td>
<td></td>
<td>Middle</td>
<td>9</td>
<td>147.7</td>
<td>190</td>
<td>182.5</td>
</tr>
<tr>
<td>$300^\circ\text{C}$</td>
<td></td>
<td>End</td>
<td>91</td>
<td>192.0</td>
<td>891</td>
<td>172.2</td>
</tr>
<tr>
<td>$\pm0.25%$</td>
<td>0.1849</td>
<td>Start</td>
<td>1</td>
<td>107.3</td>
<td>86</td>
<td>147.9</td>
</tr>
<tr>
<td>$10^{-3}\text{s}^{-1}$</td>
<td></td>
<td>Middle</td>
<td>8</td>
<td>128.0</td>
<td>150</td>
<td>146.1</td>
</tr>
<tr>
<td>$300^\circ\text{C}$</td>
<td></td>
<td>End</td>
<td>86</td>
<td>147.9</td>
<td>250</td>
<td>144.4</td>
</tr>
</tbody>
</table>
Table III.4: The rates of cyclic hardening and softening stages

<table>
<thead>
<tr>
<th>Condition</th>
<th>$\frac{d \sigma_{\text{max}}}{d (\log N)}$</th>
<th>$\frac{d \sigma_{E}}{d (\log N)}$</th>
<th>$\frac{d X}{d (\log N)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>±0.7% -10^{-3}s^{-1}-20°C</td>
<td>60.1</td>
<td>-20.4</td>
<td>Not applicable</td>
</tr>
<tr>
<td>±0.7% -10^{-3}s^{-1}-300°C</td>
<td>107.6</td>
<td>-40.4</td>
<td></td>
</tr>
<tr>
<td>±0.4% -10^{-3}s^{-1}-20°C</td>
<td>31.3</td>
<td>-19.9</td>
<td></td>
</tr>
<tr>
<td>±0.4% -10^{-3}s^{-1}-300°C</td>
<td>51.4</td>
<td>-33.2</td>
<td>5.8</td>
</tr>
<tr>
<td>±0.25%-10^{-3}s^{-1}-20°C</td>
<td>9.5</td>
<td>-12.0</td>
<td>-</td>
</tr>
<tr>
<td>±0.25%- 10^{-3}s^{-1}-300°C</td>
<td>22.5</td>
<td>-8.4</td>
<td>7.9-16.3</td>
</tr>
</tbody>
</table>

III.4.2. The duration of cyclic deformation response stages

Since the material shows high phase stability against phase transformation during low cycle fatigue loading at an intermediate total strain amplitude [95], the cyclic hardening and softening responses of AISI 316L are only associated with the movement of dislocation. Moreover, because the cyclic hardening and softening responses of metallic materials are not noticeably influenced by the presence of microstructurally small cracks (even if they are already formed) for smooth specimens (Figure III.8b); the transient cyclic responses are characteristics of the material response as a consequence of the underlying physical mechanism, i.e. the dislocation movement. This is underpinned by a fact that the accumulated plastic strain to reach the middle of the cyclic hardening stage is respectively about 13% and 11% of that accumulated...
throughout the hardening stage for all strain amplitude conditions at 20°C and at 300°C. The plastic strains accumulated during the primary cyclic hardening ($p_{h1}$) and softening ($p_s$) stages are respectively shown in Figure III.11 and Figure III.12. These have been used to establish the relationships between the plastic strain amplitude of the first cycle ($\varepsilon_p^{a}$) and plastic strains accumulated during primary hardening ($p_{h1}$) (and during softening ($p_s$)). These relationships are shown in Figure III.11 and Figure III.12.

![Graph](image URL)

**Figure III.11**: Accumulated plastic strain to reach the end of cyclic hardening stage at 20°C (black points), and 300°C (red points).

The relationship between the plastic strain amplitude of the first cycle and the accumulated plastic strain during the hardening response stage is very well represented by an exponential law, i.e. $\varepsilon_p^{a} = a_{h1} \exp(b_{h1} \times p_{h1})$. The value of $b_{h1}$
(which is the slope of this fitting function in the \( \log(\varepsilon^a_p), p \) scale) is shown in Figure III.11. The value of \( b_{h1} \) for the 20°C condition is about double that for the 300°C condition. This means that cross slip activity takes place more slowly at 300°C. The rate of cross slip activity during cyclic loading at 300°C is half that at 20°C (see also Sect. IV.4.6). This differs from usual behaviour of dislocations where the mobility of dislocations is usually higher for higher temperatures, i.e. cross slip can take place earlier and faster at higher temperatures due to the thermal activation. The reason for this contrast is the strong interaction between dislocations and solute atoms (C, and N) at 300°C, which inhibits the cross slip of dislocations, and causes the dynamic strain ageing phenomenon (as briefly mentioned in Sect. II.9 and will discussed in detail in Sect. IV.4). The mechanism of DSA for this material at 300°C and its consequence on the material deformation response will be discussed in detail in Sect. IV.4 and Sect. V.3.

Figure III.12: Accumulated plastic strain to reach the end of cyclic softening stage at 20 and 300°C.
The relationship between $\varepsilon_p^h$ and the $P$, however cannot be represented by a same exponential law for 20 and 300°C conditions (Figure III.12), which is due to the incidence of a secondary cyclic hardening stage. The secondary cyclic hardening stage associates with the interaction between dislocations and a corduroy structure of vacancy (Sect. IV.4.7). This indicates that the manner of dislocation movement under the presence of different sources of obstacles strongly affects the duration of cyclic deformation response stage, in particular the cyclic softening response.
III.5. Quantitative analysis of max stress, back stress and effective stress

The cyclic deformation response is quantitatively characterised by the rates of change of maximum stress, of back stress, and of effective stress (Figure III.7), which are given in Table III.5. The rates of change of these stresses are of great importance since they can shed light on the roles of back stress and effective stress (and their associated microstructural features) on the material cyclic response. The rate of change of back stress decreases with reducing strain amplitude (Figure III.13). This is also the case for effective stress (Figure III.14). This means that lower imposed plastic strain induces less strain incompatibility (i.e. lower back stress); and less effective stress due to lower numbers of dislocations. Moreover, the rates of change of back stress during the primary cyclic hardening and softening response stages are greater than those of effective stress, in particular at 20°C (Table III.5). It means that the back stress is mainly responsible for the material cyclic response, in particular at room temperature.

The rates of change of effective and back stresses are both higher at 300°C compared to those for equivalent conditions at 20°C (Table III.5, Figure III.13 and Figure III.14). The difference in the rate of change of effective stress between 20 and 300°C is however higher than that of back stress. This indicates that the difference between cyclic deformation response of the material at 20°C and 300°C is mainly due to the change in effective stress. In particular, during the secondary cyclic hardening at 300°C, the rate of change of effective stress is interestingly much larger than that of back stress (Table III.5). In other words, the change in effective stress is mainly responsible for secondary cyclic hardening response at 300°C.

The relationships between internal stresses (effective stress and back stress) and microstructural condition will be exhaustively discussed in Chapter V in order to establish their links.
Figure III.13: The change in back stress during cyclic loading at 20°C (black lines) and at 300°C (red lines).

(Note: the back stress for ±0.7% -10^{-3} s^{-1} -20°C after 2000 cycles seemingly exhibits secondary cyclic hardening behaviour. However, this incidence is probably not due to the material deformation response since there is already an obvious influence of the presence of fatigue cracks on the deformation response after 2000 cycles (Figure III.8b)). This incidence is also observed for other testing conditions, which is coincident with the start of a significant divergence of apparent E moduli during reverse loading from compressive and tensile peak stresses (see also Sect. VII.3.2).
Figure III.14: The change in effective stress during cyclic loading at 20°C and at 300°C.
Table III.5: The rates of change of maximum stress, back stress and effective stress (Figure III.7)

<table>
<thead>
<tr>
<th>Conditions</th>
<th>$\frac{d\sigma_{\text{max}}}{d(\log N)}$</th>
<th>$\frac{dX}{d(\log N)}$</th>
<th>$\frac{d\sigma_{E}}{d(\log N)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclic hardening</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>±0.7% $10^3$ s$^{-1}$ 20°C</td>
<td>60.1 ± 2.5</td>
<td>54.5 ± 2.4</td>
<td>3.7 ± 2.3</td>
</tr>
<tr>
<td>±0.7% $10^3$ s$^{-1}$ 300°C</td>
<td>107.6 ± 1.5</td>
<td>89.1 ± 5.1</td>
<td>13.7 ± 4.7</td>
</tr>
<tr>
<td>±0.4% $10^3$ s$^{-1}$ 20°C</td>
<td>31.3 ± 0.6</td>
<td>25.4 ± 2.3</td>
<td>4.8 ± 2.0</td>
</tr>
<tr>
<td>±0.4% $10^3$ s$^{-1}$ 300°C</td>
<td>51.4 ± 0.3</td>
<td>37.4 ± 1.0</td>
<td>10.7 ± 1.4</td>
</tr>
<tr>
<td>±0.25% $10^3$ s$^{-1}$ 20°C</td>
<td>9.5 ± 1.1</td>
<td>8.1 ± 2.5</td>
<td>2.8 ± 2.7</td>
</tr>
<tr>
<td>±0.25% $10^3$ s$^{-1}$ 300°C</td>
<td>22.5 ± 0.2</td>
<td>16.4 ± 2.4</td>
<td>5.3 ± 2.2</td>
</tr>
</tbody>
</table>

| Cyclic softening    |                                        |                        |                                  |
| ±0.7% $10^3$ s$^{-1}$ 20°C | −20.4 ± 0.4                         | −19.3 ± 4.1           | −1.4 ± 4.1                       |
| ±0.4% $10^3$ s$^{-1}$ 300°C | −40.4 ± 0.2                         | −26.2 ± 0.9           | −13.9 ± 0.8                      |
| ±0.4% $10^3$ s$^{-1}$ 20°C | −19.9 ± 0.1                          | −14.0 ± 0.4           | −6.3 ± 0.4                       |
| ±0.25% $10^3$ s$^{-1}$ 20°C | −12.0 ± 0.2                          | −6.4 ± 0.9            | −6.1 ± 0.9                       |

| Secondary cyclic hardening |                                        |                        |                                  |
| Not applicable            |                                        |                        |                                  |
III.6. Analysis of loop shape

The loop shape of hysteresis stress-strain loop is quantitatively characterised by the change in loop shape parameter (Figure III.8a). Significant changes in hysteresis loop shape during cyclic loading have been observed (Figure III.15). At the onset of cyclic deformation, the hysteresis loop shape is quite rectangular, indicating the work hardening rate is small. However it becomes more pointed during the primary cyclic hardening, indicating an increase in the hardening coefficient. During the softening stage, the loop shape slightly changes back to be more rectangular. Correspondingly, the magnitude of the loop shape parameter first decreases in value during cyclic hardening, then slightly increases during softening. The increase after the decrease of $V_H$ is believed to be associated with the activation of PSBs [19]. The loop shape parameter significantly decreases again after a large number of cycles which is found to be coincident with a considerable divergence between elastic moduli measured during reverse loading transients from compressive and tensile peak stresses. The elastic modulus divergence after a large number of cycles is normally due to the presence of fatigue cracks [96].

Figure III.15: The evolution of loop shape parameter during cyclic loading at 20°C (solid lines) and at 300°C (dashed lines)
### III.7. Analysis of the serration of flow stress at 300°C

Flow stress serration was weakly experienced, and disappeared very quickly during cyclic loading with a strain amplitude of ±0.25% (Figure III.9d). In contrast, the flow stress serration is clearly seen at conditions of ±0.7 and ±0.4% (Figure III.16 and Figure III.17). The following section therefore concentrates on the change in DSA activity for the two latter conditions by means of examining change in the stress serration characteristics, e.g. the max DSA transient and the serration length quantities (Figure III.6).

#### III.7.1. Flow stress serration during cyclic loading

The max DSA transient and serration length quantities rapidly decrease during the first cyclic hardening stage, and almost vanished during the cyclic softening stage (Figure III.16). Figure III.16 & Figure III.17 show that the lower the strain amplitude, the lower and the quicker the reduction of the stress serration quantities during the first two cyclic response stages. This means that DSA rapidly becomes less active during the first two cyclic response stages. It is however interesting to note that the serration quantities increasingly become more significant after the cyclic softening phase. In particular the tendency is clearly observed during secondary cyclic hardening for the ±0.4%-condition (Figure III.17a, b, d). The change in the stress serration activity results from different short range interactions between dislocations and solute atoms, which is fully revealed in Sect. IV.4.
Figure III.16: Evolution of (a) serration length and (b) max DSA transient during reverse loading transients from compressive peak stress with respect to number of cycles at condition of ±0.7% -10^{-3}s^{-1} -300°C. Serration flow stress activity of selected cycles during (c) the first and second cyclic response stages, and (d) the almost stabilised response stage. Open circles show where the serration activity starts.
III.7.2. Flow stress serration during reverse loading transients from compressive and tensile peak stresses

It is worthwhile to note that the activity of serration flow stress is different during tensile going transients (i.e. reverse loading from the compressive peak stress) and during compressive going transients (i.e. reverse loading from the tensile peak stress) (Figure III.18). The difference is more pronounced during the softening response stage. Higher strain amplitudes result in more distinct differences. The DSA transient and serration
lengths (Figure III.6) are in general greater during reverse loading transients from tensile peak stress than during reverse loading transients from compressive peak stress, except towards the end of fatigue life. Upon further loading, the differences become negligible. The different activity of serration flow stress is found to be due to the hydrostatic stress dependence of point defects which will be explained in detail in Sect. IV.4.8.

Figure III.18: Difference of activity of stress serration characteristics between during reverse loading transients from compressive peak stress (tensile going) and from tensile peak stress (compressive going) at conditions of: (a-b) ±0.4%-10^{-3}s^{-1} -300°C, and (c-d) ±0.7%-10^{-3}s^{-1} -300°C.
III.8. Concluding remarks on cyclic deformation response

AISI 316L exhibits a complex cyclic deformation response at 20 and 300°C, i.e. cyclic hardening followed by cyclic softening and finally an almost stabilised cyclic response or secondary cyclic hardening response stage before failure. The main differences in the cyclic deformation response of the material at 300°C compared to that at 20°C are:

i) flow stress is serrated at 300°C (and not at 20°C),
ii) the strain rate sensitivity of cyclic deformation response is negative at 300°C while positive at 20°C,
iii) the hardening rate of the first cyclic hardening phase at 300°C is higher than that at room temperature,
iv) there is a secondary hardening phase at 300°C which occurs for lower strain amplitude conditions (i.e. ±0.4, and ±0.25%). This does not occur at 20°C.

At 300°C, flow stress serration is examined in every cycle in order to study DSA activity. Flow stress serration is weakly experienced, and disappears very quickly during cyclic loading at condition of ±0.25%. In contrast, the flow stress serration is clearly seen at conditions of ±0.7 and ±0.4%. During primary cyclic hardening, stress serrations are initially pronounced, before gradually disappearing. There is then almost no incidence of stress serration during cyclic softening, in particular for low strain amplitude tests. However, serrated flow stress reoccurs towards the end of fatigue life. Moreover, there is a slight difference in DSA characteristics with respect to the loading direction, i.e. during reverse loading transients from compressive and tensile peak stresses.

The cyclic deformation response is quantitatively characterised by the rates of change of maximum stress, of back stress, and of effective stress. The rate of change of back stress decreases with reducing strain amplitude. This is also the case for effective stress. This means that lower imposed plastic strain induces less strain incompatibility; and less effective stress due to lower numbers of dislocations. Moreover, the rate of
change of back stress during the primary cyclic hardening and softening responses are
normally greater than that of effective stress, in particular at 20°C. This indicates that
the back stress is mainly responsible for the material cyclic response. In contrast, the
secondary cyclic hardening response after certain numbers of cycles is mainly due to
the change in effective stress.
Chapter IV: Microstructural evolution and its relationship with cyclic deformation response

Abstract

The cyclic deformation response of the material was comprehensively characterised in Chapter III in terms of mechanical response. In order to understand the material long term response (which provides the basis to develop an evolutionary constitutive model), it is essential to i) obtain in-depth understanding of microstructural evolution during cyclic loading, and ii) establish relationships between the microstructural evolution and internal stress variables (Chapter V). The microstructural evolution of the material during different cyclic deformation response stages is systematically characterised and given in this Chapter. In order to investigate the microstructural evolution, a series of tests were performed and interrupted at selected numbers of cycles in the different stages of cyclic response. At each interruption point, specimens were examined by electron microscopy (SEM, TEM). The microstructural evolution was then qualitatively correlated with the material cyclic mechanical behaviour.
IV.I. Introduction

In order to understand the material characteristics responsible for the complex cyclic deformation response (Chapter III), it is paramount to investigate the microstructural evolution and its relation to the cyclic deformation response. There have been intensive studies of the relationship between cyclic deformation properties and the microstructural condition of austenitic stainless steels [65-67, 71, 77, 97-102]. However, these studies have mainly concentrated on investigating the microstructures existing at failure for different strain amplitudes [65, 71, 77, 97-99, 101-104]. These investigations have shown that the dislocation structures induced by cyclic loading depends on imposed plastic strain amplitude. Specifically, if a well-annealed austenitic stainless steel is cyclically strained at low plastic strain amplitudes, the dislocation structure is present in the form of planar arrangements (associated with stacking fault, pile-ups etc.) until failure, which is almost similar to the initial condition except the dislocation density is higher at the end of life. At intermediate plastic strain amplitudes, persistent slip bands and veins coexisting with dislocation-free regions (i.e. channels) are observed at failure. At high plastic strain amplitudes, well-organised structures, namely wall/channel or cellular structures, are observed at failure.

While there are existing comprehensive investigations on dislocation structure evolution and its correlation with cyclic deformation response [67, 82, 105-107], these are either more focused on the microstructural condition present after a high number of cycles or at failure. It is shown in this paper that the fatigue response of austenitic stainless steels like some other metals [60] significantly changes during the first cycles. For AISI 316L at temperatures between 20 and 300°C, the cyclic hardening stage occurs only during the first cycles, occupying a small fraction of lifetime (Figure III.9a). For some conditions, the softening response stage also occupies a relatively small fraction of life. Subsequently, dislocation structures are present in a complex form comprising co-existing sub-domains of well-defined wall/channel structure, fragmented wall/channel structure, and cells [105]. The evolution of microstructure throughout life
and in particular during the first cycles therefore deserves to receive more careful examination.

Transmission electron microscopy (TEM) provides only a 2-D projection of a small portion of the actual microstructure along a certain beam direction. It can therefore be difficult to appreciate the development of actual dislocation structures, in particular when the complex form (which comprises co-existing sub-domains of well-defined wall/channel structure, fragmented wall/channel structure, and cells) is present. There have been a number of studies which examine the spatial arrangement of dislocations during cyclic loading by observing thin foils whose planes are oriented to certain angles with respect to the loading axis [65-67, 71, 98]. These studies provide a comprehensive knowledge of the spatial arrangement of dislocations. However, the existence of different families of structure types, fragmented wall/channel structures etc. during cyclic loading has not been clearly explained by models developed in these investigations. A part of this chapter is therefore given to construct a new 3-D model to represent the observed changes in dislocation structures for high strain amplitude.
IV.1. Experimental methods

Tested specimens were sectioned in parallel to the loading axis using a diamond cutting machine in order to prepare samples for microstructural observations. The samples were mechanically polished to produce thin plates of 0.1mm thickness which were then punched out to produce discs having a diameter of 3mm. These were marked to indicate the loading direction. The discs were then electrolytically polished using a double jet device (TenuPol5) with an electrolyte solution of acetic acid and perchloric acid to provide specimens for TEM investigation. Thereafter, samples were observed in a Philips CM30 transmission electron microscope at 300kV with a double tilt holder (Figure IV.1a). The whole area around the thin hole in the centre of a TEM sample was firstly examined to choose about 3-5 grains which were the most representative of the microstructural condition of the fatigued testpiece. The mark of the loading direction (LD) was aligned along the holder axis in order to preserve the loading direction during observations. Selected area electron diffraction and Kikuchi patterns were used to identify the grain orientation relative to the loading axis. In addition, the samples were also investigated at lower magnifications in order to more effectively extend the scope of examination using a scanning electron microscope equipped with a scanning transmission electron detector (Figure IV.1b).

Figure IV.1: (a) Transmission electron microscope (TEM), and (b) Scanning electron microscope (SEM)

(Courtesy of ZEM, Empa)
Certain TEM images were further processed in order to highlight the diffraction contrast of dislocations. Scanning lines were drawn horizontally and vertically over these images and the number of point intersections with dislocations counted. For each image, a total of 18 scanning lines were plotted as nine vertical lines and nine horizontal lines to form a squared grid (Figure IV.2).

The dislocation density was then determined using the following formula:

\[
\rho = \frac{1}{t} \frac{1}{N} \sum_{i=1}^{N} \left( \frac{n_i}{L_i} \right) \tag{IV.1}
\]

where: \( n_i \) is the number of intersections of the \( i^{th} \) scanning line with dislocations, \( L_i \) is the length of the \( i^{th} \) scanning line, \( t \) is the thickness of the foil from which the image was obtained, \( N \) is the number of scanning lines (with \( N = 18 \)). The dislocation density was determined from TEM images with magnifications as low as possible (normally at magnifications from 3300x to 6000x) to increase the statistical significance. There were about 5 images taken for each examined grain. For each scanning line, the number of

Figure IV.2: The representation of dislocation density measurement.
interaction points usually varied from 20-80 points. Counted interaction points per each image was approximately about 1000 (at least 500 intersection points were counted). Numerous dislocation measurements were double checked by the author and his ETHZ colleague, Jan Lukas Eurich. Dislocation quantification is given by the average value and upper and lower bound limits. The thickness of the TEM foil was measured from the convergent-beam electron diffraction (CBED) or thickness fringes associated with the extinction distance. The importance of basing such quantities on a statistically significant observation population is recognised, and every effort has been made to limit the uncertainties in the procedure adopted here, and described above.

Dislocation structure evolution of the material was determined by examining samples obtained from an as-received (solution treated) specimen, and testpieces fatigued up to selected cycles, respectively, corresponding to the midpoint of the hardening stage, the maximum peak stress, the midpoint of the softening stage, the end of the softening stage (or the beginning of the stabilised stage), the midlife (or the midpoint of the stabilised stage) and the end of life, TEM images were often obtained under multi-beam diffraction conditions. Dislocation evolution was normally compared by using images obtained at the same multi-beam condition. Images obtained with different beam directions and for a range of tilting angles were also analysed in order to reveal dislocation arrangements in 3-D. The loading direction (LD) was preserved and indicated in TEM images by arrowed lines.

To characterise the grain orientation relative to the loading axis, selected area electron diffraction (SADP) and Kikuchi line patterns were used. Dark-field (DF) and Weak-beam (WB) techniques were also performed to thoroughly investigate fine crystal defects.
IV.2. Microstructure in the as-received condition

Figure IV.3: Dislocation condition in the as-received pipe steel

TEM observation revealed that dislocations in the as-received condition were mainly present in the form of planar structures (e.g. regularly planar dislocation arrays, dislocation pile ups close to grain boundaries (Figure IV.3a-b), stacking faults (Figure IV.3c)). In addition, there were predominant primary slip planes in the as-received condition (Figure IV.3a), which were caused by the hot metal forming process and an associated increase in dislocation density. Subsequent solution annealing could not annihilate all dislocations generated by metal forming and the total dislocation density in this condition was quite high. The total dislocation density was measured from TEM images to be about $6.5 \times 10^{13}$ 1/m$^2$. 
IV.3. Microstructural evolution and its relation to the cyclic deformation response at 20°C

In the as-received condition, dislocations are mainly present in the form of planar structures, e.g. regular arrays of dislocations, stacking faults etc. At the beginning of cyclic loading, grain-to-grain misorientations inducing plastic strain incompatibilities between grains, i.e. at an inter-granular scale, result in a significant increase in dislocation density close to the grain boundaries to preserve the continuity of material. In contrast, there is a slightly increase in dislocation density inside grains. The grain-to-grain strain incompatibilities are relieved upon further loading thanks to the activity of secondary slip (including cross slip) upon further loading. Secondary slip activities promote dislocation interactions, resulting in: 1) an increase in the annihilation rate of dislocations, leading to a slight decrease in dislocation density; 2) the rearrangement of dislocations to form lower and more stable energetic configurations, i.e. dislocation high/low density regions, to further relieve the grain-to-grain strain incompatibilities. Dislocation structures, i.e. dislocation high/low density regions, are strongly dependent on imposed plastic strain. At the end of life, dislocation structures are less well organised for lower strain amplitude tests. This is because: i) the planar character of dislocation motion persists to a higher life fraction, and ii) the tendency of the formation of well-organised wall/channel structures is increasingly diminished. Although the development of dislocation structures relieves the grain-to-grain incompatibilities, it raises other strain incompatibilities between dislocation high/low density regions on a finer scale, i.e. intra-granular scale. The stabilised condition of these strain incompatibilities is established upon further loading once dislocations seek their most stable configuration under a given testing condition. Detailed information of microstructural evolution at different testing conditions is given in the following.
IV.3.1. Microstructural evolution at the ±0.7%-condition

![Microstructural images and graph](image-url)

Figure IV.4: Dislocation evolution and its corresponding cyclic deformation behaviour of 316L steel at ±0.7%-10^{-3}s^{-1}-20°C

The evolution of microstructure of the material during cyclic loading at ±0.7% is summarised in context with its corresponding cyclic deformation response in Figure IV.4. During cyclic hardening, dislocation density significantly increases, in particular in regions close to grain boundaries (Figure IV.4a, b). At the end of the cyclic hardening stage, dislocation-dense sheets were observed (Figure IV.4c). Dislocations then rearrange during the softening stage due to the strong activation of secondary slip, finally resulting in the formation of dislocation walls/channels (Figure IV.4d). Upon further loading, a strong activity of multiple slip at this strain amplitude condition facilitates wall/channel structures to develop into a cellular structure towards the end of life (Figure IV.4e-f). The activity of secondary slip systems creates more connections between walls, resulting in a labyrinth structure (Figure IV.4g). Towards the end of fatigue life, cellular structure consequently becomes more equiaxed. Cellular structures more effectively confine the movement of mobile dislocations than wall/channel structures. They thereby gradually compensate for the softening effect caused by the formation of channels and the activity of persistent slip bands (PSBs) which were observed from the later part of the softening response stage (Figure IV.4h). More
detailed information of dislocation evolution during cyclic hardening, softening and stabilised response stages is given below.

IV.3.1.1. Hardening stage

![Dislocation condition during the cyclic hardening stage](image)

Figure IV.5: Dislocation condition during the cyclic hardening stage

The hardening stage occupies a relatively small fraction of fatigue life (about 30 cycles, i.e. less than 1% of total fatigue life). During the hardening stage (after 8 cycles), dislocations were still mainly present in the form of planar structures as observed by TEM in the [001] and [-112]-beam directions (Figure IV.5). The dislocation state was similar to that in the as-received condition, except the planar structures in this condition were more tangled (i.e. within planar structures the dislocation density was higher and dislocation interactions were more complex), and stacking faults were less often observed. Figure IV.4b and Figure IV.5a show dense dislocation arrangements close to a grain boundary, whereas dislocations in the middle of the same grain were much less dense and showed more obvious evidence of planar structures (Figure IV.5b).
At the end of the hardening stage (after 30 cycles), the dislocation density was observed to be much higher than that of the as-received condition. Dislocations were dense, tangled and distributed homogeneously (Figure IV.4c). In addition, TEM images showed that the incidence of primary and secondary slip was almost equal over a grain (Figure IV.6a), which results in the homogeneous distribution of dislocations at this stage. However, the activation intensity of the slip planes was not uniform, especially near grain boundaries (Figure IV.6b). Predominant active slip planes divided grains into sub-domains in which there were less intensely active slip planes (Figure IV.6c). In addition, within sub-domains, further interactions of slip planes resulted in low dislocation density regions surrounded by high dislocation density regions (Figure IV.6c). The total dislocation density measured from TEM images in the [011]-beam direction was about $2.4 \times 10^{14}$ $1/m^2$ at the end of the hardening stage.

**IV.3.1.2. Softening stage**

During the softening stage (after 90 cycles), thick veins considered as pre-mature walls, separated by long dislocation-free regions (i.e. channels) were often observed...
The thick veins/walls at midway through the softening stage further developed into well-organised walls which were thinner and denser as observed at the end of the softening stage (Figure IV.7a and b). Labyrinth structures were already seen at the end of the softening stage (Figure IV.7a). Persistent slip bands (PSBs) started to be observed in this condition. Since veins/walls consisted of very dense dislocation structures, it was difficult to measure their contained dislocation density. It was not feasible to measure the total dislocation density in this condition from multibeam TEM images.

Figure IV.7: Dislocation structures at the end of the softening phase with respect to the loading direction. (a) Labyrinth structure, (b) Wall/channel structure
IV.3.1.3. Stable mechanical response stage

At midlife and the end of life, dislocation structures were rather identical (Figure IV.4e-f). In general, well-organised dislocation walls separated by clear channels were observed in planes closely parallel to the primary slip planes (Figure IV.4e). Walls and channels were shorter than those observed after 700 cycles. In a grain context, however, the evidence clearly showed that there were different families of wall/channel sub-domains (Figure IV.8). There are 3 different wall/channel sub-domains shown in Figure IV.8a. The first sub-domain occupies a small area in the lower left, the second one occupies the upper area, and the last one is present in the lower right area of Figure IV.8a. In addition, the first and second sub-domains are orientated in the same direction but separated by a narrow boundary, whereas the third sub-domain is inclined to the others and fragmented structures are observed between them. Persistent slip bands are often observed at the both conditions (Figure IV.4h). A labyrinth structure is also seen at this stage (Figure IV.4g).

![Figure IV.8: Different families of wall/channel sub-domains and their boundaries](image-url)
A series of tilted images was obtained (Figure IV.9) from a specimen at the midlife condition in order to clearly reveal the 3-dimensional arrangement of dislocation structures. Some marked positions and areas were used to follow the change of dislocation features at these locations during tilting. Figure IV.9 shows that cells at locations 1, 2, 3 and 4 appeared to change into elongated cells and finally into walls and channels with tilting. In contrast, the elongated cell structure at location 5 appeared to change to an organised wall/channel structure, and further to become a not so well-organised wall/channel structure after tilting to 70° and then 82° respectively. Areas marked by a dashed rectangle (containing wall/channel structures) and an ellipse (containing cells) changed into a labyrinth structure and a fragmented structure, respectively.
Figure IV.9: Influence of tilt angle on dislocation structures at the mid-life cycle. Tilting axis was the holder axis (or the loading direction). Positions and areas were marked in order to follow the change of dislocations features during the tilting series.

Although microstructures were almost identical for the midlife and the end of life conditions, there were some differences between the respective dislocation structures. Firstly, the cell walls were thinner at the end of life. Secondly, dislocations in channels are lower towards the end of life. Thirdly, the cell length progressively decreased with number of cycles. Finally, round cell walls and shorter channels were more often observed at the end of life than those at the midlife condition.
IV.3.1.4. Evolution of dislocation condition from planar structures to a cellular structure

The initial condition of the steel is believed to play an important role in subsequent microstructural evolution which in turn strongly affects the mechanical response of that material during cyclic loading. Having low stacking fault energy, dislocations in the virgin material prefer to be present in planar forms [108]. In addition, nitrogen atoms can also result in the formation of short range ordering of nitrogen and chromium atoms [64, 109, 110], which in turn can contribute to the planar structure of dislocations [63, 111]. Under cyclic loading, the dislocation density first increases as a consequence of the imposed strain amplitude. The increase in dislocation density makes dislocation interaction become more complicated, with the subsequent formation of more tangled structures (Figure IV.5). However, low stacking fault energy and short range ordering restricts cross-movement of dislocations. As a result, dislocation structures during early cycles are still planar structures (e.g. stacking faults, regular pile ups and planar tangled bundles).

During cyclic loading, dislocations move and can annihilate themselves by interacting with opposite sign dislocations. In addition, since the incidence of tangled structures increases with dislocation density and such structures can act as obstacles for the movement of dislocations, the formation of tangled structures induce local stress which promotes the activation of cross slip and secondary slip planes [65, 112, 113]. Moreover, the formation of dislocation junctions lowers the activation energy for cross slip [74, 75]. Cross-slip can further enhance dislocation annihilation. As a result, the total dislocation density tends to reach its equilibrium state, i.e. remaining almost constant after reaching a maximum level.

Although the total dislocation density does not increase with cycle number, dislocation structures progressively change under the activation of cross slip and secondary slip planes. Under the influence of cross slip, screw dislocations cancel opposite-sign screw dislocations and shorten edge dislocations in primary slip planes resulting in dislocation-free regions, i.e. channels [65]. In addition, the activation of secondary slip
planes facilitate interactions between dislocations which are responsible for the formation of dislocation-dense regions, i.e. veins or walls, as shown in Figure IV.4d. Upon further cyclic loading, dislocation walls become denser. Meanwhile screw dislocations continuously annihilate other opposite-sign screw dislocations in channels, resulting in clear channels and thinner walls. Since the dislocation density within the walls is much higher than that within the channels, the formation of wall/channel structures creates high plastic incompatibilities. It is necessary to form such a structure in order to relieve these high plastic incompatibilities. In practice, their relief can be achieved by the further activation of secondary slip systems. Secondary slip creates more connections between walls and further transforms the wall/channel structure into more restricted areas of cellular structures. Simultaneously, dislocations are also mutually trapped while moving, leading to another source of cellular structure formation. Upon further cyclic loading, the combined effects of activation of secondary slip systems and the mutual trapping of dislocations cause a decrease in the cell size and cellular length. Consequently, there are more equiaxed cellular structures formed during cyclic loading.

IV.3.1.5. 3-D construction of dislocation evolution from planar structures to a cellular structure

Spatial dislocation structures are shown to form after a certain number of cycles, i.e. from the end of the hardening regime. Since TEM images are a 2-D projection of the actual structure, the obtained TEM images are different depending on the direction of observation. As a result, it is difficult to rebuild the 3-D arrangement of dislocation structures. Nevertheless, the tilting function of TEM enables the 3-D dislocation structures to be fully revealed by images taken for a series. After tilting a sample through 82° in a series of steps, Figure IV.9 shows that cells change either into wall/channel structures or disappear. In addition, either wall/channel structures or labyrinth structures can be observed at the same location during tilting. Based on the
observations, a 3-D representation of the developing dislocation structure is proposed in Figure IV.10 and involves:

- The formation of a lamella-like structure of dislocations in primary slip planes during the hardening stage (Figure IV.11) with some regions having higher numbers of dislocations (Figure IV.3a, and Figure IV.4b). During further cyclic loading, the lamella-like structure is first divided into sub-domains by the activation of secondary slip systems (Figure IV.6b, c and Figure IV.10a).

- The formation of more sub-domains upon further cyclic loading after the late hardening stage (Figure IV.6c) and during the softening stage (Figure IV.12). In each sub-domain, dislocations rearrange to form high and low density structures (i.e. walls and channels) (Figure IV.4d). Dislocation wall and channel structures differ between sub-domains in terms of their dimension and/or their axial/crystallographic orientations (Figure IV.8, Figure IV.12 and Figure IV.10b).

- The transformation in each sub-domain of walls and channels into cellular structures during the early softening stage, but more definitely from the late softening stage. Cellular structures become more equiaxed during further cyclic loading (Figure IV.4e-f, Figure IV.10c).
Figure IV.10: Schematic representation of development of dislocation structures in AISI 316L steel during LCF loading. (a) Dislocation planar structures. (b) Different sub-domains as a consequence of multiple slip activity. (c) Different families of cellular structure formed at different sub-domains.

Figure IV.11: Primary plane of dislocations during the cyclic hardening stage.
A lamella-like structure such as that shown in Figure IV.10a consists of some connections between dislocation-dense sheets. During further cyclic loading, the lamella-like structure is divided into, for example, five sub-domains identified as 1 to 5 in Figure IV.10b. The first three sub-domains are mainly created by the activation of cross-slip and secondary slip systems. The fourth sub-domain is created because there are always some regions, especially close to grain boundaries, in which dislocation density during the first cycles is already higher (e.g. dislocation pile-ups close to grain boundaries, Figure IV.4b). High dislocation density areas close to grain boundaries cause internal stresses, thereby they can induce cross-slip and secondary slip [65, 112, 113] to take place earlier than in the remaining structure, and this can result in the formation of different sub-domains. After a certain number of cycles, the lamella-like structures in the first sub-domains to form are still evolving into cellular structures, whereas others are fully occupied by cellular structures (Figure IV.10c). In addition, cellular structures in sub-domains 1, 2, 3, and 4 are different in their shape while their axial orientations are the same. By contrast, a cellular structure in sub-domain 5 (e.g. the region bounded by a dashed red ellipse in Figure IV.12) is inclined
to the others. A front-view in Figure IV.4e, g shows longitudinal sections of cellular structures in sub-domains 3 and 4. Meanwhile, a top-view in Figure IV.4f shows a cross section of cellular structure in sub-domains 1, 2, and 3.

The proposed scenario provides a plausible explanation for the development of discontinuous wall/labyrinth structures, different families of cellular structures, and the fragmentation of cellular structures. In particular, the wall discontinuities shown in Figure IV.7a and Figure IV.8a can result from the activation of secondary slip planes. Upon further loading, connections between walls can be made at the position of the discontinuities, resulting in the shortening of channels and the formation of labyrinth structures (Figure IV.4g).

Since sub-domains evolve in different ways as demonstrated in Figure IV.10a, b, consequent cellular structures are slightly different from sub-domain to sub-domain, causing different families of cellular structure as observed in Figure IV.8a. The fragmentation of wall/channel structures (Figure IV.8a) has been observed elsewhere [105]. The existence of different cellular families gives an alternative explanation for the existence of fragmented regions. The interaction of two different families of cellular structure can cause a fragmented region between them if they are orientated in different directions, or a narrow boundary if they are both orientated in the same direction (Figure IV.8a). A coexistence between wall/channel structures and fragmented structures can therefore be observed if one looks at a longitudinal section of two cellular structure families.

IV.3.1.6. Relation of microstructural condition to cyclic deformation response

The total dislocation density is observed to increase from $6.5 \times 10^{13}$ $(1/m^2)$ to $2.4 \times 10^{14}$ $(1/m^2)$ after 30 cycles. This increase in dislocation density and the consequent formation of tangled planar structures is responsible for cyclic hardening during the first cycles. Although the formation of well-organised structures leads to difficulties in measuring the total dislocation density by multi-beam TEM observations, the total
dislocation density is believed to remain almost constant after achieving a maximum. As a consequence, the cyclic hardening rate is lower and the mechanical response of the material is stabilised. Dislocations rearrange into specific structures which gradually take over from dislocation density in the determination of mechanical response. Since the dislocation density within channels (which have a larger volume fraction than that of the walls) is very low, dislocations can move easily within channels; resulting in the cyclic softening response. Cellular structures are believed to more effectively confine the movement of mobile dislocations than wall/channel structures. In addition, upon further loading, cell length decreases, thereby gradually compensating for the softening effect caused by persistent slip bands and channels; resulting in the final saturation of the cyclic response.

IV.3.2. Strain amplitude dependence

Since imposed plastic deformation is lower for smaller strain amplitude conditions (e.g. ±0.4 and ±0.25%), dislocations develop in different ways to that of the ±0.7%-condition (Figure IV.13 and Figure IV.14):

- Dislocations (both boundary dislocations and interior dislocations) are less dense (Figure IV.13b-c), resulting in smaller rates of the first cyclic hardening response (Table III.4).
- Dislocation configuration exhibits a strong tendency towards planar character for lower strain amplitude conditions. The tendency persists to a higher life fraction when the strain amplitude decreases. For instance, stacking faults were still observed until the middle of life at the ±0.25%-condition (Figure IV.14a). Towards the end of fatigue life, a relative portion of material is occupied by ill-organised dislocation walls (Figure IV.14b) or dislocation carpets (Figure IV.14c), i.e. dense of planar dislocation networks, which is similar feature discussed by Kuhlmann-Wilsdorf [114].
- At the end of life, dislocation structure is less well-organised (Figure IV.13e-f, Figure IV.14). The effectiveness of dislocation structures on the restriction of
dislocation movement is therefore smaller. In addition, the plastic strain incompatibility between dislocation high/low density regions is also less. It explains why the peak stress at the saturation stage is lower for smaller strain amplitude conditions (Figure III.9, and Table III.2).

Figure IV.13: Microstructural evolution at the ±0.4%-condition

Figure IV.14: Microstructural evolution for the ±0.25%-condition: (a) Preponderant planar glide activity enduring until late in the fatigue life. (b) Very ill-organised dislocation veins or (c) dislocation carpet formation at the end of fatigue life.
IV.3.3. Concluding remarks on the microstructural evolution and mechanical response during cyclic loading at 20°C

During strain-controlled cyclic loading at room temperature, the microstructure of AISI 316L steel evolves from a planar dislocation structure into a lamella-like structure. Dislocations then rearrange during the softening stage due to the strong activation of secondary slip (including cross slip), finally resulting in the formation of dislocation-high/low-density structures. For high strain amplitudes, dislocation-high/low-density regions finally evolve into a cellular structure which becomes more equiaxed if the material can endure longer. The sub-division of grains by dominant slip systems during the first cycles and the activation of secondary slip (including cross slip) cause the formation of different families of dislocation structures (e.g. different families of cellular structure for the ±0.7% -10^{-3}s^{-1}-20°C condition). A detailed investigation of dislocation condition development from planar structures to the cellular structure during different mechanical stages has provided the basis for a new 3-D representation of dislocation structure evolution in AISI 316L for the ±0.7% condition. The 3-D representation well accounts for the complexity of dislocation structures of the alloy during cyclic loading under highly imposed plastic strain amplitude.

The cyclic hardening response is found to be associated with an increase in the total dislocation density, whereas the softening response is related to the strong activity of secondary slip (which includes cross slip). Secondary slip results in the rearrangement of dislocations (which causes the formation of dislocation-high/low-density regions, e.g. dislocation walls/channels). The more organised and most stable structure under a given testing condition at 20°C is responsible for the stabilised stage of the cyclic response. More detailed links between microstructural condition and cyclic deformation response are established when the physical interpretations of internal stresses are rigorously studied in Chapter V.

Lowering strain amplitude induces different dislocation structures which results in different material cyclic response. Dislocations (both boundary dislocations and interior
dislocations) are less generated, resulting in smaller rates of the first cyclic hardening response. Dislocation configuration exhibits a strong tendency to planar character for lower strain amplitudes. The tendency persists to a higher life fraction when the strain amplitude decreases. Consequently, the effectiveness of dislocation structures on the restriction of dislocation movement is therefore smaller for lower strain amplitudes. In addition, the plastic strain incompatibility between dislocation high/low density regions is also smaller for more planar dislocation structures. As a result, the peak stress at the saturation stage is lower for smaller strain amplitude conditions.
IV.4. Dynamic strain ageing of AISI 316L at 300°C and its influence on cyclic deformation response

AISI 316L is expected to be susceptible to dynamic strain ageing in the temperature range of 200 to 600°C, and with strain rates of $10^{-4}$ to $10^{-2}$ s$^{-1}$, leading to its complex cyclic deformation response (see also Sect. V.3). At 300°C, solute atoms can segregate into dislocation cores resulting in solute atom atmospheres around dislocations. Solute atom atmospheres can then restrict dislocation movement, resulting in the starvation of mobile dislocations, thereby requiring additional stress in order to unlock arrested dislocations. Once applied stress reaches a critical value, a mass of dislocations move, causing a drop in stress. Serrated flow takes place as a consequence of the repeatedly pinning and unpinning of mobile dislocations due to the presence of solute atom atmospheres [40].

Figure IV.15: Cottrell atmosphere of Boron around an edge dislocation in B-doped FeAl. The element distributions of Fe, Al, and B were obtained by atom probe microscopy

(Reprinted from [115] with permission of Annual Reviews).
A way to form solute atom atmospheres around dislocations (Figure IV.15) due to elastic strain interaction and the mechanism of dynamic strain ageing (DSA) was first comprehensively examined by Cottrell and Bilby [80]. The formation of solute atom atmospheres in short-range-ordered clouds had already been proposed by Snoek [81] before the hypothesis of Cottrell. An alternative way to form solute atom atmospheres was suggested by Suzuki [44]. Suzuki anticipated that solute atoms preferably segregate into faulted areas surrounded by partial dislocations due to chemical interaction. The Suzuki hypothesis therefore plays an important role for materials having low stacking fault energy, in particular austenitic stainless steels.

The original Cottrell DSA theory was further developed by combination with theories involving vacancy-assisted solute diffusion [40] and pipe diffusion [116]. This development successfully explained why flow stress serration is observed after a certain degree of plastic deformation, i.e. critical strain. However, the original theory cannot explain the negative strain rate sensitivity of materials observed in the DSA regime. Based on the idea of Sleeswyk [117], McCormick [118] and then van den Beukel [119] pointed out that ageing should take place during the time over which mobile dislocations arrested at obstacles. Once mobile dislocations approach dislocation forests in which there are abundant point defects, they will be arrested for some time ($t_a$). If mobile dislocations are kept for a sufficiently long time in dislocation forests, point defects can diffuse into mobile dislocations and then form atmospheres to strengthen locking mobile dislocations. The time required to let point defects diffuse into mobile dislocations is called the ageing time, $t_a$. Depending on the relative relation of $t_a$ to $t_r$, stress serration can take place or not. During cyclic loading, $t_r$ increases due to the increase in dislocation density. In contrast, solute atoms (C, N) can diffuse more quickly with the assistance of vacancies and the presence of dislocations. $t_a$ therefore decreases due to increases in both dislocation density and vacancies during plastic deformation of crystals. When this mechanism is combined with a dislocation junction strengthening mechanism, it can fully explain the negative strain rate sensitivity since lower strain rate causes longer arrest times, and dynamic strain ageing will be more likely to happen [120, 121]. All existing theories of DSA imply that DSA should be
increasingly more active when vacancy concentrations and the strengths of the
dislocation junctions increase. Sect. III.7.1 however shows that DSA first becomes less
active during cyclic hardening and softening periods, and then becomes more active
again. This evidence requires further investigation to reveal the mechanism of DSA,
and this is the purpose of the second half of this chapter.

Although there have been a number of studies investigating the low cycle fatigue
behaviour of austenitic stainless steels in the DSA regime [104, 111, 122-125], it is
noted that there has been not enough attention paid to how DSA evolves during cyclic
loading. The most comprehensive study regarding the general cyclic deformation
response and corresponding microstructural evolution in the regime of DSA for AISI
316L is the series of investigations of Gerland and his co-workers conducted in vacuum
[82, 105-107]. Changes in the stress-strain hysteresis loop characteristics (which are
given in Sect. III.7) can provide detailed information of DSA. In the second part of this
chapter, the microstructural evolution is carefully examined to throw light on the DSA
mechanism exhibited by AISI 316L during cyclic loading at 300°C.

In addition, special attention is given to the development of the corduroy structure since
this was previously attributed to the secondary cyclic hardening of AISI 316L during
cyclic loading at elevated temperature in vacuum without any irradiation [82]. The first
observation of corduroy structure consisting of rows of loops or black spots was in fact
obtained by Adamson and his colleagues when they studied the neutron irradiation
damage in Zirconium alloys [126]. The presence of corduroy structure in several
materials including AISI 316L is normally attributed to irradiation damage [127].
Gerland and his colleagues however showed that the structure can form during cyclic
loading without any irradiation damage but in vacuum. In this study, it is shown for the
first time that the corduroy structure can also form during cyclic loading at 300°C in
laboratory air.
IV.4.1. Microstructural evolution at ±0.7%-10^{-3}s^{-1}-300°C

Figure IV.16: Microstructural evolution along with cyclic deformation response at ±0.7%-10^{-3}s^{-1}-300°C. (a-e) Dislocation structures evolve from planar to cellular. (f) Finally a corduroy structure is observed towards the end of fatigue life.

There are some similarities in the microstructural evolution of the material for cyclic loading at ±0.7%-10^{-3}s^{-1}-300°C and ±0.7%-10^{-3}s^{-1}-20°C (which are described in Sect. IV.3), such as: 1) a significant increase in dislocation density during the first cycles (Figure IV.16a-b), 2) the re-arrangement of dislocations to form dislocation tangles during the cyclic softening response stage (Figure IV.16c), and 3) the formation of a dislocation cellular structure towards the end of fatigue life (Figure IV.16d). The incidence of point defects is however much more profuse at 300°C than at room temperature (Figure IV.16b). In addition, there is evidence of localised deformation destroying the well-organised cellular structure towards the end of fatigue life (Figure IV.16e). A striking feature observed at 300°C is well-aligned tiny point defects along certain orientations which are occasionally seen in cell interiors (Figure IV.16f). This feature is examined in more detail in the next section.
**IV.4.2. Strain amplitude dependence**

When reducing the strain amplitude at 300°C, the same strain amplitude dependence of the microstructural evolution is observed as it is previously described at the room temperature condition ([Sect. IV.3.2](#)), i.e.:

1. Lower numbers of dislocations when reducing strain amplitude,
2. Dislocations arrange in a more planar way for lower strain amplitude conditions,
3. Dislocation structure is less well-organised at the end of life, e.g. discontinuously dense dislocation walls are observed instead of dislocation cellular structure at late cyclic loading for the ±0.4% condition ([Figure IV.17d](#)), and there was no dislocation wall/channel structures for the ±0.25% condition towards the end of life ([Figure IV.18](#)).

![Figure IV.17: Microstructural evolution along with cyclic deformation response at ±0.4%-10^{-3}s^{-1}-300°C. (a-d) Dislocation structures evolved from planar structures to ill-organised discontinuous walls. (e-f) Corduroy structure was observed in dislocation-poor regions during the slightly secondary cyclic hardening response stage.](#)
Moreover, numerous point defects are also observed at lower strain amplitudes of ±0.25 and ±0.4% (10^{-3}s^{-1}-300°C) (Figure IV.17b and Figure IV.18a, c) similar to the ±0.7%-10^{-3}s^{-1}-300°C condition. The striking feature of well aligned point defect clusters is more preponderant, and increasingly develops during the secondary cyclic hardening stage (Figure IV.17d-f). This feature is called corduroy structure, which has already been observed in the same material cyclically loaded in vacuum by Gerland [106, 107].

Figure IV.18: Microstructural condition at late the fatigue life for ±0.25%-10^{-3}s^{-1}-300°C condition.

IV.4.4. Corduroy structure and corduroy contrast

As mentioned previously cyclic straining at 300°C produces profuse point defects. Excess point defects thermodynamically tend to coalesce to form corduroy structure during further loading. Corduroy structure is seen to form after a number of cycles at all conditions tested at 300°C, in particular in low strain amplitude tests. The following discussion of the formation of corduroy structure and the presence of corduroy contrast is mainly influenced by results from the ±0.4%-10^{-3}s^{-1}-300°C test, but it also represents the development of the structure at the other testing conditions. Different types of corduroy structure are observed during the fatigue of AISI 316L at 300°C. The first type (mainly faulted dislocation loops) forms as a result of the coalescence of tiny point defects along a common orientation (Figure IV.19a-b). This type of corduroy structure
consists of a regular alignment of faulted dislocation loops in \{111\} planes along a <112> direction. It is characterised by its homogeneous distribution and its fine dimensional structure (Figure IV.19b), e.g. its width of about 5nm and its inter-distance between lines of dislocation loops of 18nm. The second type (Figure IV.19d-e) comprises mainly unfaaulted dislocation loops which can result from the movement of superjogs and the activity of multiple slip (Figure IV.19c) or interactions between dislocations and faulted dislocation loops to form perfect dislocation loops [128, 129]. The second type consisting of dislocation loop lines with a width of about 10-20nm and a spacing of about 30-70nm apart is coarse and irregularly-distributed in contrast to the first type. Both types of corduroy structure can act as obstacles to dislocation movement (Figure IV.19f).

Figure IV.19: Point defects produced during straining and their aggregation to mainly form corduroy structure (a, b), or dislocation loops which can results from multiple co-planar slip activity (indicated by an arrow in c) or from the movement of super jogs to form a coarse corduroy structure (d, e). Corduroy structure can restrict the movement of dislocations (f).
Figure IV.20: Corduroy structure and corduroy contrast in (a) bright field, and (b, c) dark field images at exactly the two beam condition. (c-d) Change in corduroy contrast with change in $g$ during tilting. Lines of less pronounced point defects appear exactly where corduroy contrast had been observed. The arrow in (c-e) indicates the reference point.

Corduroy contrast, which is alternating contrast intensity fringes, is usually observed along with corduroy structure. Gerland et al. [82, 106, 107] investigated both corduroy structure and corduroy contrast in AISI 316L during cyclic loading. They found that corduroy contrast was normally not evident in regions of well-organised corduroy structure. They however failed to reveal any microstructural feature associating with this contrast. Figure IV.20a & b show a region where both corduroy structure and corduroy contrast were observed. The corduroy contrast slightly changed with diffraction vector, $g$, during tilting in agreement with other observations. It is interesting to note that lines of less pronounced point defects appeared exactly where the
corduroy contrast had occurred during further tilting (Figure IV.20c & d). This evidence indicates that corduroy contrast is due to very small point defects.

IV.4.5. Locking mechanisms & cyclic deformation response

Figure IV.21: Lomer-Cottrell locking during cyclic loading of AISI 316L at (a) room temperature, and (b) 300°C. (c) Profuse point defects produced during cyclic straining at 300°C form atom atmospheres which restrict movement of mobile dislocations (d).

At room temperature, in the case of a coarse grain size and without the presence of precipitates, it is widely accepted that dislocations in FCC materials are mainly locked
by the formation of Lomer-Cottrell sessile dislocations during slip [50, 51, 130]. This is called the Lomer-Cottrell locking mechanism. During cyclic loading of AISI 316L, straight dislocation segments of Lomer-Cottrell sessile dislocation junctions are often observed at both room temperature and 300°C (Figure IV.21a&b), which indicates that Lomer-Cottrell locking is active at both temperature conditions. However, in the presence of profuse point defects (Figure IV.21c) which facilitate the formation of solute atom atmospheres to restrict the movement of dislocations (Figure IV.21d), an additional locking mechanism due to solute atom atmospheres comes into effect at 300°C. Consequently, a higher rate of the first cyclic hardening at 300°C compared to 20°C (Table III.4) results from the activity of both Lomer-Cottrell locking and solute atom atmosphere locking mechanisms. The Lomer-Cottrell locking mechanism is mainly active at 20°C.

Solute atom atmospheres can be either: 1) in a disordered form, i.e. Cottrell and Suzuki atmospheres [44, 80]; or 2) in short-range ordered form, i.e. Snoek atmospheres [81]. Three types of solute atom atmosphere can lock dislocations, i.e. Cottrell, Suzuki, and Snoek locking mechanisms, respectively. Special attention however must be given to the Suzuki locking mechanism since AISI 316L is a FCC material with low stacking fault energy. Since dislocations dissociate into partial dislocations due to low stacking fault energy, solute atoms can therefore segregate to faulted areas to form Suzuki atmospheres. Suzuki atmospheres have two effects:

- Firstly, they further lower the stacking fault energy of material, resulting in a more planar character of the dislocation structure, thereby promoting the localisation of plastic deformation. The tendency for a planar configuration of dislocations during cyclic loading of AISI 316L at 300°C is extended to higher cycle numbers as the strain amplitude decreases.

- Secondly, they participate in locking dislocations.

Because flow stress serration is a material response under the combination of both locking mechanisms due to point defects and the localisation of plastic deformation, the Suzuki locking mechanism plays an important role on the flow stress serration.
Figure IV.22: (a) Solute atoms diffuse to mobile dislocations to form disordered clouds, i.e. a Suzuki atmosphere, at the end of fatigue life. (b) Short-range-ordered Snoek atmosphere forms around dislocations after 30,000 cycles for a testing condition of ±0.25%.

Since C and N are light elements, TEM imaging cannot reveal the clear configuration of solute atom atmospheres of C and N around dislocation lines. The structure of solute atom atmospheres of C and N however can be realised from the analysis of electron diffraction patterns [47]. In this study, selected area diffraction patterns (SADPs) of regions where dislocations were pinned by solute atom atmospheres were therefore recorded in order to check if there is any evidence of disordered or short-range-ordered structures of solute atom atmospheres. Figure IV.22a clearly shows that there was no diffuse scattering in the diffraction pattern (the inset image of Figure IV.22a). Solute atoms in a faulted area (indicated by the arrow) were therefore disordered (i.e. a Suzuki atmosphere forms in this faulted area). Figure IV.22b shows a region where dislocations were pinned by solute atom atmospheres after 30,000 cycles. In contrast to the region in Figure IV.22a, clear evidence of diffuse scattering in the diffraction pattern was observed in this region (the inset image of Figure IV.22b), which indicates the presence of short range ordering [47]. This indicates that dislocations in this region of Figure IV.22b were pinned by Snoek atmospheres. Once solute atom atmospheres form, they can lock mobile dislocations as shown in Figure IV.21d.
The evidence referred to above implies that the locking due to solute atoms is at first of the Suzuki type. Upon further loading, Snoek atmospheres form to strengthen the locking effect of solute atoms on the movement of dislocations. It is interesting to note that the Suzuki atmosphere is observed in every response stage of cyclic loading. However, the Snoek atmosphere is only observed during the secondary cyclic hardening stage (Figure IV.22b). The Snoek atmosphere therefore partly contributes to the secondary cyclic hardening response of the material.

IV.4.6. Planar slip character and stress serration activity

Dislocations are more prone to planar character once materials plastically deform in the regime of DSA [82, 131]. These authors believed that the formation of solute atom atmospheres is attributable to the planar character of the dislocation structure. However, there has been no clear evidence to indicate why, and what kind of solute atom atmospheres is responsible for the planar character. This present study clearly shows that the more planar character of dislocation structures is partially due to an effect of the lower stacking fault energy of Suzuki atmospheres as discussed in Sect. IV.4.5. Although dislocation interactions increase dislocation junctions which enhance cross slip upon further loading, the formation of short-range-ordered Snoek atmospheres after a number of cycles can further suppress the wavy movement of dislocations due to the short-range-ordered structures which promote the planar dislocation character at 300°C [63]. The enhanced planar movement of dislocations due to the strong interaction between solute atoms and dislocations at 300°C explains why the $b_{h1}$ (Figure III.11) for conditions at 300°C is almost half of that for equivalent conditions at 20°C.

The DSA of AISI 316L at 300°C is a repetition of locking and unlocking both edge and screw dislocations most likely due to Suzuki atmospheres which cause intense serration at the beginning of cyclic loading. The locking causes a rapid increase in flow stress. Once stress reaches a critical value, a mass of dislocations are suddenly unpinned from the locking atmosphere, resulting in stress drop. During loading,
however, the mobility of edge dislocations drops quickly due to the formation of dislocation junctions, e.g. Lomer-Cottrell junctions, once secondary slip systems activate. During loading, if they are further locked by solute atom atmospheres, most edge dislocations are immobile. Edge dislocations therefore mainly contribute to the work hardening rate, but do not contribute to flow stress instability. In contrast, if screw dislocations are locked by Suzuki atmospheres, they can escape from solute atom atmospheres under sufficient stress because the formation of dislocation junctions lowers the activation energy for cross slip [74, 75]. Locking and unlocking screw dislocations, which are more mobile with the assistance of dislocation junctions, are mainly responsible for the stress serration upon further loading. The stress serration should therefore attenuate during cyclic loading (Figure III.16 and Figure III.17), in particular during the cyclic softening response stage over which dislocation cross-slip is most active, i.e. screw dislocations can easily cross slip to get away from solute atom atmospheres. The formation of Snoek atmospheres after a number of cycles however strengthens the locking effect of solute atoms, resulting in the re-occurrence of the stress serration. Since Snoek atmospheres are formed under the assistance of stress, Snoek atmospheres are more readily formed with increasing imposed stress. This explains why: (1) the activity of stress serration is not totally phased out during the cyclic softening stage for the ±0.7%-condition (Figure III.16), as happens for the ±0.4%-condition (Figure III.17), and (2) there is no clearly observed re-occurrence of stress serration for the ±0.25%-condition. The competition between the formation of Snoek atmospheres and the strong activity of cross slip due to a high number of dislocation junctions for the ±0.7%-condition is likely to make the re-occurrence of the stress serration take place later than that for the ±0.4%-condition. It consequently provides a full explanation for the contradiction between our observations with existing DSA theory which predicts that DSA should be more active when dislocation density and vacancy concentrations increase.
IV.4.7. Corduroy contrast, corduroy structure and cyclic deformation response

As shown in Figure IV.19a new slip bands are activated during cyclic loading, destroying former ones, leading to formation of a new corduroy structure along the orientation of the latter slip activity (in agreement with the study of Gerland et al). An observation in Figure IV.20 indicates that corduroy contrast results from lines of small point defect clusters of the remnant point defects of decayed corduroy structure. Lines of small point defect clusters can be also of newly formed corduroy structure. It is noted here that very small point defect clusters can be revealed in TEM as black spots in BF images of multi-beam conditions, or black-white contrast in two-beam conditions (i.e. in the dynamic condition) [132]. Lines of very small point defect clusters therefore induce alternating black-white contrast fringes under two-beam conditions. Small clusters of point defects of the decayed corduroy structure consequently result in corduroy contrast under dynamic image conditions as shown in Figure IV.20a-c, and in lines of point defects when \(g\) changes. This fully explains why corduroy contrast is: 1) only clearly observed under two-beam conditions, 2) normally discontinuous, 3) often observed in the early stage of the formation of newer corduroy structure, and 4) almost non-existent once the well-organised corduroy structure forms.

Once corduroy structure forms, it can act as obstacles to dislocation movement (Figure IV.19f), which in turn contributes to the hardening effect of the material. Gerland et al. originally suggested that corduroy structure is responsible for the secondary cyclic hardening response of AISI 316L in vacuum [82]. The hypothesis is therefore quite widely accepted to explain the secondary cyclic hardening of austenitic stainless steel in air or other testing conditions without showing clear evidence of corduroy structure. Cho et al. recently provided evidence which is believed to be corduroy contrast of AISI 316LN after cyclic loading in 310°C deaerated water [133]. The evidence was however most likely the contrast of stacking faults. In addition, as discussed in Sect. IV.4.5, Snoek atmospheres form after a number of cycles, and can also lock mobile dislocations, resulting in the material hardening. Consequently, not only the corduroy structure but also solute atom atmospheres are responsible for the secondary cyclic
hardening response of the material. The re-active phase of the flow stress serration after several hundred cycles for the ±0.4%-condition (Figure III.17) (or after several thousand cycles for the ±0.7%-condition (Figure III.16)) indicates that Snoek atmospheres are already present during the re-active phase (see also Sect. IV.4.6). Secondary cyclic hardening is however not observed for the ±0.7%-condition (where the corduroy structure just weakly forms at the end of fatigue life), and occurs when the corduroy structure is more well-formed for the ±0.4%-condition (after about 5000 cycles). The contribution of corduroy structure to the secondary cyclic hardening response is therefore very likely to be greater than that for Snoek atmospheres.

To the author’s knowledge, it is the first time that a comprehensive examination of corduroy structure of cyclically strained AISI 316L in air without irradiation damage has been conducted in order to fully understand its appearance, and its relation to the material cyclic deformation response.

### IV.4.8. Hydrostatic-stress dependence

The mobility of point defects (vacancies or interstitial atoms), \( D_{v,i}^* \), is proportional to

\[
\exp\left(-\frac{\Delta G_{v,i}^*}{kT}\right)
\]

where \( \Delta G_{v,i}^* \) is Gibbs energy of activation of diffusion. The subscripts of \( v \) and \( i \) stand for vacancy and interstitial atoms, respectively. According to thermodynamic theory,

\[
\Delta G_{v,i}^* = \Delta E_{v,i}^* - T\Delta S_{v,i}^* - \sigma^{ext} \Delta V_{v,i}^*
\]  

(IV.1)

where \( \sigma^{ext} \), \( \Delta E_{v,i}^* \) and \( \Delta V_{v,i}^* \) are the external stress, activation energy, and activation volume, respectively. If only the influence of hydrostatic stress is considered, i.e. \( \sigma_{11} = \sigma_{22} = \sigma_{33} = -p \) where \( p \) is the pressure, one can get

\[
\Delta G_{v,i}^* = \Delta E_{v,i}^* - T\Delta S_{v,i}^* + p\Delta V_{v,i}^*
\]
Therefore, $D^*_i = D^0 |_{p=0} \times \exp(-\frac{p\Delta V^*_i}{kT})$ \hspace{1cm} (IV.2)

where $D^0 |_{p=0}$ is the mobility of point defects under condition of no hydrostatic pressure.

Eqn. (IV.2) indicates that the pressure dependence of point defect mobility depends on the state of pressure and activation volume when point defects are introduced. The demonstration of crystal volume change due to the formation of a vacancy or an interstitial atom at an octahedral position is given in Figure IV.23a-c. For the sake of simplicity, the introduction of point defects into a simple cubic material at positions which are equivalent to the case of an FCC material is depicted. Indeed, the introduction of both vacancy or interstitial atoms into FCC materials induces an expansion of volume (Figure IV.23), it means that the mobility of vacancy and solute atoms both increase during tensile going stress state (i.e. $p<0$) and decrease during compressive going stress state ($p>0$), since $\Delta V^*_{v,i} > 0$.

However, if one roughly calculates volume expansions caused by the introduction of a vacancy and an interstitial atom into an FCC material, it is apparent that the extent of direct influence of pressure on the vacancy or the solute atom is very different. For instance, If an atom is considered as a hard sphere, atomic volume is: $\Omega = \frac{\pi a^3}{12\sqrt{2}}$

where $a$, the lattice parameter of AISI 316L, is about 3.60 Å. The radius of an octahedral hole in AISI 316L (Figure IV.23d) is

$r = (a - a\sqrt{2}/2) / 2 = 0.527\text{Å}$ \hspace{1cm} (IV.3)
The nominal radii of C, and N are respectively 0.77 and 0.72 Å. The insertion of an atom of C or N induces a volume expansion ($\Delta V^c$) of about 0.15$\Omega$ or 0.11$\Omega$ which is fairly small compared to the $\Delta V^v$ of a vacancy which is about 0.5$\Omega$ for metallic materials [1]. This means that pressure is unlikely to have a significant effect on the mobility of C and N, but it would strongly influence the mobility of vacancies. It should be remembered that vacancy mobility is promoted during reverse loading transients from compressive peak stress, but suppressed during reverse loading transients from tensile peak stress. In other words, the mobility of vacancies after load reversal from tension peaks is higher than that after load reversal from compression peaks. As mentioned-above, solute atom atmospheres form in the presence of vacancies. This
means that DSA should take place earlier during reverse loading transients from tensile peak stress in the cycle than during reverse loading transients from compressive peak stress. In other words, the serration length is longer for reverse loading transients from tensile peak stress than for during reverse loading transients from compressive peak stress (Figure III.18a, c).
IV.5. Concluding remarks on the microstructural evolution and cyclic deformation response

In the as-received condition, dislocations are mainly present in the form of planar structures. Dislocation density increases during cyclic hardening, and results in dislocation-dense sheets with some interconnections between them at the end of the cyclic hardening stage. Dislocations rearrange during the softening stage due to the activation of secondary slip (which includes cross slip), resulting in high-density/low-density regions of dislocations. Upon further loading, high-density/low-density regions becomes more well-organised structures, e.g. walls/channels. Depending on imposed strain amplitude, different types of dislocation structure are formed towards the end of fatigue life. At the ±0.7% -10^{-3}s^{-1}-20°C test, the strong multiple slip facilitates wall/channel structure to develop into a cellular structure. The activity of secondary slip systems creates more connections between walls, resulting in a labyrinth structure. Towards the end of fatigue life, cellular structure consequently becomes more equiaxed. Cellular structures are believed to more effectively confine the movement of mobile dislocations than wall/channel structures, resulting in a final saturation response stage on the material at this testing condition.

Since imposed plastic deformation is lower for smaller strain amplitude conditions (e.g. ±0.4 and ±0.25%), dislocations develop in slightly different manner to that of the ±0.7%-condition:

- Dislocations are less dense, resulting in smaller rates of change of the first cyclic hardening response.
- At the end of fatigue life, dislocation structures are less well-organised. The strength of dislocation walls and cell-to-cell misorientation are therefore lower. The density of dislocations inside channels is however higher for lower strain amplitudes. This indicates that dislocation structures would develop further if the material could endure higher numbers of cycles. It also means that the strength of dislocation structures (i.e. the total dislocation density) is mainly responsible for lower peak stress at the saturation stage for lower strain amplitude conditions.
- Dislocation configuration exhibits a strong tendency to planar character for lower strain amplitude conditions. The tendency extends to a higher number of cycles when the strain amplitude decreases. Towards the end of fatigue life, a relative portion
of material is occupied by ill-organised dislocation walls (for the ±0.4% test condition) or dislocation carpets (for the ±0.25% test condition). The tendency to generate organised dislocation wall/channel or cellular structure is increasingly diminished.

During cyclic loading at 300°C, 316L stainless steel exhibits: 1) more significant primary cyclic hardening than that at room temperature, 2) cyclic softening, and 3) an almost stabilised response stage or a secondary cyclic hardening if premature fracture does not intervene. The more significant primary and secondary cyclic hardening responses of the material are attributed to dynamic strain ageing which is due to the strong interaction between solute atoms and dislocations. At the beginning of cyclic loading, Suzuki atmospheres of solute atoms forming around dislocations, along with Lomer-Cottrell junctions, can effectively lock dislocations, in particular edge dislocations, resulting in the pronounced primary cyclic hardening. During the secondary cyclic hardening phase, additional solute atom atmospheres, i.e. Snoek short-range-ordered atmospheres, form to strengthen locking effects. In addition, corduroy structure is also observed during this stage. The two conditions are responsible for the secondary cyclic hardening response.

Suzuki atmospheres of solute atoms lower the stacking fault energy of the material, resulting in the more planar character of dislocation structures. Snoek atmospheres are also responsible for the planar characteristic of dislocation structures upon further loading due to their short-range-ordered characteristics. In addition, solute atom atmospheres effectively lock dislocations, resulting in higher immobile dislocation density during cyclic loading at 300°C, as well as higher density of dislocation walls towards the end of fatigue life, e.g. the dislocation wall/channel is more organised for the ±0.7%-condition at 300°C compared to the equivalent condition at room temperature.

DSA strikingly manifests in flow stress serrations which are very active at first, and then gradually disappear. There is then almost no stress serration during the cyclic softening stress response regime in particular in low strain amplitude tests. Finally, DSA reoccurs after a further number of cycles. The first active phase of serration is due to the interaction of Suzuki clouds with both edge and screw dislocations. Upon further loading, edge dislocations are almost immobile because of both Lomer-Cottrell and
Suzuki locking. In contrast, the formation of dislocation junctions can facilitate extended screw dislocations to cross slip since the activation energy of partial dislocation recombination is reduced. Consequently, stress serration decreases gradually during the first cyclic response phase, then almost disappears during the softening phase over which cross-slip is most active. Snoek atmospheres of solute atoms however form after a number of cycles, and these are responsible for the re-occurrence of stress serration. Stress serration is also found to be different during reverse loading transients from compressive and tensile peak stresses, and this is attributed to the effect of hydrostatic pressure on the mobility of point defects, especially vacancies. 

An effort has been made in the present study to fully understand the presence of corduroy contrast and its links with corduroy structure. It is clearly shown that corduroy contrast is indeed the image under dynamic two-beam conditions of corduroy structure consisting of lines of very small point defect clusters. This study provides a comprehensive explanation for corduroy contrast characteristics, which are: 1) only clearly observed under exact two-beam conditions which fade out once $g$ changes, 2) discontinuous, 3) often observed in the early stages of formation of newer corduroy structure, and 4) almost non-existent once the well-organised corduroy structure forms.
Chapter V: The relationship between interactions of dislocations and internal stresses

Abstract:

The evolution of dislocation densities and of dislocation microstructures during cyclic loading of AISI 316L is quantitatively characterised in this Chapter. In addition, corresponding internal stresses are also quantified for every cycle, and comprehensively analysed. These systematic characterisations are served to establish the relationships between the evolution of dislocation condition and internal stresses, and ultimately to obtain thorough insights into the complex cyclic response of AISI 316L. In the beginning of cyclic loading, a great number of dislocations pile up close to grain boundaries as a consequence of grain-to-grain strain incompatibility, leading to a significant increase in long-range inter-granular back stress. The activity of secondary slip (including cross slip) during cyclic loading on the one hand relieves the inter-granular back stress, but on the other hand raises the intra-granular back stress due to the formation of dislocation-high/low-density regions. Moreover, the dependence of the established relationships on the variation of boundary loading conditions is revealed. The back stresses are mainly responsible for the cyclic deformation response at high strain amplitudes where dislocations tend to move more quickly in a wavy manner. In contrast, the effective stress, coupling with short-range dislocation interactions, plays an insignificant role on the material cyclic response for wavy slip conditions, but increasingly becomes more important for planar slip conditions. The additionally strong short-range dislocation interaction at 300°C (initially with solute atoms, and later in life with corduroy structure) however induces a significant increase in effective stress, leading to its influential role on the material cyclic response.
V.1. Introduction

Polycrystalline materials, for which dislocations incline to move in a more planar manner during fatigue loading, usually manifest a complex cyclic deformation response due to the changing configuration character of dislocations. For example, significant changes in the density and configuration of dislocations during fatigue loading make AISI 316L exhibit a complex cyclic deformation response at 20 and 300°C, i.e. cyclic hardening followed by cyclic softening and finally an almost stabilised cyclic response or a secondary cyclic hardening response stage before failure (Chapters III, IV) [90, 96, 134]. In addition, the alteration of loading conditions also leads to the variation of dislocation condition, resulting in different material response. These cause difficulties to understand material cyclic deformation response, in particular its history/amplitude dependence. Since the deformation behaviour of polycrystalline materials inherently associates with microstructural condition [36, 40, 42, 43, 83, 90, 135], these difficulties can be solved if one can obtain thorough understanding of: (1) the comprehensive relationships between the evolution of dislocation condition and internal stress variables which characterise the deformation response of materials, and (2) how these relationships change with boundary conditions.

The introduction of internal stress variables (e.g. isotropic (or effective) stress, and back stresses) provides great capabilities to effectively characterise the elasto-plastic behaviour of metallic materials [84, 136-141]. Moreover, the physical interpretations of these internal stress variables are also intensively discussed over past decades [40, 83, 91, 142, 143]:

1) Effective stress ($\sigma_{\text{eff}}$) consists of the initial effective stress ($\sigma_{\text{eff}}^0$) and an additional increment of yield stress ($\sigma_{\text{y}}^*$) during loading. The initial effective stress is required to make dislocations overcome the local resistance to dislocation movement at the initial condition of the material (which includes the intrinsic resistance, e.g. periodic lattice resistance, and the extrinsic resistance, e.g. the phonon drag and the initial restriction of movement induced by the
presence of other dislocations). $\sigma^e_\epsilon$ is the result of a change in the motion resistance due to the variation of short-range-ordering interactions of dislocations with themselves (e.g. Lomer-Cottrell sessile dislocation junctions [50, 51]) and with other defects (e.g. solute atoms), resulting in a change in the flow stress upon further loading [40, 83]. Within the principal stress co-ordinates, yield stress is therefore the radius of the yield surface. The change in effective stress is responsible for the expansion or contraction of the yield surface [84].

2) Back stress ($X$) associates with long range interactions of collective dislocations which arise due to inhomogeneous plastic deformation between microstructural heterogeneities of materials on different scales, e.g. grains, subgrains, cell blocks, and families of dislocation walls/channels [40, 85-91]. Microstructural heterogeneities are either present before loading or formed during loading, and possess different strength with respect to loading direction, causing the inhomogeneous deformation between them under loading.

The material of interest in this study is the AISI 316L steel which is widely used in engineering industry, for example in the pipe cooling circuits of light water nuclear power plants for which the working temperature is about 300°C. Understanding the fatigue behaviour of the material under such conditions is required to verify the safety factors applied to the material in service duty. As referred to above, the material exhibits complex cyclic deformation response at 20 and 300°C. To comprehend the material deformation response, the evolution of microstructural features responsible for internal stresses of AISI 316L during cyclic loading at 20 and 300°C is exhaustively studied in this chapter. Systematic observations of samples fatigued to selected numbers of cycles in the different stages of cyclic mechanical response are made in order to quantify dislocation densities and the dimensions of dislocation microstructures. In addition, corresponding internal stresses are measured from the stress-strain hysteresis loop of every cycle. The evolution of internal stresses is analysed, and then related to dislocation condition. Moreover, the history and amplitude dependence of the roles of internal stresses on the material mechanical
response is revealed. The detailed relationships between microstructural condition and the material deformation response are thereby discussed and established. The established relationship will provide the basis to develop a physically-based constitutive model which is discussed in Chapter VI.
V.2. Relationships between effective/back stresses and the microstructural condition during cyclic loading at 20°C

V.2.1. Cyclic hardening response stage

In the case of well-annealed FCC materials during early cyclically plastic deformation, dislocations pile up close to grain boundaries to accommodate the different degree of plastic deformation between grains (Figure IV.4b and Figure V.1a, b). This evidence means that during the early stage of cyclic loading, dislocations close to grain boundaries (hereinafter called boundary dislocations, $\rho_b$) are mostly of the Geometrically Necessary Dislocation (GND) type [86]. The inter-granular back stress $X_{\text{inter}}$ associated with inhomogeneous plastic deformation between grains of materials therefore increases significantly in the beginning with the increase in $\rho_b$. According to the Taylor relationship [36] and the recent understanding of the relationship between internal stress with its corresponding dislocation type [144-146], a given internal stress can relate to the square root of the density of the corresponding dislocation type ($\sqrt{\rho_b}$).

During plastic deformation, the dislocation density inside grains (hereafter named interior dislocations, $\rho_I$) also increases, but at a slower rate, leading to a lower gradient of dislocation density between regions close to grain boundaries and regions inside grains, i.e. lessening the grain-to-grain strain incompatibility. Moreover, the secondary slip activity relieves the grain-to-grain plastic strain incompatibility, and also results in the abundance of interior dislocations and a more homogeneous distribution of dislocations over grains (Figure V.1c). $X_{\text{inter}}$ therefore decreases with the increase in $\sqrt{\rho_I}$. Consequently, $X_{\text{inter}}$ arising due to cyclic loading (Figure III.2) is proportional to the difference in the development of boundary dislocations and interior dislocations, i.e.

$$2X_{\text{inter}} \propto (\sqrt{\rho_b} - \sqrt{\rho_I})$$  \hspace{1cm} (V.1)
Figure V.1: (a) Grain-to-grain plastic strain incompatibilities, (b) dislocation pile ups close to a grain boundary, (c) Strong activity of secondary slip and a homogeneous distribution of dislocations over a grain, (d) dislocation walls and channels, and (e) the fragmentation of grains into different groups of cellular structure in order to accommodate the plastic strain incompatibilities.

The increase in dislocation density also results in more frequent interactions. In particular, when the secondary slip is strongly active, the Lomer-Cottrell interaction of dislocations (Sect. II.3.4) (which is the main kind of the short range interaction of dislocations themselves in FCC materials [50, 51, 83, 130]) results in the formation of sessile dislocation junctions (Figure V.2), leading to an increase in effective stress. Since Lomer-Cottrell sessile junctions form due to the activity of secondary slip which relates to the increase in interior dislocations, the increment of effective stress during cyclic loading ($2\sigma_{R}^{K}$) due to the interactions between dislocations is mainly proportional to $\sqrt{\rho_{I}}$, i.e.

$$2\sigma_{R} \propto \sqrt{\rho_{I}} \quad \text{(V.2)}$$
V.2.2. Cyclic softening and stabilised response stages

Once interior dislocations become abundant and occupy a much larger portion than boundary dislocations, total dislocations which are mainly constituted by interior dislocations start rearranging to form dislocation high/low density regions, and seek to form their most stable configuration under a given condition. In addition, the slip activity and the rearrangement of dislocations also participate in the process of grain fragmentation to a greater scale (Figure V.1e). The grain fragmentation manifests in the form of: 1) distinct cell blocks containing a number of ordinary well-organised dislocation cells for high degree plastic deformation [88], 2) the presence of families of dislocation cellular structure [90], or 3) regions without distinct boundaries although they can be revealed by changes in misorientation-induced contrast over a large number of dislocation walls [89]. Grain fragmentation and the formation of dislocation high/low density regions have two effects:
They contribute to the relief of the inter-granular back stress $X_{\text{inter}}$ until the requirement of material continuity between grains is completely fulfilled by the response of collective fragmented volumes (Figure V.1e). This indicates that $X_{\text{inter}}$ achieves its stable value after a number of cycles once dislocation structures are well formed, i.e. when the stabilised condition of material response is established. At this condition, $X_{\text{inter}}$ associates with dislocations of GND type (named stabilised inter-granular GNDs ($\rho_{\text{GNDs-inter}}$)). These GNDs are the minimum required number of dislocations to accommodate the grain-to-grain plastic strain incompatibility at the stabilised condition, and are present mainly in regions close to boundaries of fragmented volumes since the grain-to-grain plastic strain incompatibility is accommodated by the collection of fragmented volumes. Moreover, since there is no clear distinct boundary between fragmented volumes, it is not possible to correctly measure $\rho_{\text{GNDs-inter}}$ by TEM. However, the stabilised inter-GNDs can be derived from the flow stress of a qualified cycle at the stabilised condition if $X_{\text{intra}}, \sigma_E$ at the stabilised condition is known, which is discussed in detail later (Sect. VI.5.1.1).

On a finer scale, i.e. intra-granular scale, the re-arrangement of dislocations creates other strain incompatibilities between dislocation high/low density regions [147], e.g. dislocation walls/channels (Figure V.1d). Consequently, the intra-granular back stress $X_{\text{intra}}$ increases with the development of these fine dislocation structures until the most stable structures form, i.e. the dislocation densities within dislocation high/low density regions reach their saturation values [148]. As a result, $X_{\text{intra}}$ evolves with another type of GNDs ($\rho_{\text{GNDs-intra}}$) which are present in the interface between high density region and low density region dislocation structures. It is worth to note that the back stress associated with this type of GNDs is proportional to the difference between the square root density of dislocations within walls (named wall dislocations, $\rho_w$) and within channels (called channel dislocations, $\rho_c$), and the fraction of walls ($f_w$) [144,
Therefore, the intra-granular back stress arising due to the formation of dislocation wall/channel structures during cyclic loading (2X_{\text{intra}}) can be described as follows.

\[ 2X_{\text{intra}} \propto \left( \sqrt{\rho_w} - \sqrt{\rho_c} \right) \times f_w \quad (V.3) \]

Once the stabilised condition of material response is established, the requirement of material continuity is totally fulfilled both at the inter-granular scale and the intra-granular scale. A minimum quantity of dislocations is required to move back and forth in dislocation-low regions to accommodate the imposed plastic strain during cyclic loading.
V.3. Relationships between effective/back stresses and the microstructural condition during cyclic loading at 300°C

The relationships between the evolution of internal stresses and microstructural condition established for 20°C are in general also applicable for 300°C. However, the strong short-range interaction between solute atoms and dislocations at 300°C promotes the formation of solute atom atmospheres around dislocations. The solute atom atmospheres induce higher densities of both boundary and interior dislocation types, which influence all internal stresses, in particular for effective stress. Moreover, later in the fatigue life, the formation of a corduroy structure (Figure V.3) induces a significant increase in $\sigma_e$ (Table III.5). The strong interaction between point defects and dislocations induces significant increases in all internal stresses. This explains why metallic materials typically exhibit a strong tendency to being harder in the DSA regime. However, thermal activation (when the loading temperature increases from 20 to 300°C) lowers internal stresses. Back stresses much less depend on the thermal activation (their temperature dependence only relates to the temperature dependence of elastic modulus). A more significant increase in dislocation densities due to the strong interaction between solute atoms and dislocations in the DSA regime can therefore compensate for the softening due to the thermal activation, resulting in almost identical values for back stresses at 20 and 300°C (Figure III.13). In contrast, DSA cannot compensate for the decrease of the effective stress due to the thermal activation. This causes the effective stress for AISI 316L to be still lower at 300°C (Figure III.14). Consequently, the maximum stress for 300°C is lower than that for equivalent conditions at 20°C for this steel (Figure III.9).
Figure V.3: (a) Profuse vacancies produced at 300°C facilitate the formations of (b) solute atom atmospheres and (c, and d) corduroy structure, both of which are responsible for the significant increase in the effective stress.

The jerky manner of dislocation motion due to the locking effect of solute atom atmospheres makes the relief of strain incompatibilities more difficult at 300°C. In addition, since solute atom atmospheres effectively lock dislocations, a higher number of dislocations are required to be generated during cyclic loading at 300°C (Figure V.4a). Moreover, the cell dimension depends on the total dislocation density [151, 152]. This explains why the diameter of dislocation cells for the ±0.7%-300°C condition is smaller than that for the ±0.7%-20°C as shown in (Figure V.4b). Although thermal activation lowers the absolute value of back stresses due to the decrease in the elastic modulus, the quicker development of dislocation microstructures into well organised configurations results in a more rapid increase in the intra-granular back stress at
300°C. These consequently result in the higher rates of change of back stress at 300°C (Table III.5).

Although the rates of change of effective and back stresses are both higher for 300°C (Table III.5, Figure III.13 and Figure III.14), the difference in the rates of change of effective stress between 20 and 300°C is however higher than that of back stress. This indicates that the difference between cyclic deformation response of the material at 20°C and 300°C is mainly due to the change in effective stress. In particular, during the secondary cyclic hardening at 300°C, the rate of change of effective stress is interestingly much larger than that of back stress (Table III.5).
V.4. Strain amplitude dependence

Table V.1: Relative ratios of the rate of change of effective stress and of back stress to that of maximum stress.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>$\frac{d\sigma^p_E}{d(\log N)}$</th>
<th>$\frac{dX^p}{d(\log N)}$</th>
<th>$\frac{d\sigma_{sof}^E}{d(\log N)}$</th>
<th>$\frac{dX_{sof}}{d(\log N)}$</th>
<th>$\frac{d\sigma^{se}_E}{d(\log N)}$</th>
<th>$\frac{dX^{se}}{d(\log N)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>±0.7% 20°C</td>
<td>0.06</td>
<td>0.91</td>
<td>0.07</td>
<td>0.95</td>
<td>0.07</td>
<td>0.95</td>
</tr>
<tr>
<td>±0.7% 300°C</td>
<td>0.13</td>
<td>0.83</td>
<td>0.07</td>
<td>0.95</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>±0.4% 20°C</td>
<td>0.16</td>
<td>0.81</td>
<td>0.32</td>
<td>0.70</td>
<td></td>
<td></td>
</tr>
<tr>
<td>±0.4% 300°C</td>
<td>0.21</td>
<td>0.73</td>
<td>0.39</td>
<td>0.72</td>
<td>1.70</td>
<td>1.05</td>
</tr>
<tr>
<td>±0.25% 20°C</td>
<td>0.28</td>
<td>0.85</td>
<td>0.51</td>
<td>0.53</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>±0.25% 300°C</td>
<td>0.24</td>
<td>0.73</td>
<td>0.51</td>
<td>0.60</td>
<td>0.80</td>
<td>0.25</td>
</tr>
</tbody>
</table>

The relative ratio of effective stress (or back stress) to that of maximum stress is made in order to obtain a more comprehensive understanding of the relative contribution of internal stress to the cyclic deformation response when strain amplitude changes. **Table V.1** shows the relative ratios of the rate of change of effective stress (and of back stress) to that of maximum stress. These ratios represent the contribution of effective stress and back stress to the material cyclic response. Lowering imposed plastic strain amplitude, the relative ratio of effective stress clearly increases while the relative ratio of back stress decreases. This indicates that the contribution of effective stress to the cyclic hardening/softening response increasingly becomes greater for
lower strain amplitudes (i.e. for more planar character of dislocation structures). In contrast, back stress becomes less important to the cyclic deformation response when strain amplitude decreases.

Figure V.5: The evolution of the interior dislocation density for ±0.7%-10^{-3}s^{-1} (solid lines) and for ±0.4%-10^{-3}s^{-1} (dashed lines) at (a) 20°C and (b) 300°C.

It is important to remember that for the lower strain amplitude condition: (1) less dislocations are generated (Figure V.5), (2) the planar structure of dislocation persists to a higher life fraction, and (3) less organised dislocation structures form towards the end of life. Because smaller imposed plastic strains induce lower grain-to-grain plastic strain incompatibilities, the inter-granular back stress is less for lower strain amplitudes. The planar character of dislocation structure is more predominant for lower strain amplitudes. In particular for the ±0.25% condition, stacking faults and dislocation carpets are present towards the end of fatigue life (Figure IV.14). The degree of well organised configuration of dislocations is therefore quantitatively characterised by the wall-to-wall spacing and wall thickness (only for the ±0.4 and ±0.7% conditions), which are shown in Figure V.6. This clearly shows that for lower strain amplitudes: (i) dislocation walls are further apart (i.e. dislocation wall spacing is higher), and (ii) dislocation wall thickness wider, indicating less dense dislocation walls. Since intra-granular back stress associates with the strength of dislocations (i.e. the density of wall
dislocation) and the distance between walls, the back stress becomes smaller with reducing strain amplitude.

The decrease in intra-granular back stress with lowering of strain amplitude is however not only due to the degree of organisation of dislocation structures towards the end of fatigue life, but also depends on the characteristics of dislocation configuration. Small imposed plastic strain results in a very slight change in the dislocation number during cyclic loading, i.e. dislocations are very mobile for well-annealed FCC materials. Dislocations therefore tend to form the lowest energetic configuration structure, a structure close to the equilibrium state [114, 153], which associates with negligible long-range stress fields. In contrast, for high imposed strain conditions, dislocations are dense and move in an intermittent manner (e.g. avalanche motion of dislocations such as PSBs which is the case for the ±0.4 and ±0.7% conditions), to form a self-organised dislocation structure (SODS). The SODS is a non-equilibrium thermodynamic configuration, and associates with long-range stress fields [154, 155]. In other words, intra-granular back stresses arising during plastic deformation for which dislocation movement is prone to be planar in character are much smaller than those of high imposed strain for which dislocation motion tends to be wavy. This explains why:

Figure V.6: Dislocation wall dimension (a) at 20°C and (b) 300°C: spacing and thickness for the ±0.4 (lines with symbols) and ±0.7% (lines without symbols)
1) At higher strain amplitudes for which well-organised dislocation walls/channels form, e.g. at the ±0.7%-condition, the influence of back stress on the cyclic deformation response far outweighs that of effective stress.

2) In contrast, at low strain amplitudes (e.g. ±0.25%) for which the lowest energetic configuration dislocation structures form, the material cyclic response is almost equally due to changes in back stress and effective stress.

At 300°C although dislocations move in a more planar way, dislocation density is higher and the movement of dislocations is very intermittent due to solute-atom/dislocation interactions, the increase in back stress being higher than for 20°C.
V.5. Concluding remarks on the internal stress-microstructural condition relationship

Effective stress is responsible for the isotropic expansion (or contraction) of yield surface. This internal stress associates with the short-range-ordering interactions of dislocations with themselves (e.g. Lomer-Cottrell sessile dislocation junctions) and with other small defects (e.g. solute atoms and nano-voids). At 20°C, the Lomer-Cottrell junctions are the main source for the increase in effective stress. The junctions are formed due to the activity of secondary slip. In addition, the activity of secondary slip results in the abundance of interior dislocations. Effective stress is therefore proportional to interior dislocations. However, at 300°C the strong short-range interaction between dislocations and solute atoms (C, and N) (and later with corduroy structure) significantly increases the effective stress. This is mainly responsible for the more marked primary cyclic hardening and the secondary cyclic hardening response.

In the beginning of cyclic loading, inter-granular back stress (associating with the plastic strain incompatibility between grains) relates to the number of dislocations in the regions close to grain boundaries. Upon further loading, the activity of secondary slip relieves the grain-to-grain plastic strain incompatibility, and also results in a more homogeneous distribution of dislocations over grains. Once interior dislocations become abundant and occupy a much larger area fraction than boundary dislocations, total dislocations start rearranging to form dislocation high/low density regions (and seek to form their most stable configuration). The dislocation rearrangement on the one hand contributes to the further relief of the grain-to-grain plastic strain incompatibility, but on the other hand raises strain incompatibilities between dislocation high/low density regions on a finer scale. If the stabilised condition of microstructural condition is established, the requirement of material continuity is totally fulfilled both at the inter-granular scale and the intra-granular scale. A minimum quantity of dislocations is required to move back and forth in dislocation-low regions to accommodate the imposed plastic strain during cyclic loading.
Back stresses are mainly responsible for cyclic stress response, in particular in the case of wavy slip mode conditions. Effective stress plays a much less significant role on the material cyclic response for wavy slip conditions, but increasingly becomes more important for planar slip conditions. It means that lowering imposed strain amplitude makes the effective stress play a more important role, while lessening the contribution of back stress.
Chapter VI: Physically-based evolutionary constitutive modelling

Abstract

This chapter will describe how the understanding obtained in the previous chapters provides a foundation for the development of a physically-based evolutionary constitutive model, which aims to accurately represent the complex cyclic deformation response of the material. The developed constitutive model reflects the change in microstructural condition and its relationship with internal stress variables during cyclic loading at both 20 and 300°C. The model parameters are identified by systematic evaluations of mechanical and microstructural observations from a number of interrupted fatigue tests for a single test condition. The capability of the model is then verified by predicting the material cyclic response for different testing conditions at 20 and 300°C. The verification evaluation shows that the proposed model (1) accurately describes the complex cyclic elasto-plastic deformation behaviour of AISI 316L for constant strain amplitude cyclic loading, and (2) well predicts the cyclic deformation response for variable strain amplitude fatigue loading.
VI.1. Introduction

In many modern practical applications, in order to improve the reliability of lifetime assessment procedures, it is desirable to have a constitutive model which accurately describes the history dependence of long-term mechanical response, e.g. cyclic deformation response. This demand can be met by developing evolutionary constitutive model equations. As a consequence of taking insufficient account of underlying physical mechanisms, phenomenological constitutive models can be incapable of accurately simulating material mechanical behaviour once the boundary conditions extend beyond the considered mechanism range. In particular, considerable changes in the cyclic response of many polycrystalline materials are observed during early loading cycles [60, 61, 90]. This can make phenomenological models unable to accurately describe the history dependence of cyclic deformation response (even when the boundary conditions do not change), since they often characterise the situation at the saturated response stage. In order to improve the descriptive capability, one can still use phenomenological models, but there is then the need to repeatedly identify parameters for numbers of cycles of different cyclic response stages. An alternative approach is to provide a satisfactory physical basis for the internal state variables so that the evolutionary constitutive model relates to any change in important physical quantities. The cyclic deformation response is expected to be accurately predicted if the knowledge of microstructural evolution during cyclic loading is gained. Over past decades, there have been great developments of state-of-the-art techniques which allow us to obtain a deep understanding of the microstructural evolution during loading, e.g. Chapters II-V. Consequently, this study adopts the latter approach, and aims to develop evolutionary constitutive model equations on the basis of the relationship between internal state variables and microstructural condition, which is obtained by a systematic investigation of both mechanical and microstructural evolution during cyclic loading (Chapters III-V).

In order to develop such an evolutionary constitutive model, it is necessary to have: (1) suitable internal state variables characterising the plastic deformation behaviour of the
material, and (2) a relationship between physical features and the internal state variables during cyclic loading. Internal state variable theory was first established in a thermodynamics framework by Coleman and Gurtin [156], and then further developed to solve problems in metal plasticity [157]. The introduction of isotropic and kinematic hardening rules [84, 136, 137] to model the elasto-plastic behaviour of metallic materials led to the adoption of internal stress variables (i.e. isotropic (or effective) stress, and back stresses) with some sense of physical mechanisms [138-141]. Evolutionary model equations with back stresses during cyclic loading were also developed [138, 158-162]. However, the evolution of back stresses in these implementations either: (1) did not directly relate to the development of microstructure, or (2) related only to a microstructural condition where dislocation walls and channels had already formed [163, 164]. It is therefore desirable to develop evolutionary constitutive models on the basis of the link between internal stress variables and microstructural condition. Much more effort has been made over the last decades to take account of underlying mechanisms into the development of such constitutive models. Because the movement of dislocations is the main physical basis for plastic deformation over a very wide range of loading conditions [40, 41, 53, 83], dislocation densities and their evolution have been greatly incorporated into constitutive models as microstructure-related internal state variables. Existing dislocation-based constitutive models have attempted to take the evolution of different types of dislocation densities (e.g. mobile, immobile, wall, cell interior dislocation densities) into account to continuously improve the capability of constitutive models for monotonic loading condition [36, 165-169]. In these implementations, there is no effort to relate the evolution of different dislocation densities to isotropic and kinematic internal stresses, which can make them incapable of simulating the path dependence of deformation response for reverse loading problems. Moreover, for cyclic loading of polycrystalline materials, deformation behaviour evolves differently to that for monotonic loading [170]. Although Estrin and his colleagues introduced a back stress term to extend the approach for cyclic loading conditions [171], this extension unfortunately applies only for dislocation wall/channel microstructures and is cumbersome to apply for multiple
cycle situations. Consequently, it is necessary to establish more effective and comprehensive links between internal stresses and the development of dislocation condition during cyclic loading from an as-received condition (where there is no presence of dislocation walls and channels) to a condition of well-organised dislocation structures. The mechanical response and corresponding microstructural evolution during cyclic loading of AISI 316L have been intensively examined in Chapters III and IV. The effective and comprehensive relationships between internal stresses and the development of dislocation condition during cyclic loading have been consequently established in Chapter V. This chapter will describe how the understanding obtained in the previous chapters provides a basis for the development of a physically-based evolutionary constitutive model, which aims to accurately represent the complex cyclic deformation response of the material.
VI.2. General formulation of dislocation density evolution

The evolution of dislocation density during plastic deformation is proportional to the generation rate of dislocations \( \frac{d\rho_i^\text{gen}}{d\varepsilon_p} \) minus the annihilation rate of dislocations \( \frac{d\rho_i^\text{ann}}{d\varepsilon_p} \). While the rate of the generation of dislocations is found to be inversely related to the dislocation mean free path, that of the annihilation of dislocations linearly depends on dislocation density \([148, 172, 173]\), i.e.

\[
\frac{d\rho_i}{d\varepsilon_p} = \frac{d\rho_i^\text{gen}}{d\varepsilon_p} - \frac{d\rho_i^\text{ann}}{d\varepsilon_p} = k_1 \frac{1}{L_i} - k_2 \rho_i \tag{VI.1}
\]

Where: \( k_1 \) and \( k_2 \) are coefficients characterising the proportionalities of the generation rate to the dislocation mean free path, and of the annihilation rate to the dislocation density, respectively.

\( L_i \) is the mean free path of type \( i \) dislocations which depends on grain size, inter-distance of particles, and the density of type \( i \) dislocations. If there are no precipitates present, \( \frac{1}{L} = \sqrt{\rho} + \frac{1}{D_g} \). In the case of relatively coarse grain size (e.g. \( D_g \approx 60 \times 10^{-6} \text{m} \)) and the dislocation density is relatively high \( \rho \geq 10^{13} \text{1/m}^2 \):

\[
\frac{1}{D_g} \approx \frac{1}{60 \times 10^{-6} \text{m}} < \sqrt{10 \times 10^6 \text{1/m} \leq \sqrt{\rho}} . \]

Therefore, the dislocation mean free path is mainly governed by the square root of dislocation density, i.e. \( \frac{1}{L_i} \propto \sqrt{\rho} \). If the total dislocation density is incorporated as only one dislocation density parameter into the evolution of the dislocation mean free path, then \( \frac{1}{L_i} = k_i \sqrt{\rho} \) with \( k_i \approx 8 \) to \( 80 \) in order to take account of the formation of dislocation substructures. During cyclic loading of planar-slip materials, dislocations evolve from planar configurations to dislocation
wall/channel structures, resulting in the most significant changes in the cyclic
deformation response. It is insufficient to describe the transient stage of dislocation
evolution using the total dislocation density as only one microstructural parameter and
a proportionality constant, \( k_i \approx 8 \) to 80. In this study, the dislocation densities
associated with arrangements involving different inhomogeneous plastic deformation
conditions are treated differently. The geometrical configuration of dislocation
substructures is taken into account by defining the wall fraction \( f_w \). The dislocation
mean free paths associated with different kinds of dislocation arrangements are
spontaneously different, and inherently evolve with the density of corresponding
dislocation populations. Moreover, it has been shown that \( \frac{1}{L_i} = k_i \sqrt{\rho_i} \) (with \( k_i = 1 \)) is
very applicable for condition where dislocations are present in the Taylor lattice
configuration, where there is no dislocation wall/channel structure [40]. The equality,
\( \frac{1}{L_i} = k_i \sqrt{\rho_i} \) (with \( k_i = 1 \)), is therefore used in this current approach.

In these circumstances, Eqn. (VI.1) becomes

\[
\frac{d \rho}{k_1 \sqrt{\rho} - k_2 \rho} = d \varepsilon_p
\]

(VI.2)

Eqn. (VI.2) is solved in the Appendix 1 to become:

\[
\sqrt{\rho} = \frac{k_1}{k_2} \left( 1 - e^{-k_2 \varepsilon_p} \right)
\]

(VI.3)

with \( k_1' = k_1 / 2 \), and \( k_2' = k_2 / 2 \)

Essmann and Mughrabi used the same form of Eqn. (VI.1) to successfully describe the
evolution of dislocation densities within walls and channels [148]. In addition, GNDs
(which are present due to inhomogeneous plastic deformation between different
strength phases) can be related to corresponding internal stresses in the same way as
the Taylor relationship [144, 149]. Estrin et al. [174] following the Mughrabi approach
similarly showed that each type of dislocation population inside walls and channels can relate to corresponding internal stresses. In other words, a given internal stress ($\sigma_i$) can then relate to a specific type of dislocation ($\rho_i$) by the Taylor-like relationship:

$$\sigma_i = M\mu b\sqrt{\rho_i}$$  \hspace{1cm} (VI.4)

Where: $M$ is the average Taylor factor, which describes the crystallographic dependence of external stress on shear stress. During plastic deformation, the presence and the slip of dislocations induce changes in crystallographic orientation, resulting in the evolution of Taylor factor in particular for large plastic deformation [175, 176]. Necessary dislocations preferentially align to a certain crystallographic orientation to accommodate plastic strain incompatibilities. The presence of GNDs and their movement induce crystal rotation much more significantly than those of other types of dislocation. The Taylor factor should therefore be different for different kinds of dislocation, and can evolve during loading. In the case of small plastic deformation, the value of $M$ is considered constant (=3), and the same for all kinds of dislocations for the sake of simplicity. $\alpha = 0.4$ is a geometric constant of the elastic interaction of dislocations, $\mu$: Shear modulus, $b$: Magnitude of Burger’s vector ($b=0.258\text{nm}$)

Combining Eqns. (VI.3) and (VI.4) gives:

$$\sigma_i = \frac{C_i}{g_i} \left( 1 - e^{-g_i^* \epsilon_p} \right)$$ \hspace{1cm} (VI.5)

with $C_i = M\alpha\mu b k_1^i$, $g_i = k_2^i$
VI.3. Dislocation-based model for evolutionary equations of internal stresses

The stress during the plastic hysteresis loop has been constitutively separated into effective stress and the superposition of an inter-granular and an intra-granular back stress. The total stress response of the material can thus be described by a superposition of terms as in Eqn. (VI.5), in which *i* is summed over “inter”, “intra” and “effective” stress components. In the following sections, a description is given of how the cyclic evolution of the stress response can be modelled by establishing the relationship between \( C_{\text{inter}} / g_{\text{inter}} \) (and \( C_{\text{intra}} / g_{\text{intra}} \)) and the evolution of dislocation condition with respect to the cyclically accumulated plastic strain. The modelling equation set representing such relationships has been developed by the author on the basis of intensive microstructural and mechanical characterisations as presented in Chapters III-V.

VI.3.1. Inter-granular back stress

The cyclic plastic deformation hysteresis is in part due to an inter-granular back stress that relates to the plastic strain \( \varepsilon_p \) according to Eqn. (VI.5) as follows,

\[
X_{\text{inter}} = \left( \frac{C_{\text{inter}}}{g_{\text{inter}}} \right) \times \left( 1 - e^{-g_{\text{inter}} \varepsilon_p} \right) \tag{VI.6}
\]

The microstructural condition also evolves from cycle to cycle which is captured by the description of the evolution of \( C_{\text{inter}} / g_{\text{inter}} \). As explained in Sect. V.2, \( C_{\text{inter}} / g_{\text{inter}} \) does not simply depend only on a specific type of dislocation, but rather on several dislocation features. In the beginning, it increases significantly with the increase in boundary dislocations, and reduces its rate of increase upon further loading due to the activity of secondary slip (which results in higher numbers of dislocations inside grains).
It finally reaches a saturation value once the stabilised condition is established. \( C_{\text{inter}} / g_{\text{inter}} \) during cyclic loading can be consequently expressed by:

\[
2C_{\text{inter}} / g_{\text{inter}} = M \alpha G b \times \left[ f_b \times \left( \sqrt{\rho_b} - \sqrt{\rho_I} \right) + \sqrt{\rho_{\text{GNDs-inter}}} \right]
\]  

(VI.7)

Where: \( \rho_{\text{GNDs-inter}}^{\text{sat}} \) is the minimum of GNDs which are required to accommodate grain-to-grain plastic strain incompatibility at the stabilised condition. These GNDs can disperse over grains, but are mainly present in regions close to boundaries between sub-domains (e.g. fragmented volumes). Although these GNDs cannot be correctly measured by TEM as mentioned in Sect. V.2.2, \( \rho_{\text{GNDs-inter}}^{\text{sat}} \) can be determined if \( X_{\text{inter}} \) at the stabilised condition is known.

Assuming one can derive the evolution of both boundary and interior dislocations (\( \rho_b \) and \( \rho_I \)) with respect to \( p \) according to Eqn. (VI.3), the evolution of \( C_{\text{inter}} / g_{\text{inter}} \) with respect to \( p \) can be written as:

\[
\frac{2C_{\text{inter}}}{g_{\text{inter}}} = M \alpha G b \times \left[ f_b \times \left( \frac{k_{2-b}}{k_{1-b}} \left( 1 - e^{-k_{2-b}p} \right) - \frac{k_{2-I}}{k_{1-I}} \left( 1 - e^{-k_{2-I}p} \right) \right) + \rho_{\text{GNDs-inter}}^{\text{sat}} \right]
\]  

(VI.8)

Here:

\[
\frac{k_{2-b}}{k_{1-b}} \left( 1 - e^{-k_{2-b}p} \right), \text{ and } \frac{k_{2-I}}{k_{1-I}} \left( 1 - e^{-k_{2-I}p} \right)
\]

respectively represent the evolution of \( \rho_b \) and \( \rho_I \) with respect to \( p \).
\[ k_{1-b} / k_{2-b} = \sqrt{\rho_{b}^{hom}}, \quad \text{and} \quad k_{1-I} / k_{2-I} = \sqrt{\rho_{I}^{hom}} \]

are respectively the square root densities of boundary dislocations and interior dislocations at the end of the cyclic hardening stage when dislocations are distributed rather homogeneously throughout grains \((k_{1-b} / k_{2-b} \geq k_{1-I} / k_{2-I})\). \(k_{2-b}\), and \(k_{2-I}\) respectively characterise the rates of change of \(\rho_{b}\) and \(\rho_{I}\) with respect to \(P\) (where \(k_{2-b} \gg k_{2-I}\)).

\(f_{b}\): is the area fraction over which boundary dislocations occupy. TEM observation shows that the regions of boundary dislocations are usually smaller than 20% of the whole grains \(f_{b} \leq 0.2\).

**VI.3.2. Intra-granular back stress**

Following the same approach used for the formulation of inter-granular back stress, and based on Eqn. (VI.5) and Sect.V.2.2, \(X_{\text{intra}}\) during the plastic deformation of a cycle can be represented by the following equation:

\[
X_{\text{intra}} = \left( C_{\text{intra}} / g_{\text{intra}} \right) \times \left( 1 - e^{-g_{\text{intra}}^{2} p} \right) \tag{VI.9}
\]

The evolution of \(X_{\text{intra}}\) is proportional to the development of dislocation high/low density structures, e.g. dislocation walls/channels. \(C_{\text{intra}} / g_{\text{intra}}\) relates to the change of \(\rho_{\text{GNDs-intra}}\) which are present in the interface between high density regions (e.g. walls) and low density regions (e.g. channels) of dislocations. Dislocation high/low density regions are always present even in the as-received solution treated condition. \(2C_{\text{intra}} / g_{\text{intra}}\) thereby equals \(M a \mu b \sqrt{\rho_{\text{GNDs-intra}}^{0}}\) at the beginning. \(\rho_{\text{GNDs-intra}}^{0}\) is the initial value of \(\rho_{\text{GNDs-intra}}\) and should be close to zero if the dislocation density in the as-received condition is very low. This type of GND can be proportional to the difference between square root densities of dislocations of hard phase (\(\rho_{w}\)) and of soft phase (\(\rho_{s}\)).
\( \rho_c \), and the fraction of hard phase \((f_w) [144, 149, 150]. \) \( C_{\text{intra}} / g_{\text{intra}} \) increases with \( P \) until \( \rho_w, \rho_c \) and \( f_w \) obtain their saturation values.

Therefore,

\[
\rho_{\text{GNDs-intra}} = f_w^{sat} \times \left\{ \left( \sqrt{\rho_w^{sat}} - \sqrt{\rho_c^{sat}} \right) \left[ 1 - e^{-k_{2-w} P} \right] + \sqrt{\rho_{\text{GNDs-intra}}^0} \right\} \\
= f_w^{sat} \times \left\{ \Delta \sqrt{\rho_{w-c}} \left[ 1 - e^{-k_{2-w} P} \right] + \sqrt{\rho_{\text{GNDs-intra}}^0} \right\}
\]  

(VI. 10)

Combining Eqns. (VI.10) with (VI.5), the evolution of \( C_{\text{intra}} / g_{\text{intra}} \) is thereby written as

\[
2C_{\text{intra}} / g_{\text{intra}} = M \alpha G b f_w^{sat} x \left\{ \Delta \sqrt{\rho_{w-c}} \left[ 1 - e^{-k_{2-w} P} \right] + \sqrt{\rho_{\text{GNDs-intra}}^0} \right\} 
\]  

(VI.11)

Where \( k_{2-w} \) relates to the rate of development of dislocation walls with respect to \( P \).

The rate of dislocation rearrangement (which results in the formation of dislocation structures) is usually smaller than the rate of change in dislocation density, i.e.

\( k_{2-w} \leq k_{2-f} \).

\( \rho_w^{sat} \) is the saturated value of dislocation density within the walls. In the case of well-organised cell structures, \( \rho_w^{sat} \) can be calculated from the cell size \((d_c)\) and the cell-to-cell misorientation \((\theta) [177-179].\)

\( \rho_c^{sat}, \) and \( f_w^{sat} \) are the saturated values of dislocation density in the channels, and of the area fraction of walls.

\( d_c, \theta, \rho_c^{sat} \) and \( f_w^{sat} \) can be quantified by TEM investigation.
VI.3.3. Effective stress

The evolution of effective stress during cyclic loading is modeled by

$$\sigma_E = \sigma_E^0 + \sigma_E^R \quad (VI.12)$$

Where: $\sigma_E^0$ and $\sigma_E^R$ are respectively the initial value of effective stress, and the increment of effective stress during cyclic loading. The short range interaction of dislocation between themselves is the main cause of the increment of effective stress at 20°C, while interactions of dislocations with solute atoms and later in life with the corduroy structure additionally contribute to the change in effective stress at 300°C (Sect. V.3). The increment of effective stress ($\sigma_E^R$) can be decomposed into three components:

1) The first effective stress component associates with the short range interaction of dislocations with themselves. This type of effective stress is called $\sigma_E^{R(dis)}$. At 20°C, this effective stress component dominates and overwhelms other components.

2) The second one ($\sigma_E^{R(s)}$) is due to the short range interaction of dislocations with solute atoms.

3) The third one ($\sigma_E^{R(c)}$) is due to the short range interaction between dislocations and the corduroy structure.

VI.3.3.1. Evolution of effective stress associating with the dislocation-dislocation interaction

Lomer-Cottrell dislocation/dislocation interactions take place due to the activity of secondary slip, resulting in the formation of sessile dislocation junctions. The rise in the
number of sessile Lomer-Cottrell junctions thanks to the activity of secondary slip causes the increase in the effective stress. The activity of secondary slip also contributes to the abundance of interior dislocations. Therefore, the density of these junctions can be approximated as a fraction \((f_j)\) of grain interior dislocations \((\rho_I)\). Consequently, the \(\sigma_{E}^{R\,(dis)}\) relates to \(\rho_I\).

\[
2\sigma_{E}^{R\,(dis)} = M\alpha \mu b \sqrt{\rho_I} = M\alpha \mu b f_j \rho_I = M\alpha \mu b \left(\frac{k_{1-j}}{k_{2-j}}\right) \times \left(1 - e^{-k_{2-j}P}\right) \tag{VI.13}
\]

Where: \(\frac{k_{1-j}}{k_{2-j}} = \sqrt{\rho_{I}^{att}} = \sqrt{f_j \rho_{I}^{att}}\),

and \(k_{2-j}\) characterises the rate of change of dislocation sessile junctions.

\[
k_{2-j} = [k_{2-1}, \ k_{2-b}]
\]

Therefore,

\[
2\sigma_{E}^{R\,(dis)} = 2\sigma_{E}^{0} + M\alpha \mu b \left[ \frac{k_{1-j}}{k_{2-j}} \left(1 - e^{-k_{2-j}P}\right) \right] \tag{VI.14}
\]

**VI.3.3.2. Evolution of the change of effective stress due to interactions of dislocations with other types of point defects**

The interaction between dislocations and solute atoms (C, and N) leads to the formation of Suzuki atmospheres and later in fatigue life to the formation of Snoek atmospheres (Sect. IV.4.5). The observed change in the intensities of both Max DSA transient and Serration length (Figure VI.1) indicates that the strength of Suzuki atmospheres is greater than that of Snoek atmospheres. The contribution of the solute atom-dislocation interaction to effective stress is mainly dominant during the first cyclic hardening response phase where the Suzuki solute atom atmospheres are strongly active. Later in fatigue life, this contribution is however gradually overwhelmed by the
development of the corduroy structure (Sect. IV.4.7) [134]. In addition, at 300°C, solute atom atmospheres effectively lock dislocations, resulting in a higher stored dislocation density during cyclic loading. The contribution of this interaction to the increment of effective stress can be represented by the higher increase in dislocation densities at 300°C, and can be incorporated into $\sigma_E^{R\text{(dis)}}$ by using a higher value of $k_{1-j}/k_{2-j}$. The value of $k_{1-j}/k_{2-j}$ at 300°C can be obtained on the basis of the dislocation density measurement during the cyclic primary hardening at 20 and 300°C, e.g. Figure V.4.

Figure VI.1: The DSA activities during cyclic loading at: (a)-(b) $\pm 0.7\%-10^3\text{s}^{-1}\text{-}300^\circ\text{C}$, and (c)-(d) $\pm 0.4\%-10^3\text{s}^{-1}\text{-}300^\circ\text{C}$

After a high number of cycles during cyclic loading at 300°C, the corduroy structure starts to continuously develop. The interaction between corduroy structure and dislocations results in considerable increase in the effective stress, leading to the secondary cyclic hardening. Since the additional increase in effective stress during this
The cyclic hardening stage is well represented by a linear function in the $\sigma_E (\log p)$ scale, the $\sigma_E^{R(c)}$ can be therefore expressed by the following logarithmic function:

$$2\sigma_E^{R(c)} = a_c \{ \log p - \log p_c \}$$

(VI.15)

Where: the MacCauley bracket $\{ x \} = \begin{cases} x & \text{if } x \geq 0 \\ 0 & \text{if } x < 0 \end{cases}$

$p_c$ is the plastic strain accumulated to form the well-developed corduroy structure. The accumulated plastic strain $p_c$ is dependent on strain amplitude. The lower strain amplitude, the earlier formation of well-organised corduroy structure. The relationship between the $p_c$ and the imposed plastic strain amplitude ($\varepsilon^a_p$) is established (Figure VI.2):

$$p_c = 16730 (\varepsilon^a_p)^3$$

![Graph](image)

Figure VI.2: The relationship between the accumulated plastic strain to reach the end of the cyclic softening phase and imposed plastic strain amplitude.
The secondary cycle hardening phase is observed for both the ±0.25 and ±0.4% conditions. The rate of additional increase in the effective stress during the beginning of this cyclic response stage are the same for both loading cases (Figure VI.3). The earlier presence of fatigue cracks for the ±0.4% test however interferes with the macroscopic manifestation of the change in the effective stress during the secondary cyclic hardening response. If fatigue cracks would be not formed earlier for ±0.4% test, the rate of change of effective stress should be similar to that of the ±0.25% condition. The model of the evolution of effective stress associating with the development of the corduroy structure is therefore made on the basis of the observed increase in $\sigma_E$ for the ±0.25% condition. Fitting the increment of effective stress with respect to $p$ gives:

$$2\sigma_E^{R(c)} = 9.56\{ \log p - \log p_c \}$$

(VI.16)
VI.3.3.3. Evolution of effective stress

From Eqn. VI.12, VI.14 and VI.16, the evolution of the effective stress due to the short range interaction of dislocations with themselves and with other point defects is represented by:

\[
2\sigma_E = 2\sigma_E^0 + M\alpha\mu b \left[ \frac{k_{1-j}}{k_{2-j}} \left(1 - e^{-k_{2-j}p} \right) \right] + 9.56\{\log p - \log p_c\} \quad (VI.17)
\]
VI.4. Modelling the evolution of maximum stress during cyclic loading

From Eqns. (VI.8), (VI.11), and (VI.17), the evolution of stress range versus the accumulated plastic strain curve is expressed by Eqn. (VI.18):

\[
\Delta \sigma = M \alpha G b \left\{ f_b \times \left[ k_{1+b} \left( 1 - e^{-k_{2+b}P} \right) - k_{1+b} \left( 1 - e^{-k_{2+b}P} \right) \right] + \sqrt{f_{GNDs} \times \rho_{sat}^{GNDs}} \right\} \left\{ 1 - e^{-s_{intra}\varepsilon_p^u} \right\} \\
+ M \alpha G b \left\{ f_w \times \left[ \Delta \rho_{W-\varepsilon}^{GNDs} \times \left( 1 - e^{-k_{2-w}P} \right) + \sqrt{\rho_{GNDs-intra}^{GNDs}} \right] \right\} \left\{ 1 - e^{-s_{intra}\varepsilon_p^u} \right\} \\
+ 2\sigma_r^0 + M \alpha G b \left[ k_{1+b} \left( 1 - e^{-k_{2+b}P} \right) \right] + 9.56 \{ \log p \cdot \log p_c \}
\]
VI.5. Application to model cyclic deformation response at 20°C

The application of the developed model is presented in this section. The parameter identification procedure is first discussed. Identification of the model parameters requires a knowledge of the dislocation density in the high density regions (e.g. dislocation walls). While this dislocation density is difficult to measure, it can be calculated when the cellular structure is well organised. The ±0.7% strain amplitude test results are therefore chosen to identify model parameters since a well-organised cellular structure is observed for this fatigue mid-life condition (Figure IV.4). The capability of the model is then used to predict the cyclic deformation response for other test conditions. However, on account of the strain amplitude dependence of dislocation evolution characteristics (Sect. IV.3.2) and its consequences relating to change in the internal stresses (Sect. V.4), three model parameters need to be adjusted to accurately predict the cyclic deformation response of the material for lower strain amplitudes.

VI.5.1. Parameter identification

VI.5.1.1. Identifying parameters which characterise flow stress at the stabilised condition

It is first necessary to identify a stabilised cycle condition, i.e. one for which dislocations are present in their most stable configuration. This is a cycle for which the back stresses and the effective stress attain their corresponding saturation values. It is also important that fatigue cracks do not have a noticeable influence on material elastoplastic response during the stabilised cycle to ensure that flow stress is due only to dislocation motion. At the cycle 1500 of the ±0.7% - 10^{3}s^{-1} - 20°C condition, stable dislocation structures already form, and there is no significant influence of the presence of fatigue cracks on the stabilised deformation response of the material. In other words, this cycle fulfils the requirements to identify microstructural parameters for characterising flow stress at the stabilised response condition.
Figure VI.4: The quantification of important dislocation features: (a) The density of boundary dislocations ($\rho_b$), (b) The density of interior dislocations ($\rho_i$), (c) The density of dislocations inside of channels ($\rho_c$), wall thickness ($d_w$), and wall spacing ($d_c$).
Figure VI.5: (a) A TEM image of dislocation wall/channel structure and (b) corresponding selected area diffraction pattern/Kikuchi lines for channel B. The analysis of Kikuchi lines at locations A and B gives the misorientation between A and B to be about 0.29 to 0.31°.

(Note: yellow lines set the reference, dashed red lines represent the position of the Kikuchi lines at location B which corresponds to those (blue lines) at location A).

Figure VI.6: Quantification of dislocation condition during cyclic loading at ±0.7% - 10^{-3}s^{-1} - 20°C (see also Sect. IV.1, and Sect. VI.5.1.2)
Since dislocations within walls are extremely dense, it is impractical to directly measure the wall dislocation density from TEM images, even with the Dark-field technique. Therefore, this study first quantifies dislocation densities in the channels ($\rho_c$), wall dimensions (i.e. wall spacing ($d_w$) and wall thickness ($d_d$)), and the cell-to-cell misorientation ($\theta$) (Figure VI.4 and Figure V.6a). The wall dislocation density is then calculated through the cell-to-cell misorientation and the cell size. As shown in Figure VI.6, the channel dislocation density slightly falls off, and attains an almost constant value after the midlife cycle. The thickness of dislocation walls was about 0.21 µm at the beginning, and then deceased to 0.11 µm, which is consistent with a slight increase in channel width. The average cell-to-cell misorientation carefully measured by the shift and the tilt of Kikuchi patterns is about 0.33° (=0.0058 rad) at the midlife condition (Figure VI.5). The cell-to-cell misorientation can vary usually from 0.2° in the middle of a sub-domain to 1° if one of two cells belongs to different sub-domains. The dislocation density within walls at the stabilised condition can therefore be calculated based on the information of the cell-to-cell misorientation and the cell size as expressed in Eqn. (VI. 22) [177-179]:

$$\rho_w^{sat} = \frac{\kappa \theta}{b d_c}$$  \hspace{1cm} (VI. 19)

Here: $\kappa$ is a geometric constant, and is of the order from 3 – 21 ([177-179]).

$\theta$ is the cell-to-cell misorientation which was of 0.0058 rad at the midlife cycle.

$d_c$ = 0.5 µm

$b$: Magnitude of Burgers vector, $b$ = 0.258 nm

$$\rho_w^{sat} \bigg|_{\kappa=3, 12, and 21} = \frac{\kappa \times 0.0058}{2.58 \times 10^{-9} \times 0.5 \times 10^{-6}} \bigg|_{\kappa=3, 12, and 21}$$

$$= 1.4 \times 10^{14}, 5.4 \times 10^{14}, \text{ and } 9.4 \times 10^{14} \text{ }/\text{m}^2$$
Since dislocations within walls are usually very high (normally in the order of magnitude of \(10^{15} \text{ 1/m}^2\)), the calculated values with \(\kappa = 12 - 21\) give a more reasonable estimation of \(\rho_w^{sat}\) (i.e. \(\rho_w^{sat} = 5.4 \times 10^{14} - 9.4 \times 10^{14} \text{ 1/m}^2\)), which is used for subsequent calculations.

\(\rho_c^{sat}\) for the midlife condition is about \(4 \times 10^{13} \text{ 1/m}^2\).

Therefore, \(\Delta \sqrt{\rho_{w-c}} = \sqrt{\rho_w^{sat}} - \sqrt{\rho_c^{sat}} = 1.7 \times 10^7 - 2.4 \times 10^7 \text{ 1/m}\).

Moreover, the average value of wall fraction is \(f_w^{sat} = \frac{d_w}{d_w + d_c} = \frac{0.11}{0.11 + 0.5} = 0.18\).

The difference in dislocation density between high density and low density dislocation regions in the as-received condition (\(\sqrt{\rho_{GND-intra}}\)) is about \(5 \times 10^{13} \text{ 1/m}^2\). The average value of intra-granular back stress at the balanced condition is therefore calculated according to Eqn. VI.11 to be:

\[
2X_{intra}^{sat} = \frac{C_{intra}^{sat}}{g_{intra}} \left(1 - e^{-\frac{g_{intra}}{E} p}\right) = M \alpha Gb \left(f_w^{sat} \left(\Delta \sqrt{\rho_{w-c}} + \frac{\sqrt{\rho_{GND-intra}}}{\rho_{GND-intra}}\right)\right) = 96 - 128 \text{ MPa}
\]

From Eqns. (VI.6), (VI.9) and (VI.12), the flow stress of the reversal loading from compression peak of the \(\pm 0.7\%\)-condition is represented by:

\[
2\frac{C_{intra}^{sat}}{g_{intra}} \left(1 - e^{-\frac{g_{intra}}{E} p}\right) + 2\frac{C_{intra}^{sat}}{g_{intra}} \left(1 - e^{-\frac{g_{intra}}{E} p}\right) + 2\sigma_E^{sat}\quad (\text{VI.20})
\]

Since every microstructural parameter is obtained with error bands, it is required to perform a fitting of the flow stress of the reversal loading from compression peak stress by Eqn. VI.20 to get the best fit within the scatter bands. The flow stress of the reversal
loading from compression peak of the stabilised cycle is fitted by Eqn. 20 (Figure VI.7) with the following constraints:

\[ + 2 \frac{C_{\text{intra}}^{\text{sat}}}{g_{\text{intra}}^{\text{sat}}} \left(1 - e^{-g_{\text{intra}}^{\text{sat}} E_p^{\text{a}}} \right) = 96 - 128 \text{MPa} \]

\[ + \frac{C_{\text{inter}}^{\text{sat}}}{g_{\text{inter}}^{\text{sat}}} > \frac{C_{\text{intra}}^{\text{sat}}}{g_{\text{intra}}^{\text{sat}}}; \text{This is because the inter-granular back stress acts over the grain-to-grain scale which is longer range than the intra-granular scale, i.e. the contribution of } \chi_{\text{inter}} \text{ to the flow stress is higher than } \chi_{\text{intra}} \]

+ and \( 2 \sigma_{\varepsilon}^{\text{sat}} = 236 \text{ MPa} \) measured from the same hysteresis loop of the qualified cycle.

The fitting gives \( 2 \frac{C_{\text{intra}}^{\text{sat}}}{g_{\text{intra}}^{\text{sat}}} \left(1 - e^{-g_{\text{intra}}^{\text{sat}} E_p^{\text{a}}} \right) = 115 \text{MPa} \) and values for other parameters of Eqn. VI.20 (Table VI.1).
Figure VI.7: Identification of internal stresses at the stabilised response condition.

The inter-GNDs at the stabilised condition is therefore calculated as follows

\[
\rho_{\text{GNDs-inter}}^{\text{int}} = \left( \frac{2C_{\text{int}}^{\text{int}} \left( 1 - e^{-g_{\text{int}}^{\text{int}} \varepsilon_{p}} \right) }{g_{\text{int}}^{\text{int}} M \alpha Gb} \right)^2 \approx 0.8 \times 10^{14} \text{ m}^{-2}
\]

Considering that \(2X_{\text{intra}}^{\text{int}} = 96–128\), the lower and upper limits of \(\rho_{\text{GNDs-inter}}^{\text{int}}\) should be about \(0.6 \times 10^{14} \text{ m}^{-2}\) and \(1 \times 10^{14} \text{ m}^{-2}\), respectively.

<table>
<thead>
<tr>
<th>(C_{\text{inter}}^{\text{int}}) (MPa)</th>
<th>(g_{\text{inter}}^{\text{int}})</th>
<th>(C_{\text{intra}}^{\text{int}}) (MPa)</th>
<th>(g_{\text{intra}}^{\text{int}})</th>
<th>(\sigma_{E}^{\text{int}}) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>4.9</td>
<td>105</td>
<td>1.55</td>
<td>118</td>
</tr>
</tbody>
</table>
VI.5.1.2. Identifying parameters of evolutionary equations of internal stresses

Model equations representing the evolution of inter-granular and intra-granular back stresses, and effective stress are given in Eqs. (VI.8), (VI.11) and (VI.14), respectively. In order to identify material parameters in these equations, it is necessary to measure the microstructural features defining $\rho_b$, $\rho_1$, $\rho_c$, $d_c$, $d_w$, $\theta$, $f_w$, $\rho_w$ as a function of the cycle number (or $p$). This was intensively done by TEM for the $\pm 0.7\%$-condition (Figure VI.6). TEM measurements were made from at least five images taken from different locations. The scatter band of the TEM measurement provides values for the upper and lower limits of the required parameters.

At 20°C, there is no strong interaction between dislocations with other types of point defects, i.e. $\sigma_E = \sigma_E^0 + \sigma_E^{R(d)}$. Moreover, the material exhibits a stabilised cyclic deformation response after a large number of cycles. The yield stress reaches its saturation value ($\sigma_E^{R(d)\text{-sat}}$) after a high number of cycles. The yield stress at the stabilised condition is calculated by $\sigma_E^{\text{sat}} = \sigma_E^0 + \sigma_E^{R(d)\text{-sat}}$. The initial value of yield stress is therefore, $\sigma_E^0 = \sigma_E^{\text{sat}} - \sigma_E^{R(d)\text{-sat}}$. Since most of the model parameters are required to identify at the stabilised condition, it is more convenient to represent the evolution of yield stress by the following equation.

\[
2\sigma_E = 2\sigma_E^{\text{sat}} - 2\sigma_E^{R(d)\text{-sat}} + 2\sigma_E^{R(d)} \\
= 2\sigma_E^{\text{sat}} - M_{\alpha \mu b} \frac{k_{1-j}}{k_{2-j}} + M_{\alpha \mu b} \left[ \frac{k_{1-j}}{k_{2-j}} \left( 1 - e^{-k_{2-j}p} \right) \right] \\
= 2\sigma_E^{\text{sat}} - M_{\alpha \mu b} \frac{k_{1-j}}{k_{2-j}} e^{-k_{2-j}p}
\]  

(VI. 21)
For the room temperature condition, the Eqn. VI.18 therefore becomes

\[
\Delta \sigma = M \alpha G b \left\{ f_x \left[ k_{1-b} \left( 1 - e^{-k_{2-b} p} \right) - k_{2-b} \left( 1 - e^{-k_{2-b} p} \right) \right] + \sqrt{f_{\text{GND}} \times \rho_{\text{sat}}} \left( 1 - e^{-g_{\text{sat}} \bar{\sigma}^{\text{sat}}} \right) \right\} \\
+ M \alpha G b \left\{ f_x \left[ \Delta \rho_{\text{sat}}^{\text{sat}} \times \left( 1 - e^{-k_{2-b} p} \right) + \sqrt{f_{\text{GNDs-intra}} \times \rho_{\text{intra}}} \left( 1 - e^{-g_{\text{intra}} \bar{\sigma}^{\text{intra}}} \right) \right] \right\} \\
+ 2\sigma_x^{\text{sat}} - M \alpha G b \frac{k_{1-j}}{k_{2-j}} e^{-k_{2-j} p}
\]"", (VI. 22)

**Figure VI.6** shows the TEM quantification of dislocation features during cyclic loading. Boundary dislocations are observed to significantly increase during the early part of the hardening response stage, whereas interior dislocations increase with a lower rate. After a number of cycles, due to the activity of secondary slip, interior dislocation density finally reaches a value which is slightly less than that for boundary dislocations (**Figure IV.4c**). Interior dislocations at the end of cyclic hardening are however abundant, and occupy a much larger area than that of boundary dislocations. To make \( \rho_b \) reach the saturated value after about 30 cycles, \( k_{2-b} \) falls in the range of 0.06 - 0.08. \( \rho_I \) obtains its saturation value once the generation rate and annihilation rate of interior dislocations are almost equal, i.e. walls and channels are pretty well formed. At the ±0.7%-condition, wall and channel structures can rather well form at the earliest after about 400 cycles, \( k_{2-I} \) should therefore fall in the range from 0.004 to 0.005.

The result of the microstructural quantification provides values of \( k_{1-b} / k_{2-b} = \sqrt{\rho_b^{\text{sat}}} \), \( f_x / k_{2-b} = \sqrt{\rho_I^{\text{sat}}} \), \( \Delta \rho_{\text{GNDs-intra}}^{\text{sat}} \), \( \Delta \rho_{\text{GNDs-inter}}^{\text{sat}} \) which are summarised in **Table VI.2**. By fitting the stress range versus the accumulated plastic strain using Eqn. VI.22 with upper and lower limits of measured parameters, the final values of measured parameters \((\sqrt{\rho_b^{\text{sat}}}, f_x; \sqrt{\rho_I^{\text{sat}}}; \rho_{\text{GNDs-intra}}^{\text{sat}}; f_x^{\text{sat}}; k_{2-b}; k_{2-I}; \Delta \rho_{\text{GNDs-inter}}^{\text{sat}})\) and unmeasured parameters (i.e. \( f_x, k_{2-I} / k_{2-b} = \sqrt{\rho_I^{\text{sat}}} \)) are identified. With the obtained values of model parameters, the evolution of back stresses can be modelled as shown in **Figure VI.8**.
Table VI.2: The parameter identification of Eqn. (VI.22)

<table>
<thead>
<tr>
<th></th>
<th>$\rho_{b}^{\text{hom}}$ ($x10^{14}$)</th>
<th>$f_b$ ($x10^{14}$)</th>
<th>$\rho_{I}^{\text{hom}}$ ($x10^{14}$)</th>
<th>$k_{2-I}^{\text{approx}}$ ($x10^{14}$)</th>
<th>$\rho_{\text{GND-intra}}^{\text{sat}}$ ($x10^{14}$)</th>
<th>$\Delta \rho_{\text{inter}}^{\text{sat}}$ ($x10^{7}$)</th>
<th>$f_{w}^{\text{sat}}$</th>
<th>$\rho_{j}^{\text{sat}}$ ($x10^{14}$)</th>
<th>$k_{2-j}^{\text{sat}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Unit</strong></td>
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<td>1/m$^2$</td>
<td>1/m$^2$</td>
<td>1/m</td>
<td>1/m</td>
<td>1/m</td>
<td></td>
<td>1/m$^2$</td>
<td></td>
</tr>
<tr>
<td><strong>Lower</strong></td>
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<td>0.06</td>
<td>0.1</td>
<td>0.8</td>
<td>0.004</td>
<td>0.6</td>
<td>1.7</td>
<td>0.5</td>
<td>0.004</td>
</tr>
<tr>
<td><strong>Upper</strong></td>
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<td>0.08</td>
<td>0.2</td>
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<td>0.005</td>
<td>1.0</td>
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<tr>
<td><strong>Identified</strong></td>
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<td>0.0665</td>
<td>0.2</td>
<td>1.0</td>
<td>0.0046</td>
<td>1.0</td>
<td>0.5</td>
<td>1.9</td>
<td>0.18</td>
</tr>
</tbody>
</table>

Figure VI.8: Modelling the evolution of back stresses during cyclic loading
VI.5.2. Discussions

An effective constitutive cyclic plasticity model equation set based on microstructural observations has been developed to represent the complex hardening, softening and stabilised responses of AISI 316L during cyclic loading at 20°C. This equation set comprises relationships which define the stress-strain hysteresis loop and the way it evolves during fatigue loading. In particular, the stress-strain hysteresis loop is defined by Eqn. VI.20, which originates from Eqns. VI.6, 9 and 12; and the evolution is defined by Eqn. VI.22, which originates from Eqns. VI.8, 11 and 14.

Multiaxial loading facilitates strong multiple slip activity, resulting in the more rapid formation of dislocation tangled structures, i.e. dislocation walls and channels quickly form during multiaxial loading [180]. The evolution of microstructural condition during multiaxial cyclic loading is found to be qualitatively similar to that during uniaxial condition. The dislocation condition basically evolves from planar structures to the wall/channel structure during multiaxial cyclic loading for AISI 316L [180, 181]. Consequently, if one appropriately considers the latent hardening and the more rapid development of dislocation wall/channel structure, the proposed model of the evolution of internal stresses can be readily expanded to multiaxial cyclic loading situations. Moreover, the similarity of Eqn. VI.19 with the two term back stress equation of Chaboche [140] means that this new microstructurally based equation set will be readily implementable in commercial finite element programs such as Abaqus and Ansys.

VI.5.2.1. Modelling of Cyclic Deformation Response for ±0.7% (10⁻³ s⁻¹ - 20°C)

For a strain amplitude of ±0.7%, a strain rate of 10⁻³ s⁻¹, and a temperature of 20°C, Figure VI.9 shows that the stress-strain hysteresis loop and evolutionary cyclic deformation responses of AISI 316L are very well predicted using the proposed equation set and the microstructurally determined parameters summarised respectively in Table VI.1 (Eqn. VI.20) and Table VI.2 (Eqn. VI.22).
VI.5.2.2. Modelling of Cyclic Deformation Response for ±0.4, and ±0.25% (10^{-3}s^{-1} - 20^\circ C)

It is clear from comparing the microstructural conditions observed in LCF tests conducted with strain amplitudes of ±0.7% (Figure IV.4), ±0.4% (Figure IV.13), and ±0.25% (Figure IV.14) in Sect. IV.3, and Figure V.5 and Figure V.6, that the microstructural evolution characteristics are indeed dependent on the strain amplitude, resulting in different internal stress state evolution characteristics for lower strain amplitudes (Sect. V.4). It is necessary to compensate for the strain amplitude dependence of all the model parameters identified with respect to a strain amplitude of ±0.7% (Table VI.1 and Table VI.2) to model the cyclic deformation response for lower strain amplitude (±0.25 and 0.4%) conditions. In fact, on the basis of the influence of strain amplitude on microstructural condition and its consequence on the internal stress state (Sect. V.4), and the insignificant role of effective stress on cyclic response compared with those of back stresses, there is a need to adjust only three parameters in Eqn. VI.22 (i.e. $\int_b \mu$, $\sqrt{\mu_{\text{hom}}^{\text{hom}}}$ and $\Delta \sqrt{\mu_{\text{hom}}^{\text{hom}}}$) to better represent the evolution of $X_{\text{inter}}$ and $X_{\text{intra}}$.
Understanding the relationships between internal stresses and the microstructural evolution (Sects. V.2 and V.4, Figure V.5a and Figure V.6a) provides useful information to substantiate and identify these adjustments. The quantification of dislocation features (Figure V.5a and Figure V.6a) is however unnecessary to obtain values of the adjusting factor. In the following, a simple way is presented to determine these adjustments directly from the material deformation response during the first few loading cycles at lower strain amplitudes (±0.25 and 0.4%). This way of parameter identification quickly determines model parameters for different loading condition which is useful when applying this proposed model for other problems.

A higher imposed plastic strain induces higher plastic strain incompatibilities between grains. Boundary dislocations are therefore expected to occupy a smaller area fraction for a lower imposed plastic strain amplitude. During plastic deformation in the first cycle, the intra-granular back stress is negligible compared to the inter-granular back stress (Figure VI.8). The area fraction of boundary dislocations \( f_b \) is adjusted by the relative ratio of the back stress amplitude of the first quarter cycle 

\[
\frac{X^{(0.4\% \text{ or } 0.25\% \text{ rel.}}}{X^{(0.7\%)}}
\]

The higher grain-to-grain plastic strain incompatibilities also requires higher \( \sqrt{\rho_{\text{hom}}} \) during the cyclic hardening response stage. In addition, the activity of multiple slip is also stronger, resulting in more frequent interactions of dislocations and a higher density of interior dislocations for higher strain amplitudes (Figure V.5a). Consequently, a well-organised wall/channel structure is developed more rapidly for higher strain amplitudes (Figure V.6a). Consequently, both \( \sqrt{\rho_{\text{hom}}} \) and \( \Delta \sqrt{\rho_{\text{w-c}}} \) reduce in proportion to the plastic strain amplitude. By the end of cyclic hardening, (1) the boundary dislocation density almost achieves its saturation level (Figure VI.6), and (2) dislocation-high/low-density regions start to progressively form (Figure IV.4c). In addition, it is assumed that both \( \sqrt{\rho_{\text{hom}}} \) and \( \Delta \sqrt{\rho_{\text{w-c}}} \) reduce in proportion with the strain amplitude. These two parameters can therefore be modified.
by $X^{(\text{peak})}_{\pm0.4\%, \pm0.25\%} / X^{(\text{peak})}_{\pm0.7\%}$ (Table VI.3). The effectiveness of these microstructurally based adjustments is demonstrated for strain amplitudes of $\pm0.4$ and $0.25\%$, respectively in Figure VI.10 and Figure VI.11, which both show that the proposed model well predicts the long-term cyclic deformation response of the material. The predictive capability of the model is slightly less effective for the first cycles at a strain amplitude of $\pm0.25\%$ (Figure VI.10a, b) due to the fact that dislocations move in a more planar manner in these conditions (Sect. IV.3.2 and V.4). The prediction nevertheless becomes much better after an increasing number of cycles (Figure VI.11d-f).

Table VI.3: Adjusting factors for conditions of $\pm0.4\%$, and $0.25\%$ account for the amplitude dependence of internal stresses.

<table>
<thead>
<tr>
<th></th>
<th>$X^{(1)}_{\pm0.4%, \pm0.25%}$</th>
<th>$X^{(\text{peak})}_{\pm0.4%, \pm0.25%}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Values</td>
<td>0.78, 0.54</td>
<td>0.73, 0.41</td>
</tr>
</tbody>
</table>
Figure VI.10: The prediction of material cyclic deformation response at ±0.4% - 10^{-3}s^{-1} - 20°C by the proposed model. Comparison of experimental vs. modelled data of: (a) peak stresses, and (b-f) the hysteresis loops of selected cycles (at different stages of cyclic response).

Figure VI.11: The prediction of material cyclic deformation response at ±0.25% - 10^{-3}s^{-1} - 20°C. Comparison of experimental vs. Modelled data of: (a) peak stresses, and (b-f) the hysteresis loops of selected cycles (at different stages of cyclic response).
The concept of the model formulation proposed here on the basis of the relationships between internal stresses and microstructural condition might be used to describe the cyclic deformation response of AISI 316L under much different boundary loading conditions for which the interaction of dislocations involves more microstructural features, e.g. point and volume defects. For instance, at elevated temperatures in the regime of dynamic strain aging, the strong short-range interaction of dislocations with solute atoms (e.g. C and N) results in a strong influence of the effective stress on the material response, leading to more marked primary cyclic hardening and the incidence of the secondary cyclic hardening [134, 182]. The modelling of the cyclic deformation response of the material at 300°C (i.e. 573K) is discussed in the following section.
VI.6. Modelling the cyclic deformation response at 300°C (573K)

VI.6.1. Thermally activated constitutive equation

In this section, the proposed constitutive model is extended to describe the kinetic process of discrete-obstacle controlled plastic deformation in order to represent the temperature dependence of the evolution of the internal stresses.

A density $\rho_{i}^{mo}$ of mobile dislocations of dislocation type $i$ (e.g. boundary, interior, wall and channel dislocations) moves through discrete obstacles with an average velocity $\bar{v}$, resulting in a strain rate of [183].

$$\dot{\gamma} = \rho_{i}^{mo} b \bar{v} \quad \text{(VI.23)}$$

The average velocity $\bar{v}$ of a density $\rho_{i}^{mo}$ of mobile dislocations with the thermal activation:

$$\bar{v} = \beta b \nu \exp(-\frac{\Delta G(\tau_i)}{kT}) \quad \text{(VI.24)}$$

Where $\beta$ is a dimensionless constant, $b$ is the magnitude of Burgers vector, and $\nu$ is frequency.

$\Delta G(\tau_i)$ is the Gibbs free energy of activation for the cutting or by-passing of an obstacle. For box-shaped and regular-spaced obstacles:

$$\Delta G(\tau_i) = \Delta F \left( 1 - \frac{\tau_i}{\tau} \right) \quad \text{(VI.25)}$$

Where: $\Delta F$ is the total free energy (the activation energy) required to overcome the obstacles without the aid from external stress.
\( \hat{\tau} \) is the stress needed to move dislocations through obstacles without the assistance of thermal energy. This stress equals the yield strength of materials at 0 K.

\( \tau_i \) is the stress associated with dislocations of type \( i \).

For irregular-shaped and random distributed obstacles [184],

\[
\Delta G(\tau_i) = \Delta F \left( 1 - \left( \frac{\tau_i}{\tau} \right)^m \right)^n
\]

(VI.26)

Where \( m, n \) describe the shape of obstacles, with

\[
0 \leq m \leq 1 \\
1 \leq n \leq 2
\]

The activation energy to let dislocations pass obstacles depends on the strength of the obstacles. For strong obstacles, e.g. large or strong precipitates, \( \Delta F \) is large, \( m \) and \( n \) are insignificant, and both can be chosen to be equal to 1 [1].

**Table VI.4: Characteristics of obstacles, thermal activation and athermal strength [1].**

<table>
<thead>
<tr>
<th>Obstacle strength</th>
<th>( \Delta F )</th>
<th>( \hat{\tau} = \eta \frac{\mu b}{L_i} )</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong</td>
<td>( 2\mu b^3 )</td>
<td>( \eta &gt; 1 )</td>
<td>Dispersions, large and strong precipitates</td>
</tr>
<tr>
<td>Medium</td>
<td>( 0.2 - 1\mu b^3 )</td>
<td>( \eta \approx 1 )</td>
<td>Forest dislocations, small and weak precipitates</td>
</tr>
<tr>
<td>Weak</td>
<td>( &lt; 0.2\mu b^3 )</td>
<td>( \eta \ll 1 )</td>
<td>Lattice resistance, solution strengthening</td>
</tr>
</tbody>
</table>

Note: \( L_i \) is the mean free path of type \( i \) of dislocation, \( (L_i = \sqrt{\rho_i}) \)
The discrete-obstacle controlled strain rate of the density $\rho_{i}^{mo}$ of moving mobile dislocations is then

\[ \dot{\gamma} = \rho_{i}^{mo} b v \]

\[ = \left( \frac{\tau_{i}^{mo}}{\alpha G b} \right)^2 b \beta b v \exp \left[ -\frac{\Delta F}{kT} \left( 1 - \left( \frac{\tau_{i}}{\tau} \right)^m \right)^n \right] \]

\[ = \dot{\gamma}_0 \exp \left[ -\frac{\Delta F}{kT} \left( 1 - \left( \frac{\tau_{i}}{\tau} \right)^m \right)^n \right] \]

(VI.27)

Where $\tau_{i}^{mo}$ is the necessary stress to move mobile dislocations in obstacle-free regions.

$\dot{\gamma}_0$ is called the reference strain rate which is about $10^6$ s$^{-1}$ [1].

Eqn. VI.27 then becomes

\[ \tau_i = \left\{ 1 - \left[ -\frac{kT}{\Delta F} \ln \left( \frac{\dot{\gamma}}{\dot{\gamma}_0} \right) \right]^{1/n} \right\}^{1/m} \hat{\tau} \]

\[ = \left\{ 1 - \left[ -\frac{kT}{\Delta F} \ln \left( \frac{\dot{\gamma}}{\dot{\gamma}_0} \right) \right]^{1/n} \right\}^{1/m} \eta \frac{\mu b}{L_i} \]

(VI. 28)

Where $\eta$ is pre-factor of $\hat{\tau}$ which relates to the strength of obstacles (Table VI.4)
The dynamic strain ageing takes place when solute atoms diffuse into dislocation cores to form atmospheres of solute atoms around dislocation lines, which restrict the movement of dislocations, leading to a higher number of dislocations. The locking effectiveness of atmospheres of type \( s \) solute atoms relates to the concentration \( (C_s) \) of type \( s \) solute atoms on dislocation cores \((s \) is either carbon or nitrogen). To take the presence of solute atom atmospheres into account, the internal stress \( \tau_i \) in Eqn. VI.28 becomes [185]

\[
\tau_i = \left( 1 - \frac{kT}{\Delta F} \frac{C_s}{\sqrt{C_{s,0}}} \ln \left( \frac{\gamma}{\gamma_0} \right) \right)^{1/n} \eta \mu b \sqrt{\frac{C_s}{C_{s,0}}} \rho_i \quad \text{(VI.29)}
\]

Where \( C_{s,0} \) and \( C_s \) are the nominal concentration and the saturation concentration of solute atom \( s \) along dislocation lines.

For polycrystalline materials,

\[
\sigma_i = \left( 1 - \frac{kT}{\Delta F} \frac{C_s}{\sqrt{C_{s,0}}} \ln \left( \frac{\gamma}{\gamma_0} \right) \right)^{1/n} \eta \mu bM \sqrt{\frac{C_s}{C_{s,0}}} \rho_i \quad \text{(VI.30)}
\]

While the short range interaction of dislocations is strongly dependent on the thermal assistance, the thermal activation has much less influence on long-range stress fields. The temperature dependence of the long-range interaction of dislocations mainly results from the temperature dependence of elastic modulus [184]. The term
\[
1 - \left( \frac{kT}{\Delta F} \ln \left( \frac{\gamma}{\gamma_0} \right) \right)_{\text{sat}} \quad \eta \text{ is approximately close to 1 for back stresses.}
\]

Consequently, the terms \( \frac{C_{\text{sat}}}{g_{\text{sat}}}, \frac{C_{\text{intra}}}{g_{\text{intra}}} \) at 300°C (573K) can be directly obtained from an equivalent condition at 20°C (293K) (in Eqn. VI.20) by the following relationship:

\[
\begin{align*}
\frac{C_{\text{sat}}}{g_{\text{sat}}} \bigg|_{573K} &= \frac{C_{\text{sat}}}{g_{\text{sat}}} \bigg|_{293K} = \frac{\mu}{\mu_{1573}} = 0.8 \\
\frac{C_{\text{intra}}}{g_{\text{intra}}} \bigg|_{573K} &= \frac{C_{\text{intra}}}{g_{\text{intra}}} \bigg|_{293K}
\end{align*}
\]

The evolution of back stresses is effectively represented by Eqns. VI.8 and VI.11 with

\[
\begin{align*}
\rho_{\text{hom}}^{\text{inter}} \bigg|_{573K} &= \left( \frac{C_s}{C_s,0} \right) \rho_{\text{hom}}^{\text{inter}} \bigg|_{293K} \\
\rho_{\text{hom}}^{\text{intra}} \bigg|_{573K} &= \left( \frac{C_s}{C_s,0} \right) \rho_{\text{hom}}^{\text{intra}} \bigg|_{293K} \\
\rho_{\text{GND-inter}}^{\text{sat}} \bigg|_{573K} &= \left( \frac{C_s}{C_s,0} \right) \rho_{\text{GND-inter}}^{\text{sat}} \bigg|_{293K} \\
\rho_{\text{w}}^{\text{sat}} \bigg|_{573K} &= \left( \frac{C_s}{C_s,0} \right) \rho_{\text{w}}^{\text{sat}} \bigg|_{293K} \\
\rho_{\text{c}}^{\text{sat}} \bigg|_{573K} &= \left( \frac{C_s}{C_s,0} \right) \rho_{\text{c}}^{\text{sat}} \bigg|_{293K}
\end{align*}
\]
As referred to above, effective stress is greatly dependent on the thermal activation. The thermally-activated constitutive model equation for effective stress is obtained by combining Eqn. VI.17 with Eqn. VI.29,

\[
2\sigma_E = 2\sigma_E^0 + \left[1 - \frac{kT}{\Delta F C_s/C_{s,0}} \ln \left( \frac{\gamma}{\gamma_0} \right) \right]^{1/m} \eta \mu b M \left( \frac{C_s}{C_{s,0}} f_j \rho_l \right) + 9.56 \left( \log p - \log p_c \right)
\]

VI.6.2. Modelling of Cyclic Deformation Response for ±0.7%·10^{-3}s^{-1}-573K

In the non-DSA regime, with thermal assistance, the number of generated dislocations at higher temperatures is normally smaller than for the equivalent conditions at lower temperatures. The temperature dependence of dislocation densities is in proportion to that dependence of elastic modulus. However, due to the strong interaction of dislocations with solute atoms, dislocation densities at an elevated temperature in the DSA-regime are higher than the corresponding densities at lower temperatures (Figure V.4, Figure V.5, and Figure V.6). Consequently, the ratio of dislocation density at an elevated temperature in the DSA-regime is proportional to the relative ratio of dislocation density at the elevated temperature to that at a reference temperature in the non-DSA regime (e.g. 293K). The relative ratio of interior dislocation density at 573K (300°C) to that at 293K (20°C) was measured to be about 1.5-2 times (Figure V.4). To compensate for the thermal activation, the value of \( \frac{C_s}{C_{s,0}} \) should be,

\[
\frac{C_s}{C_{s,0}} = \frac{\rho_l|_{573K}}{\rho_l|_{293K}} \times \frac{\mu(293)}{\mu(573)} = (1.9 - 2.5)
\]
The values of $m$, $n$, $\Delta F$ and $\eta$ are identified from solving Eqn. VI.31 to obtain the measured values of $\sigma_E$ at the 1500th cycle for ±0.7%-10^{-3}s^{-1} at 293, and 573K (Table VI.5), where $f_j\rho_i = 0.6 \times 10^{14} l/m^2$ (Table VI.2).

Table VI.5: The values of $\sigma_E|_{1500}$ measured at the 1500th cycle, at 293 and 573 K

| $\varepsilon$ | T    | $\sigma_E|_{1500}$ | $f_j\rho_i$ |
|--------------|------|-------------------|-------------|
| s$^{-1}$     | K    | MPA               | 1/m$^2$     |
| $10^{-3}$    | 293  | 118               | 0.6x10$^{14}$|
| $10^{-3}$    | 573  | 87                |             |

The values of $m$, $n$, $\Delta F$, $C_i/C_{i,0}$, and $\eta$ (Table VI.6) give a good description of the temperature dependence of effective stress. With these values, the modelled values of $\sigma_E$ at the 1500th cycle at 293 and 573K are about 119 and 90MPA, respectively. The identified values of $m$, $n$, $\Delta F$ and $\eta$ in this study are very close to values obtained by Lindgren et al. in their study to model rather well the evolution of flow stress during the monotonic loading of AISI 316L over a range of temperatures from 20 to 400°C [186], which indicates that these identified values can model the temperature dependence of effective stress for this range of temperature, where the interactions between dislocations and solute atom atmospheres of C and N are responsible for DSA.
Table VI.6: The parameter identification of Eqn. VI.31

<table>
<thead>
<tr>
<th>( m )</th>
<th>( n )</th>
<th>( \Delta F )</th>
<th>( C_s / C_{s,0} )</th>
<th>( \eta )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.37</td>
<td>1.37</td>
<td>( \approx 0.74 \mu b^3 )</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

To model the evolution of internal stresses (Eqn. VI.18), it is necessary to identify the values of \( k_{2-b} \), \( k_{2-j} \approx k_{2-w} \) and \( k_{2-j} \). Because the strong interaction between solute atoms and dislocations causes the rate of cross slip activity at 573K to be half that at 293K (Figure III.11, Sect. IV.4.6), \( k_{2-b}^{573} = 0.5k_{2-b}^{293} \). The terms \( k_{2-j} \approx k_{2-w} \) and \( k_{2-j} \) are then identified by fitting the evolution of stress range versus the accumulated plastic strain by (Eqn. VI.18) with:

1) The limits of \( \rho_b^{\text{hom}}, \rho_i^{\text{hom}}, \rho_{w-c}^{\text{sat}}, \rho_{\text{GNDs-inter}}^{\text{sat}}, \) and \( \rho_j^{\text{sat}} \) (which were given in Table VI.2) being adjusted by \( C_s / C_{s,0} \), and

2) \( f_b, f_w^{\text{sat}}, \) and \( f_j \) being equal to those obtained from the parameter identification for \( \pm 0.7\% - 10^{-3} \text{s}^{-1} - 20^\circ \text{C} \) (293K) (Table VI.2).

The values of parameters in (Eqn. VI.18) for \( \pm 0.7\% - 10^{-3} \text{s}^{-1} - 573 \text{K} \) by this fitting procedure are shown in Table VI.7.
Table VI.7: Parameter identification of (Eqn. VI.18) for ±0.7%-10^{-3}s^{-1}-573K

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$\rho_{b_{_{\text{hom}}}}^{\text{hom}}$ (x10^{14})</th>
<th>$k_{2-b}$</th>
<th>$f_b$ (x10^{14})</th>
<th>$\rho_{I_{_{\text{hom}}}}^{\text{hom}}$</th>
<th>$k_{2-I}$ (x10^{14})</th>
<th>$\rho_{\text{inter-sat-GNDs}}^{\text{sat}}$ (x10^{14})</th>
<th>$\Delta \rho_{\text{inter-sat-GNDs}}^{\text{sat}}$ (x10^{14})</th>
<th>$f_{w_{\text{sat}}}^{\text{sat}}$</th>
<th>$f_j$ (x10^{14})</th>
<th>$k_{2-j}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unit</td>
<td>1/m^2</td>
<td>1/m^2</td>
<td>1/m^2</td>
<td>1/m</td>
<td>1/m^2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower</td>
<td>2</td>
<td>0.03</td>
<td>0.1</td>
<td>1.6</td>
<td>0.001</td>
<td>1.7</td>
<td>0.1</td>
<td>0.5</td>
<td>0.004</td>
<td></td>
</tr>
<tr>
<td>Upper</td>
<td>6</td>
<td>0.04</td>
<td>0.2</td>
<td>5</td>
<td>0.0025</td>
<td>3.4</td>
<td>0.28</td>
<td>1</td>
<td>0.08</td>
<td></td>
</tr>
<tr>
<td>Identified</td>
<td>3.7</td>
<td>0.036</td>
<td>0.2</td>
<td>3.2</td>
<td>0.001</td>
<td>1.82</td>
<td>0.1</td>
<td>0.5</td>
<td>1.6</td>
<td>0.065</td>
</tr>
</tbody>
</table>

The comparison between model and experimental data shows that the model is also very capable of describing the cyclic stress-strain deformation response of the material at 573K (or 300°C) (Figure VI.12). An underestimation of the deformation response during reverse loading transients from tensile peak stress is due to: 1) the parameters being identical for both reverse loading transients from compressive and tensile peak stresses, 2) the interactions between dislocations and solute atoms/corduroy structure being hydrostatic-stress dependent.
VI.6.3. Modelling of Cyclic Deformation Response for ±0.4%, and ±0.25% (10^{-3} \text{s}^{-1}-573K)

Following the same logic as described in Sect. VI.5.2.2, adjusting factors are required to take account of the strain amplitude dependence of microstructural evolution characteristics at 573K (Table VI.8). The effectiveness of the model with these microstructurally-based adjustments is demonstrated for strain amplitudes of ±0.4 and 0.25% at 573K, respectively in Figure VI.13 and Figure VI.14, which both show that the proposed model well predicts the long-term cyclic deformation response of the material. The prediction of the model slightly overestimates secondary cyclic hardening at a strain amplitude of ±0.4% (Figure VI.13a) due to the interference of the presence of fatigue cracks in the macroscopic manifestation of the change in the effective stress (referred to Sect. VI.3.3.2).
Table VI.8: Adjusting factors for conditions of ±0.4%, and 0.25% at 573K account for the amplitude dependence of internal stresses.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Adjusting Factor</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>±0.4%, ±0.25%</td>
<td>X^{(1)}</td>
<td>0.78, 0.75</td>
</tr>
<tr>
<td>±0.7%</td>
<td>X^{(1)}</td>
<td>0.59, 0.47</td>
</tr>
</tbody>
</table>

Figure VI.13: The prediction of material cyclic deformation response at ±0.4% - $10^{-3}$s$^{-1}$ – 573K by the proposed model. Comparison of experimental vs. modelled data of: (a) peak stresses, and (b-f) the hysteresis loops of selected cycles (at different stages of cyclic response).
Figure VI.14: The prediction of material cyclic deformation response at ±0.25% - $10^{-3}$s$^{-1}$ – 573K by the proposed model. Comparison of experimental vs. modelled data of: (a) peak stresses, and (b-f) the hysteresis loops of selected cycles (at different stages of cyclic response).

The effectiveness of the model predictive capability for lower strain amplitudes is slightly lower due to the more planar movement of dislocations, as explained in Sect. VI.5.2.2. In particular, the planarity of dislocation movement is enhanced at 573K (300°C) due to the strong interaction between dislocations and point defects (Sect. IV.4.6).
VI.7. Model benchmarking

In this section the effectiveness of the proposed model is benchmarked against the cyclic deformation response of AISI 316L for more independent cyclic loading conditions (e.g. variable strain amplitude fatigue loading). As shown in Sect. VI.5 and Sect. VI.6, the proposed evolutionary constitutive model successfully predicts the cyclic deformation response of the materials for lower strain amplitude conditions by introducing three adjusting factors. The objective of this section is to examine how effective the model is without adjusting factors for conditions where: the microstructural condition is first introduced to be quite similar to the identified condition (the ±0.7%-condition), but the strain amplitude is subsequently varied.

VI.7.1. Variable amplitude fatigue loading at 20 and 300°C

Cylindrical testpieces (as described in Sect. III.1) were fatigue loaded with i) constant strain rate of $10^{-3}\text{s}^{-1}$, ii) at the temperatures of 20 and 300°C, and iii) with total strain amplitude varying during cyclic loading. The testpieces were first cyclically loaded for the first 100 cycles with a total strain amplitude of ±0.8%. Afterwards, the total strain amplitude decreases by ±0.2% (Figure VI.15b). For the first 400 cycles, the decremental step of total strain amplitude after every 100 cycle was ±0.2% until the total strain amplitude of ±0.2% was reached (Figure VI.15c, d). The total strain amplitude was then increased again by ±0.2% from ±0.2% to ±0.6% (Figure VI.15e, f). The testpieces were finally fatigued at the total strain amplitude of ±0.6% until failure (Figure VI.16). The evolution of peak stresses during variable strain amplitude cyclic loading at 20 and 300°C is shown in Figure VI.16.

After the first 100 cycles with the total strain amplitude of ±0.8%, quite well-organised dislocation wall/channel structure is very likely already formed since the total dislocation density generated for this condition is higher than that for the ±0.7% condition. The microstructural condition after the next 100 cycles with the total strain amplitude of ±0.6% would be very similar to the microstructure after 300 cycles of constant strain amplitude cyclic loading of ±0.7%. During subsequent various strain
amplitude loading cycles, the degree of organisation of dislocation structures could increase, but there would be no significant change in the dislocation structure dimensions. The model prediction is made in the next section to see if the proposed model predicts well the cyclic deformation response during variable strain amplitude cyclic loading without any adjustments.

Figure VI.15: (a) Strain-controlled scheme during a step of varying strain amplitude. (b)-(f) Stress-strain hysteresis loops during varying strain amplitude.

Figure VI.16: Maximum stress evolution during variable amplitude cyclic loading at 20 and 300°C
VI.7.2. Model prediction

The model predictions of the cyclic deformation response during variable amplitude cyclic loading at 20 and 300°C were made with the model parameters identified for the constant amplitude of ±0.7% at these temperatures without the adjusting factors, respectively. The comparisons of prediction with experimental data show that the model prediction of the material deformation response during these independent benchmark test conditions is excellent (Figure VI.17 and Figure VI.18). At 300°C, the prediction of the model is slightly less effective which could be due to the more complex evolution of effective stress relating to the interactions between dislocations and point defects.

Figure VI.17: Prediction of the cyclic deformation response during variable amplitude cyclic loading at 20°C (293K) (Values of model parameters are identified from the ±0.7% - 10^{-3}s^{-1} - 20°C test condition).
Figure VI.18: Prediction of the cyclic deformation response during variable amplitude cyclic loading at 300°C (573K) (Values of model parameters are identified from the ±0.7% - 10^{-3}\text{s}^{-1} - 300°C test condition).
VI.8. Conclusions

An evolutionary physically-based constitutive model of internal variables has been successfully developed based on the gained knowledge of the relationships between microstructural condition and internal stresses during cyclic loading (i.e. Eqns VI.8, VI.11 and VI.17). In addition, the proposed model also incorporates the temperature dependence of the relationships between microstructural evolution and internal stresses (Eqn. VI.30). The model parameters are identified by systematic evaluations of mechanical and microstructural observations from a number of interrupted fatigue tests for a single test condition of ±0.7%-10^{-3}s^{-1} at 20°C (293K). The quantification of dislocation evolution for this test condition and for the equivalent condition at 300°C (573K) provides the information to determine the model parameters at 300°C. The proposed model excellently describes the complex cyclic elasto-plastic deformation behaviour of AISI 316L at 20 and 300°C.

On account of the changing characteristics of microstructural evolution and its consequence for the change in internal stresses, $f_b$, $\sqrt{P_b^{\text{min}}}$ and $\Delta \sqrt{P_{\text{wc}}^{\text{int}}}$ need to be adjusted to accurately predict the long-term cyclic deformation response for lower strain amplitudes at a given temperature. These adjustments are all determined directly from the deformation response of the material during the first few loading cycles at lower strain amplitudes (±0.25 and 0.4%).

The capability of the model is then benchmarked by predicting the material cyclic response for independent testing conditions. The model benchmarking shows that the proposed model is very capable of predicting the cyclic elasto-plastic deformation behaviour during variable amplitude fatigue loading.
Chapter VII: Fatigue crack propagation

At 300°C, when dynamic strain ageing takes place, the fatigue endurance of AISI 316L for lower strain amplitudes is lower than under equivalent conditions at 20°C. The changes in: (1) apparent elastic modulus, (2) microstructural condition, and (3) fractographic features have been exhaustively performed to reveal the reason for the life reduction. The analysis of apparent elastic modulus variations and the results of fractographic observations show that the propagation rate of fatigue cracks for conditions at 20°C are faster than under equivalent conditions at 300°C. Crack initiation however occurs earlier at 300°C, in particular for lower strain amplitude tests, due to the activity of localised deformation bands as a consequence of cyclic loading. In addition to ladder-like persistent slip bands, a form of ladder-free deformation bands is also present at 300°C after a high number of cycles, in particular at low strain amplitudes. When the fatigue life is rather short, the influence of the ladder-free deformation bands on cyclic endurance is negligible. The ladder-free type of localised bands have a strong influence on crack initiation once the material endurance increases with lowering strain amplitude, leading to the relative life reduction at the elevated temperature. In addition, the incidence of secondary cyclic hardening for lower strain amplitude tests at 300°C partly contributes to the more evident life reduction. The influence of dislocation walls on the propagation of microstructurally short fatigue cracks is also examined in this Chapter.
VII.1. Introduction

The progressive fatigue failure of smooth specimens develops as a consequence of cyclic deformation under alternating loading condition. The movement and the rearrangement of dislocations lead to localised plastic deformation bands (in particular persistent slip bands (PSBs)) which cause the formation of extrusions at the free surface. Extrusions act as stress-raisers, resulting in the initiation and propagation of microscopic short fatigue cracks [6, 11-13, 90]. Microscopic cracks then develop into long cracks. After a period of stable propagation, the crack size exceeds the critical value and overload or fast unstable fracture occurs (Figure VII.1). Depending on the loading conditions, the different crack development stages can determine the fatigue life to different extents. The surface quality of critical locations in components for many modern dynamic applications is very high. In these circumstances, the initiation and the propagation of microscopic fatigue cracks can occupy a very large fraction of fatigue life [4, 187]. Microscopic fatigue cracks can be sensitive or insensitive to the microstructural condition depending on their dimension and loading condition. Microstructurally short cracks whose length is about 1 to several grain sizes are found to be influenced by the presence of local microstructural features, in particular grain boundaries, [188-190]. The propagation of microstructurally short cracks is thought to be discontinuous. Such fatigue cracks trangranularly propagate and then arrest at a grain boundary. When the critical crack tip opening displacement is sufficiently increased due to local cyclic plasticity, fatigue cracks can overcome the boundary and propagate to the next one. In order to improve the effectiveness of fatigue lifetime assessment of components in service, it is necessary to understand the relationship between microstructural condition and fatigue damage, and in particular the initiation and propagation mechanisms relating to microstructurally short fatigue cracks.
AISI 316L is susceptible to dynamic strain ageing in the temperature range of 200 to 500°C, and with strain rates of $10^{-4}$ to $10^{-2}$s$^{-1}$. At 300°C, dynamic strain ageing is associated with the interaction of solute atoms (C and N) and mobile dislocations \[122\].

The occurrence of DSA contributes to the complex cyclic deformation response (\textbf{Sect. III.V} and \textbf{Sect. IV.4}) \[134\], and a significant reduction in fatigue life \[125, 191-193\]. The effects of creep and oxidation on the fatigue life of this material at intermediate temperatures (in particular at 300°C) are found to be negligible \[194\]. The reduction in fatigue life in this temperature regime, relative to that at ambient temperature, is often believed to be due to: (1) an enhanced degree of plastic deformation inhomogeneity which promotes the formation of fatigue cracks, and (2) pronounced cyclic hardening which can cause rapid crack propagation \[125, 193\]. Existing results for the cyclic deformation response of AISI 316L (including those of the present study) shows that although DSA can cause a higher rate of cyclic hardening, the value of peak stress due to symmetrical cyclic loading is usually lower (i.e. the stress range is smaller) when the temperature increases over the DSA temperature range (i.e. 250-500°C). The propagation rate of intermediate and long fatigue crack is known to be proportional to the stress range \[4, 26\]. The evidence indicates that long fatigue cracks would grow with a lower rate in this DSA temperature range than that at lower temperatures which

Figure VII.1: Fatigue crack development during cyclic loading of metallic materials.
are out of the DSA regime. The logic to explain the reduction of fatigue life under the DSA regime is still a matter of debate, and needs to be carefully re-examined.

In this chapter, the crack initiation endurance and the propagation rate of fatigue cracks (both short and long fatigue cracks) are quantitatively characterised by analysing the changes in apparent elastic modulus and fractographic investigation. Special attention is given to reveal the role of microstructural condition on crack initiation. The activity of localised deformation bands during cyclic loading at both 20 and 300°C is carefully examined. This examination together with fractographic investigation shed light on the consequence of the activity of localised deformation bands for the initiation of fatigue cracks. Moreover, the propagation of short fatigue cracks are comprehensively characterised based on the quantification of fatigue striation spacing and the observation of the interaction between microstructurally short fatigue cracks and dislocation walls.
VII.2. Experimental procedure

The material composition, the description of strain-controlled fatigue testing and the experimental methods to perform microstructural observations were respectively given in Table I.1, Sect. III.1 and Sect. IV.1. The measurement of rates of change for the different cyclic deformation response stages was introduced in Sect. III.3.7.

The number of cycles to 2% stress drop for every test condition at a strain rate of $10^{-3}$s$^{-1}$ (Table III.1) was identified as shown in Figure VII.2a. When macro fatigue cracks existed, at the beginning of during reverse loading transients from compressive peak stress, they remained closed due to the compressive stress state; whereas, they could open during reverse loading transients from tensile peak stress (Figure VII.2b). For this reason, the apparent elastic modulus values during reverse loading transients from compressive peak stress were always higher than those during reverse loading transients from tensile peak stress, when macro cracking was present in the gauge section. Analysis of the difference between apparent elastic moduli in during reverse loading transients from compressive and tensile peak stresses ($\Delta E_c-t$) was therefore made in order to provide information concerning the extent of long-fatigue crack advance during cyclic loading.

To investigate the propagation of short fatigue cracks, fracture surfaces were examined by Scanning electron microscopy (SEM). The SEM observation was first performed in low-resolution mode to follow the main direction of crack propagation in order to trace back to the crack initiation region of the predominant crack. After finding the region of crack initiation, areas close to this region were carefully investigated to find locations where fatigue striations were firstly noticeable by high resolution SEM ($\times$30,000-120,000 times). Afterwards, images in the direction of crack propagation after every 50-100$\mu$m (the exact value of step being dependent on the condition of the fracture surface) were recorded to provide the information of the development of fatigue striations, i.e. the propagation rate of fatigue cracks. The crack length was measured as the distance from the crack initiation point to the recorded location along the crack
path. The fatigue striation spacing was then measured in the direction of crack propagation, which was perpendicular to the curve of fatigue striations (Figure VII.2c).

Figure VII.2: (a) Representation of the fitting procedure to measure the fatigue life corresponding to 2% of maximum stress drop. (b) Crack opening and closing during a cycle causes different values of apparent elastic moduli during reverse loading from compressive and tensile peak stresses. (c) The measurement of fatigue striation spacing is made in the direction of crack propagation.

In addition, electron channelling contrast (ECC) imaging technique was performed by SEM to study the interaction between short fatigue cracks and dislocation walls. To provide samples for performing ECC imaging, sections of gauge length were cut in parallel to the loading axis using a diamond cutting machine. After mechanical polishing by carbide particle papers, they were further polished by suspension containing fine particles of different sizes (6, 3 and 1µm). These specimens were finally polished by colloidal silica solution consisting of 0.05 µm particles mixed with 7 vol % of H₂O₂. In order to obtain good electron channeling contrast, SEM observation were performed at the condition of high current mode, the working distance of about 10mm, and a low convergence angle.
VII.3. Results

VII.3.1. Fatigue life

Fatigue lives (macro-crack initiation endurances) corresponding to 2% of maximum stress drop for all tests are listed in Table VII.1. For a strain amplitude of ±0.7%, the fatigue life at 300°C is marginally higher than that at 20°C. The reduction of fatigue life for tests at 300°C becomes more significant for lower strain amplitude conditions which is in agreement with another study [194]. The fatigue life for a strain amplitude of ±0.25% at 20°C is almost double that at 300°C.

Table VII.1: The rates of cyclic hardening and softening and the 2% of stress drop crack initiation endurances

<table>
<thead>
<tr>
<th>Condition</th>
<th>Rate of cyclic hardening (MPa / Cycle)</th>
<th>Rate of cyclic softening (MPa / Cycle)</th>
<th>Rate of secondary cyclic hardening (MPa / Cycle)</th>
<th>N&lt;sub&gt;2%&lt;/sub&gt; (Cycles)</th>
</tr>
</thead>
<tbody>
<tr>
<td>±0.7% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 20°C</td>
<td>60.1</td>
<td>-20.4</td>
<td>-</td>
<td>3900</td>
</tr>
<tr>
<td>±0.7% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 300°C</td>
<td>107.6</td>
<td>-40.4</td>
<td>-</td>
<td>5400</td>
</tr>
<tr>
<td>±0.4% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 20°C</td>
<td>31.3</td>
<td>-19.9</td>
<td>-</td>
<td>17950</td>
</tr>
<tr>
<td>±0.4% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 300°C</td>
<td>51.4</td>
<td>-33.2</td>
<td>5.8</td>
<td>14850</td>
</tr>
<tr>
<td>±0.25% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 20°C</td>
<td>9.5</td>
<td>-12.0</td>
<td>7.9 - 16.3</td>
<td>80600</td>
</tr>
<tr>
<td>±0.25% - 10&lt;sup&gt;-3&lt;/sup&gt;s&lt;sup&gt;-1&lt;/sup&gt; - 300°C</td>
<td>22.5</td>
<td>-8.4</td>
<td>7.9 - 16.3</td>
<td>44600</td>
</tr>
</tbody>
</table>

VII.3.2. Analysis of the evolution of apparent elastic modulus

For a given testing condition, the apparent elastic moduli of reverse loading transients from compressive and tensile peak stresses are almost identical during the first half of the fatigue life. The absolute difference in elastic moduli of reverse loading transients
from compressive and tensile peak stresses \( (\Delta E_{c-t}) \) is almost constant and very small during the first 1,500 cycles for the ±0.7%-20°C test (Figure VII. 3a and b). After a high number of cycles, the difference however starts increasing in a prominent way (e.g. after 2,000 cycles for the ±0.7-20°C condition (Figure VII. 3a and b)). A similar tendency for the evolution of the E difference is also observed in lower strain amplitude tests. For the same temperature, a significant divergence of E moduli starts much later for lower strain amplitude tests (Figure VII. 3c and d).

Figure VII. 3: (a) The evolution of apparent elastic moduli during reverse loading transients from compressive and tensile peak stresses until the number of cycles corresponding to 2% of maximum stress drop for the ±0.7%-10^{-3}s^{-1}-20°C condition. The evolution of the E difference after a high number of cycles for: (b) the ±0.7%-10^{-3}s^{-1}-20°C condition (that for the ±0.7%-10^{-3}s^{-1}-300°C condition is not shown in this case to avoid the data superposition), (c) the ±0.4% condition at 20 and 300°C, and (d) the ±0.25% condition at 20 and 300°C.
The difference between 20 and 300°C fatigue lives is negligible for the ±0.7% condition, and becomes more significant for the lower strain amplitude conditions. The analysis of the E difference is therefore focused on the ±0.4 and ±0.25% conditions to study the influence of temperature on fatigue life. Curves representing the evolution of $\Delta E_{c-t}$ for these conditions are given in Figure VII. 3c, d. These curves were fitted by a $(10^{(aN+b)})$ function, where the coefficient $a$ represents the rate of change of $\Delta E_{c-t}$ per unit cycle in the $(\log(\Delta E_{c-t}), N)$ scale. The value of $a$ is shown in Figure VII. 3c, d to quantify how fast long-fatigue cracks propagate. The higher the value of $a$, the faster the long-fatigue cracks advance. Figure VII. 3c and d interestingly show that: (1) long fatigue cracks propagate with a lower rate at 300°C, (2) $a$ is almost identical for the same temperature irrespective of the strain amplitude. From these observations, it is concluded that, for a given strain amplitude: (1) long fatigue crack propagation rates depend on the temperature, and (2) the reduction in fatigue life at the higher temperature is unlikely to be due to the propagation rate of long fatigue cracks.

While $\Delta E_{c-t}$ clearly starts to increase earlier at 300°C than at 20°C for lower strain amplitude tests (Figure VII. 3c, d), the evidence indicates that the same tendency could be true at 300°C for the ±0.7% test (Figure VII. 3a). This indicates that although long fatigue cracks propagate more slowly at 300°C compared with those under equivalent conditions at 20°C, cracking initiates earlier at 300°C. This trend increasingly becomes greater as the strain amplitude decreases. Since fatigue cracks initiate due to the slip activity of dislocations during cyclic loading, the evolution of the microstructural condition was investigated, in particular the activity of localised deformation bands during cyclic loading.
Localised deformation bands

Fatigue cracks initiate in well annealed FCC materials due to the activation of localised slip bands, in particular PSBs. PSBs form as a consequence of the re-arrangement of dislocations during cyclic deformation, and are uniquely characterised by a ladder structure of dislocation walls and channels \cite{6, 11-13, 17, 18, 96}. The activity of PSBs was therefore carefully observed to reveal the role of PSBs on the fatigue crack initiation at two temperature conditions.

Figure VII.4: Formation of persistent slip bands during ±0.7% tests at (a) 20°C, and (b) 300°C. (c) Persistent slip bands are formed much later (about 6,000 cycles) for the ±0.4%-10³s⁻¹-20°C test. The strong activity of ladder-free localised slip bands is often observed after (d) 9,000 cycles for the ±0.4% test and (e) 20,000 cycles for the ±0.25% test at 300°C.
At 20°C, PSBs were observed by TEM and ECC at the earliest in the late stages of cyclic softening (i.e. after about 400 cycles) for the ±0.7% test (Figure VII.4a). The occurrence of PSBs is much later in lower strain amplitude tests, e.g. PSBs are first observed after 6,000 cycles for the ±0.4% test (Figure VII.4c). The volume fraction of PSBs decreases with reducing strain amplitude. Although TEM observation did not reveal the presence of PSBs for the ±0.25% test condition, it is believed that PSBs were also present after a large number of cycles for this condition, but with a much smaller volume fraction.

PSBs were also observed at 300°C, in particular for the ±0.7% test (Figure VII.4b). After a high number of cycles, a ladder-free type of deformation band formation becomes increasingly apparent with increasing cycle number (Figure IV.16e). This form of localised deformation band could be another type of PSB, but without the ladder structure. These localised deformation bands destroy the existing structures (e.g. well-organised cellular structures at the end of fatigue life for the ±0.7% test condition (Figure IV.16e), or tangled structures for the ±0.4% test condition (Figure VII.4d), or planar structures for the ±0.25% test condition (Figure VII.4e)). These ladder-free deformation bands were clearly observed after about 1500, 9000, and 20000 cycles respectively during the ±0.7, ±0.4, and ±0.25% tests.

VII.3.4. Surface examination

VII.3.4.1. Observation of free surface

The specimen gauge section surface roughness was greatly increased after cyclic loading due to the formation of localised deformation bands, e.g. PSBs at 20°C (Figure IV.4h, Figure VII.4a, c). The increase in surface roughness after fatigue is greater for higher imposed plastic strain amplitudes (Figure VII.5a, b). For the same strain
amplitude, the surface roughness is usually higher for the 300°C test condition (Figure VII.5c, d).

Figure VII.5: Surface condition at the end of fatigue life: (a) ±0.7%-10⁻³s⁻¹-20°C test, (b) ±0.25%-10⁻³s⁻¹-20°C test, (c) ±0.4%-10⁻³s⁻¹-20°C test, (d) ±0.4%-10⁻³s⁻¹-300°C test.

VII.3.4.2. Fracture surface and fatigue striations

With decreasing strain amplitude, the fracture surface becomes more faceted, i.e. fatigue cracks preferring to advance in a more crystallographic manner due to the more planar slip character of dislocations. For the same strain amplitude, more faceted fracture surfaces are observed at 300°C (Figure VII.6a, b). The crack origin is
identified from the presence of river patterns which converge to a point at the free surface (e.g. the crack initiation point marked by a small dashed circle in Figure VII.6a). The shape of the initiation region of the main crack can be approximated to be of semi-circular shape (refer to the big solid circle in Figure VII.6a). Fatigue striations belonging to the main crack were first observed after about one grain distance, two to four grain distances, and about ten grain distances respectively for the ±0.7, ±0.4, and ±0.25% test conditions. The appearance of very fine ridges recorded from the fracture surface was spot-checked by observing the side-view of short fatigue cracks in interrupted testpieces (Figure VII.6c) to confirm that they were fatigue striations (not microstructural features [195]). The spacing of the first observed fatigue striations were measured, and are given in Table VII.2. Table VII.2 clearly shows that, for almost the same crack length (and strain amplitude), the propagation rates of short fatigue cracks are lower for 300°C. This is consistent with the observation of the slower propagation rates for long fatigue cracks at 300°C in Sect.VII.3.2.

Figure VII.6: (a) Semi-circular shape of the crack close to the origin. The less faceted fracture surface at (a) ±0.4% -10^{-3}s^{-1}-20°C compared to that at (b) ±0.4%-10^{-3}s^{-1}-300°C. (c) Fatigue striations in the side-view of a short fatigue crack of about 150 μm in a testpiece fatigued at ±0.7% -300°C up to 1600 cycles have their average spacing of 0.49μm.
Table VII.2: Crack length and \( \frac{da}{dN} \) values based on fatigue striation observations.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Crack length (µm)</th>
<th>( \frac{da}{dN} ) (µm/cycle)</th>
</tr>
</thead>
<tbody>
<tr>
<td>±0.7% - 10^{-3}s^{-1} - 20°C</td>
<td>57</td>
<td>0.66</td>
</tr>
<tr>
<td>±0.7% - 10^{-3}s^{-1} - 300°C</td>
<td>62</td>
<td>0.39</td>
</tr>
<tr>
<td>±0.4% - 10^{-3}s^{-1} - 20°C</td>
<td>134</td>
<td>0.28</td>
</tr>
<tr>
<td>±0.4% - 10^{-3}s^{-1} - 300°C</td>
<td>148</td>
<td>0.21</td>
</tr>
<tr>
<td>±0.25% - 10^{-3}s^{-1} - 20°C</td>
<td>604</td>
<td>0.24</td>
</tr>
<tr>
<td>±0.25% - 10^{-3}s^{-1} - 300°C</td>
<td>1933</td>
<td>0.47</td>
</tr>
<tr>
<td>±0.25% - 10^{-3}s^{-1} - 300°C</td>
<td>686</td>
<td>0.20</td>
</tr>
<tr>
<td></td>
<td>726</td>
<td>0.28</td>
</tr>
</tbody>
</table>

**VII.3.5. Interaction between short fatigue cracks and dislocation walls**

The first noticeable fatigue striations observed by high resolution SEM were just after 1-2 grain diameters for the ±0.7% condition, which corresponds to the size of microstructurally short fatigue cracks [190]. The spacing of these fatigue striations can shed light on how microstructurally short fatigue cracks propagate in the presence of dislocation walls. The measured spacing of these striations is respectively 0.66 and 0.39µm for the ±0.7% tests at 20 and 300°C (Table VII.2). Interestingly, the striation spacing is almost equal to the spacing of dislocation walls (Figure VII.7a). The dislocation walls form quite early for these test conditions (after about 100 cycles (Figure IV.4 and Figure IV.16)). The coincidence between striation spacing and wall spacing implies that existing dislocation walls can strongly influence the propagation of microstructurally short fatigue cracks, which is indeed underpinned by the results of the ECC imaging. The ECC observations reveal that the tip of short fatigue cracks (whose length is of about 100 µm) was often arrested at (or coincide with) dislocation walls.
(Figure VII.7b). In the wake of short fatigue cracks, the crack path is observed to be slightly deflected at dislocation walls (Figure VII.7c). This mechanism of short fatigue crack propagation in the presence of existing dislocation walls is also observed for another FCC material [196]. This indicates that microstructurally short fatigue cracks are temporarily arrested by dislocation walls.

Figure VII.7: (a) Wall spacing measured at the ±0.7% conditions. (b) Microstructurally short fatigue crack went along dislocations walls. (c) A fatigue crack was deflected due to the presence of dislocation walls.
VII.4. Discussions

VII.4.3. The propagation of fatigue cracks

Compliance analysis and fatigue striation spacing measurements indicate that fatigue cracks at 300°C propagate more slowly than those under equivalent conditions at 20°C. When fatigue cracks grow to sufficient dimensions, they can leave striations in their wake. Crack growth rates associated with visible striation formation may be correlated with cyclic stress intensity factor ($\Delta K$). The propagation rate of fatigue cracks is found to be proportional to ($\Delta K/E$)$^m$, where $\Delta K = Y\Delta \sigma \sqrt{\pi a}$, and $m$ is a constant [4, 26, 197, 198]. In this study, $m=2$ as it was used in [197]. It is assumed on the basis of direct observations that crack shape is almost the same for the 20 and 300°C test conditions. For the same crack length $a$ for 20 and 300°C, the geometrical factor $Y$ is therefore almost identical. Consequently, the relative difference between the propagation rate of fatigue cracking at 20 and 300°C is approximately proportional to that of ($\Delta \sigma/E^2$). Figure VII.8 shows that the value of ($\Delta \sigma/E^2$) at 20°C is usually higher than that at 300°C for the same strain amplitude (i.e. Cyclic stress intensity factor should be higher at 20°C than (or equal to) that at 300°C for the same strain amplitude and crack size). This explains why the rate of fatigue crack propagation is lower for 300°C at the same strain amplitude. It means that the reduction of fatigue lifetime in the DSA regime definitely cannot be explained by the crack propagation rate (which was often invoked by other studies as mentioned before).

It is also possible that environment could be responsible for a reduction in fatigue life in the DSA regime since the fatigue resistance of AISI 316L in vacuum has been observed to increase in the DSA temperature regime [82, 199]. However, Mannan et al. and Hong have shown that, for a constant strain amplitude in air, fatigue life reduction is most significantly observed for strain rates (or temperatures) where DSA takes place [125, 192-194]. This means that the environment is likely to play an insignificant role on the fatigue life reduction at low temperatures in the DSA regime. Although Gerland et
al. [82, 199] observed longer fatigue endurances in vacuum, these authors did not clearly define their adopted crack initiation criterion, and the number of cycles to failure reported in their study likely corresponded to a higher value of stress drop, for which fatigue crack propagation rate can be a governing factor on the fatigue life. As shown in Sect. VII.3.2 and Sect. VII.3.4.2, the crack propagation rate of fatigue cracks can be lower for temperatures in DSA regime compared to equivalent conditions at 20°C. This can make the fatigue life determined in terms of a high stress drop criterion longer in this regime. The discussions of the influence of the fatigue crack propagation rate and environment above indicates that the reduction in fatigue endurance at 300°C relative to that at 20°C is probably due to the early initiation of fatigue cracks, in particular for lower strain amplitudes, as a consequence of the activity of localised deformation bands at 300°C.

Figure VII.8: The value of \( \left( \frac{\Delta \sigma}{E} \right)^2 \) for cycle numbers from \((0.5-1)N_{2\%}\)
VII.4.2. Fatigue crack initiation and the reduction of fatigue life at 300°C

The mechanism to initiate fatigue cracks in smooth testpieces is related to the activity of localised deformation bands. The bands cause the formation of notch-like features at the free surface during cyclic loading, resulting in stressraisers which lead to the initiation of fatigue cracks. In the regime of non-DSA, the characteristic localised deformation bands during cyclic loading of FCC materials are PSBs [11-13, 17, 18]. In a certain range of imposed shear plastic strain ($\gamma_p$), the volume fraction of PSBs is almost linearly proportional to $\gamma_p$ [19, 200, 201]. In this present study, PSBs were observed to be: (1) most active at the highest total strain amplitude (i.e. ±0.7%) for both 20 and 300°C, (2) increasingly less active for lower strain amplitudes. The strong activity of PSBs consequently results in the highest degree of roughened free surface after fatigue loading for the ±0.7% conditions (Figure VII.5).

At 300°C, a ladder-free type of deformation band increasingly develops after high numbers of cycles (e.g. four parallel localised slip bands in Figure IV.16e; Figure IV.18 and Figure VII.4d, e). These localised deformation bands comprise of a significantly large quantity of vacancies (Figure IV.18a, c and Figure IV.21c), resulting in a volume increase in the region of localised bands, and leading to the formation of extrusions on the free surface. Although the strong interaction between solute atoms and dislocation lines in the DSA regime enhances the planar character of dislocation movement, the planar character can be much different for the normal planar movement of dislocations in the low density condition in the non-DSA regime. Dislocations move in a planar manner on the same gliding plane back and forth during cyclic loading for low strain amplitude conditions where dislocation density is low. This increases the reversibility of dislocation motion, resulting in less fatigue damage during cyclic deformation [14]. The planar movement of dislocations in the DSA regime is however characterised by a strong stick-slip mechanism. The strong interaction between solute atoms and dislocations locks dislocations, resulting in a deficiency in the number of mobile dislocations necessary to accommodate prescribed plastic deformation. This leads to
an increase in the applied stress, and to the build-up of stress concentration in regions where dislocations are locked. Once the applied stress reaches a critical value, a mass of dislocations suddenly move, causing the stress drop and the plastic instabilities. This unstable movement of dislocations does not increase the reversibility of dislocation motion, but induces more fatigue damage during cyclic loading in the DSA regime due to the sudden and rapid movement of massive dislocations.

For the ±0.7% test condition, PSBs are intensively active very early in life, while ladder-free bands develop much later. This means that PSB formation is likely to be the most influential deformation mechanism responsible for crack initiation for the ±0.7% tests, resulting in the negligible difference in fatigue life between 20 and 300°C. The lower crack propagation rate at 300°C results to a slightly higher fatigue endurance at this elevated temperature compared to the equivalent condition at 20°C (Table VII.1). The volume fraction of PSBs linearly decreases with reducing strain amplitude. The material can therefore endure higher numbers of cycles for lower strain amplitudes. The contribution of the ladder-free bands to the crack initiation process becomes much greater when strain amplitude decreases. This type of localised slip band is only strongly active after a high number of cycles at this elevated temperature, and leads to fatigue crack initiation much earlier for lower strain amplitude tests at 300°C. This explains why there is a reduction in fatigue life at 300°C relative to at 20°C, and the relative life reduction increases with reducing strain amplitude at 300°C. Moreover, the material becomes harder with the number of cycles due mainly to the development of the corduroy structure at 300°C, resulting in increasing stress range during the strain-controlled test [134]. The rate of crack propagation accelerates due to a synergistic increase in the crack length and stress range after a high number of cycles. This partly leads to the reduction to be more obvious for lower strain amplitudes.
VII.4.3. Role of dislocation walls on the propagation of microstructurally short fatigue cracks

For microstructurally short fatigue cracks in smooth testpieces, there is only a small number of dislocations emitted from the crack tip during the crack opening process [202-206]. Dislocations then slip away from the crack tip and pile up at obstacles in their path. Since dislocation walls are harder phases and quite effectively restrict the movement of mobile dislocations, the emitted dislocations are piled up at dislocations walls. Local stress induced by the number of dislocations in pile ups is increased until it is sufficient to break dislocation walls. Dislocation walls thereby restrict the crack tip opening, i.e. they govern the crack advance per cycle. Once the local stress is higher than the strength of dislocation walls, microstructurally short fatigue cracks can easily cross the walls. Because of the misorientation from channel to channel, fatigue cracks slightly change their direction when they cross dislocation walls, leaving their deflected wakes (Figure VII.7c). The effectiveness of dislocation walls on the retardation of the propagation of microstructurally short fatigue cracks causes a greater difference of the propagation rate of the fatigue cracks between 20 and 300°C for the ±0.7% test condition compared to lower strain amplitudes (Table VII.2). This effectiveness can also explain a significant increase in the fatigue life of AISI 316L when it is first low-cycle fatigued (with the total strain amplitudes of ±0.5% to ±1.5%) up to a relative small number of cycles (about 250 cycles), and subsequently subjected to high-cycle fatigue loading [207, 208].
VII.5. Concluding remarks

At 300°C, when dynamic strain ageing takes place, the fatigue life of AISI 316L is found to be reduced compared to that at 20°C for lower strain amplitudes. Analysis of apparent elastic modulus and fracture surface evidence clearly shows that fatigue crack propagation rates are faster at 20°C. Crack initiation is however shown to occur earlier at 300°C, in particular for lower strain amplitude tests. This is due to the activity of localised slip bands during cyclic deformation. The strong activity of persistent slip bands is mainly responsible for the increase in the surface roughness after cyclic loading at 20 and 300°C, in particular for the ±0.7% test condition. Ladder-free slip band development is common after a high number of cycles at 300°C causing higher surface roughness at this temperature, in particular for lower strain amplitudes where the endurance is high.

For the ±0.7% test condition at 300°C the fatigue life is relatively short, and PSB formation is the predominant deformation mechanism. Ladder-free slip bands develop later in life and their influence on fatigue endurance at 300°C is therefore negligible, especially considering that the propagation rate of short fatigue cracks is rather lower at this temperature compared to that at 20°C. As the strain amplitude decreases, PSBs appear to be much less active and the material endurance increases. Crack formation occurs later in the fatigue life and ladder-free slip band development is more influential in this process. Consequently, the life reduction becomes increasingly significant with reducing strain amplitude. The occurrence of secondary cyclic hardening in lower strain amplitude tests at 300°C also makes the life reduction more apparent.

The role of dislocation walls on the propagation of microstructurally short fatigue cracks is also studied. The striation spacing of such fatigue cracks is almost equal to the spacing of dislocation walls, indicating that dislocation walls influence the propagation of microstructurally short fatigue cracks. This finding is underpinned by the results of ECC investigations which show that the path of such fatigue cracks is often deflected at dislocation walls.
Chapter VIII: Summary and conclusions

A great effort has been made over the last four years under the supervision of experts from Empa, ETHZ and PSI to systematically characterise the cyclic deformation response of AISI 316L at 20 and 300°C both in terms of mechanical response and microstructural evolution in order to develop an evolutionary constitutive model. To achieve the objective, a number of specimens (fatigued up to selected numbers of cycles, and to failure) were performed to provide data for mechanical data analysis and for microstructural observation which was intensively conducted by electron microscopy. The physical interpretations of internal stresses (effective stress, inter-granular/intra-granular back stress) have been thoroughly discussed to provide deeper insights into the relationship between microstructural condition and mechanical response of the material during cyclic loading. The comprehensive relationship between internal stresses and microstructural condition during cyclic loading were then established. In addition, the relative contribution of internal stresses to the material cyclic response has also been carefully examined to shed light on the amplitude/temperature dependence of the relationship between microstructural condition and the cyclic deformation response. An evolutionary constitutive model has been successfully developed on the basis of the established relationships between the microstructural condition and internal stresses in order to accurately describe and well predict the material cyclic deformation response at 20 and 300°C.
VIII.1. Cyclic deformation response: mechanical behaviour and microstructural evolution

AISI 316L exhibits a complex cyclic deformation response at 20 and 300°C, which can be characterised by three stages: firstly cyclic hardening, followed by cyclic softening, and finally an almost stabilised response phase (for all tests at 20°C, and ±0.7% at 300°C) or secondary hardening (for ±0.25 and ±0.4% at 300°C) before failure. The main differences in the cyclic deformation response of the material at 300°C compared to that at 20°C are: firstly, flow stress is serrated at 300°C (and not at 20°C); secondly the strain rate sensitivity of cyclic deformation response is positive at 20°C, but negative at 300°C; thirdly the hardening rate of the first cyclic hardening phase is higher at 300°C than that at room temperature; and fourthly there is a secondary hardening phase at 300°C which occurs for lower strain amplitude conditions (i.e. ±0.4, and ±0.25%) (This is not observed at 20°C).

In the as-received condition, dislocations are mainly present in the form of planar structures, e.g. regular arrays of dislocations, stacking faults etc. At the beginning of cyclic loading, grain-to-grain misorientations inducing plastic strain incompatibilities between grains, i.e. at an inter-granular scale, result in a rapid and significant increase in dislocation density close to the grain boundaries to preserve the continuity of material. In contrast, there is a slower increase in dislocation density within grains. At the end of cyclic hardening, dislocation-dense sheets are observed, in particular for higher strain amplitudes. Dislocations then rearrange during the softening stage due to the strong activation of secondary slip (including cross slip). Secondary slip activity promotes dislocation interactions, resulting in: 1) an increase in the annihilation rate of dislocations, leading to a slight decrease in dislocation density; and 2) the rearrangement of dislocations to form lower and more stable energetic configurations, i.e. dislocation high/low density regions, to further relieve the grain-to-grain strain incompatibilities. Although the development of dislocation structures relieves the grain-to-grain incompatibilities, it raises other strain incompatibilities between dislocation high/low density regions on a finer scale, i.e. intra-granular scale. The stabilised
condition of these strain incompatibilities is established upon further loading once
dislocations seek their most stable configuration under a given testing condition. This is
the case at 20°C, and is responsible for the cyclic saturation response after a high
number of cycles at this temperature condition. However, due to the continuous
development of point defects (in particular the coalescence of vacancies), the material
does not show the stabilised response after a high number of cycles, but rather a
secondary cyclic hardening.

Dislocation high/low density regions are strongly dependent on imposed plastic strain.
For high strain amplitude, the strong multiple slip activity upon further loading is
responsible for the development of the wall/channel structure into the cellular structure
towards the end of life. For lower strain amplitude tests, dislocation structures are less
well organised at the end of life. The effectiveness of dislocation structures on the
restriction of dislocation movement is therefore smaller. In addition, the plastic strain
incompatibility between dislocation high/low density regions is also less. This explains
why the peak stress at the saturation stage is lower for smaller strain amplitude
conditions.

An effort is made to understand the mechanism of the dynamic strain ageing (DSA)
phenomenon which causes the complex cyclic deformation response at 300°C. DSA
strikingly manifests in flow stress serrations which are very active at first, and then
gradually disappear. There is then almost no stress serration during the cyclic softening
stress response regime in particular in low strain amplitude tests. Finally, DSA reoccurs
after a further number of cycles. The first active phase of serration is due to the
interaction of Suzuki clouds with both edge and screw dislocations. Upon further
loading, edge dislocations are almost immobile because of both Lomer-Cottrell and
Suzuki locking. In contrast, the formation of dislocation junctions can facilitate extended
screw dislocations to cross slip since the activation barrier of partial dislocation
recombination is reduced thanks to the formation of junctions. Consequently, stress
serration decreases gradually during the first cyclic response phase, then almost
disappears during the softening phase over which cross-slip is most active. Snoek
atmospheres of solute atoms however form after a number of cycles, and these are responsible for the re-occurrence of stress serration.

The more significant primary cyclic hardening of the material is due to the strong interaction of solute atoms with dislocations. At the beginning of cyclic loading, Suzuki atmospheres of solute atoms forming around dislocations, along with Lomer-Cottrell junctions, can effectively lock dislocations, resulting in the pronounced primary cyclic hardening. During the secondary cyclic hardening phase, additional solute atom atmospheres, i.e. Snoek short-range-ordered atmospheres, form to strengthen locking effects. In addition, corduroy structure is also observed during this stage. The two conditions are responsible for the secondary cyclic hardening response. The contribution of corduroy structure to the secondary cyclic hardening response is however greater than that for Snoek atmospheres.
VIII.2. Relationship between microstructural condition and internal stresses

Inter-granular back stress associating with the long-range interaction of dislocations arises under imposed plastic deformation due to the grain-to-grain strain incompatibility. This back stress significantly evolves with the development of boundary in the beginning, and with the grain fragmentation upon further cyclic loading. The activity of secondary slip during cyclic loading on the one hand relieves the inter-granular back stress, but on the other hand raises the intra-granular back stress due to the formation of dislocation-high/low-density regions, e.g. walls and channels. $X_{\text{inter}}$ and $X_{\text{intra}}$ reach their saturated values once microstructure seeks its most stable configuration under a given testing condition. Back stresses are mainly responsible for cyclic deformation response, in particular in case of wavy slip mode conditions. In contrast, effective stress (associating with the short-range interactions of dislocations) plays a much less significant role on the material cyclic response for wavy slip conditions, but increasingly becomes more important for planar slip conditions. In case there is no strong interaction between dislocations and point defects, effective stress is proportional to the number of dislocation sessile dislocation junctions. At 300°C, the interaction between dislocations with point defects causes dynamic strain ageing, and results in a different response to that at 20°C, i.e. i) a more significant primary cyclic hardening, and ii) the occurrence of a secondary cyclic hardening response. In detail, profuse point defects generated at 300°C promote the formation of solute atom atmospheres around dislocations and later the formation of a corduroy structure, both of which restrict the movement of dislocations, inducing a significant increase in effective stress. As a result, the effective stress is mainly responsible for the different cyclic deformation response of the material at 300°C, especially for the secondary cyclic hardening response.
VIII.3. Constitutive modelling

An evolutionary physically-based constitutive model of internal variables has been successfully developed based on the knowledge gained of the relationships between microstructural condition and internal stresses during cyclic loading (i.e. Eqns VI.8, VI.11 and VI.17). In addition, the proposed model also incorporates the temperature dependence of the relationships between microstructural evolution and internal stresses (Eqn. VI.30), in particular regarding the interaction between dislocations and solute atom atmospheres which associates with the DSA phenomenon at 300°C. The model parameters are identified by systematic evaluations of mechanical and microstructural observations from a number of interrupted fatigue tests for a single test condition at 20°C (293K). The quantification of dislocation evolution for this test condition and for the equivalent condition at 300°C provides the information to identify the model parameters at 300°C (573K). The proposed model provides an excellent description of the complex cyclic elasto-plastic deformation behaviour of AISI 316L at 20 and 300°C. On account of the changing characteristics of microstructural evolution and its consequence for the change in internal stresses, $f_b$, $\sqrt{\rho_{b_{\text{hom}}}}$ and $\Delta \sqrt{\rho_{\text{sat}}}$ need to be adjusted to accurately predict the long-term cyclic deformation response for lower strain amplitudes at a given temperature.

The capability of the model is then benchmarked by predicting the material cyclic response for independent testing conditions. The model benchmarking shows that the proposed model is very capable of predicting the cyclic elasto-plastic deformation behaviour during variable amplitude fatigue loading.
VIII.4. Role of microstructural condition on the development of fatigue damage

The fatigue life of AISI 316L for lower strain amplitudes at 300°C is found to be reduced compared to equivalent conditions at 20°C. This relative reduction of fatigue life was often invoked by other studies to be due to: 1) an enhanced degree of plastic deformation inhomogeneity in the dynamic strain ageing (DSA) regime without a clear description, and 2) an increased crack propagation rate at the higher temperature. This study however shows that the fatigue crack propagation rate at 300°C is lower than that for equivalent conditions at 20°C, meaning that the reduction of fatigue lifetime in the DSA regime cannot be explained by the crack propagation rate. The influence of microstructural evolution on the fatigue damage development of AISI 316L at 20 and 300°C was therefore exhaustively examined to understand the mechanism responsible for the observed relative reduction in life at the higher temperature. Chapter VII reveals that when the fatigue life is rather short, ladder-like persistent slip bands (PSBs) are influential in the fatigue crack initiation at both 20 and 300°C. As the strain amplitude decreases, PSBs become much less active and the material endurance increases. Crack formation therefore occurs later in the fatigue life. At 300°C, ladder-free slip bands increasingly develop after a high number of cycles, leading to fatigue crack initiation much earlier for lower strain amplitude tests at this elevated temperature. This explains why there is a reduction in fatigue life at 300°C relative to at 20°C, and the relative life reduction increases with reducing strain amplitude at 300°C. The occurrence of secondary cyclic hardening for lower strain amplitude tests at 300°C also makes the life reduction more apparent.
VIII.5. Outlook

VIII.5.1. Short-range ordered atmospheres of solute atoms

Because C and N cannot be directly imaged by TEM, the configuration of solute atoms in short-range ordered atmospheres around dislocation lines is still unclear. The presence of short-range ordered atmospheres was only realised in this study by selected area diffraction patterns (Figure IV.22). It would be very interesting to reveal how solute atoms position in short-range order atmospheres by modern atom probe microscopes [115].

VIII.5.2. Further developments of the proposed evolutionary constitutive model

VIII.5.2.1. Implement into finite element framework

The similarity of Eqn. VI.20 with the two term back stress equation of Chaboche [140] means that this new microstructurally based equation set will be readily: 1) implementable in commercial finite element programs such as Abaqus and Ansys, and 2) extendable for multi-axial fatigue assessment. Regarding the multi-axial fatigue conditions, multi-axial loading facilitates strong multiple slip activity, resulting in the more rapid formation of dislocation tangled structures, i.e. dislocation walls and channels quickly form during multiaxial loading [180]. The evolution of microstructural condition during multiaxial cyclic loading is found to be qualitatively similar to that during uniaxial condition. The dislocation condition basically evolves from planar structures to the wall/channel structure during multiaxial cyclic loading for AISI 316L [180, 181]. In the near future, it will be worth evaluating how effective the evolutionary model is for multi-axial fatigue assessment if the latent hardening and the more rapid development of dislocation wall/channel structure are appropriately considered.
VIII.5.2.2. Modelling the cyclic deformation response during strain-controlled ratcheting

The proposed model capability was applied for modelling the cyclic deformation response during strain-controlled ratcheting in order to understand not only the effectiveness but also the limitation of the proposed model (which will provide some potential directions for future study) (Appendix 2). This examination of the model capability indicates that in order to improve the effectiveness of the proposed model, it is necessary to include the mean-strain dependence of microstructural evolution, in particular crystallographic anisotropy, e.g. [92, 209, 210]. In particular, it will be very helpful to understand the detailed difference between the microstructural evolution during strain-controlled ratcheting cycles and equivalent cycles during constant strain amplitude cyclic loading.

VIII.5.2.3. Incorporation of the change in vacancy concentration during cyclic loading

The extent of interaction between dislocations and point defects varies with the evolution of vacancy concentration during cyclic loading at 300°C. An improvement of the model predictive capability to better represent the evolution of effective stress can be made by considering the evolution of $C_s$ (in Eqn. VI.31) with the change in vacancy concentration (e.g. Appendix 3) [40, 80, 211].

VIII.5.3. Ladder-free slip bands

Ladder-free slip bands are influential in the fatigue crack initiation process for low strain amplitude tests of AISI 316L at 300°C, which mainly responsible for the reduction in fatigue life compared to equivalent conditions at 20°C (Chapter VII). It is important to quantify the height of extrusions which result from the ladder-free band formation. This can be done by examining the surface condition of fatigued samples by atomic force microscopy.
Appendix 1: Solution of Eqn. (VI.2)

Choosing \( u = \sqrt{\rho} \), then \( d\rho = 2udu \)

Eqn. (VI.2) \( \Leftrightarrow \frac{2udu}{k_1u - k_2u^2} = d\varepsilon_p \)
\( \Leftrightarrow \frac{2du}{k_1 - k_2u} = d\varepsilon_p \)
\( \Leftrightarrow d\ln(k_1' - k_2'u) = d\left(-k_2'\varepsilon_p\right) \)
with \( k_1' = k_1/2 \), and \( k_2' = k_2/2 \)
\( \Leftrightarrow C + \ln(k_1' - k_2'u) = -k_2'(\varepsilon_p - \varepsilon_0) \)
\( \Leftrightarrow k_1' - k_2'u = e^{-c} \times e^{-k_2'\varepsilon_p} \)
with \( \varepsilon_0 = 0 \)
\( \Leftrightarrow u = \sqrt{\rho} = \frac{k_1'}{k_2'} \times \frac{e^{-c}}{k_2'} \times e^{-k_2'\varepsilon_p} \)

Assuming that dislocation motion is the only active mechanism of plastic deformation, if \( \sqrt{\rho} = 0 \), then \( \varepsilon_p = 0 \), leading to \( \frac{k_1'}{k_2'} = \frac{e^{-c}}{k_2'} \) or \( k_1' = e^{-c} \)

Therefore, \( \sqrt{\rho} = \frac{k_1'}{k_2'} \left(1 - e^{-k_2'\varepsilon_p}\right) \)
Appendix 2: Modelling the cyclic deformation response during strain-controlled ratcheting.

In this section, the proposed model capability was applied for modelling the cyclic deformation response during strain-controlled ratcheting. Strain-controlled ratcheting tests were conducted at 20°C with a strain amplitude of ±0.4%, and a strain rate of $3.2 \times 10^{-3}$ s$^{-1}$. The mean strain was increased at different rates (0.1, and 0.01% per cycle) until a maximum value of 5% was reached (Figure A2.1). The mean strain was then kept constant. The experimental data of strain-controlled ratcheting tests was kindly provided by Giacomo Facheris of PSI. The cyclic deformation response of AISI 316L during strain-controlled ratcheting is shown in Figure A2.2. The material continuously hardens until the mean strain reaches its maximum value (5%). When the mean strain is kept constant, the material starts softening, and then reaches the stabilised response stage before failure [212].

![Figure A2.1: Strain path during cyclic tension test (i.e. strain-controlled ratcheting test).](image)
Figure A2.2: Cyclic deformation response of AISI 316L during strain-controlled ratcheting at 20°C

Figure A2.3: Modelling the cyclic deformation response of AISI 316L during strain-controlled ratcheting at 20°C.

In the beginning of cyclic loading (for both zero- and non-zero-mean strain loading conditions) the model describes well the material cyclic response (Figure A2.3a, b and...
c). However, after a number of cycles coincident with where the material obtains the peak of cyclic hardening during zero-mean-strain cyclic loading, the material still continues to hardens during the strain-controlled ratcheting test. This significant cyclic hardening of the material cannot be represented well by the proposed model (Figure A2.3a, b and d). Since the current modelling approach has not considered the effect of mean strain (mean stress) on the development of microstructure, the proposed model cannot accurately describe the cyclic deformation response of the material during strain-controlled ratcheting loading stage. The proposed model nevertheless becomes much more effective in representing the material response after a high number of cycles (Figure A2.3a, b and e). This is likely due to the fact that the material is relaxed during fatigue loading with a constant mean strain, i.e. the crystallographic anisotropy of microstructure during ratcheting disappears.
Appendix 3: Evolution of vacancy concentration during cyclic loading

The evolution of vacancy concentration can be described as follows [40, 80, 211].

\[ C_s = C_{s, \text{sat}} \left\{ 1 - \exp \left[ -\pi C_{s,0} \left( \frac{KD_s t_w}{C_{s,\text{sat}}} \right)^\gamma \right] \right\} \]

Where:

1) \( r \) is of 1/3 or 2/3 for the pipe diffusion or lattice diffusion.
2) \( C_{s,0} \) and \( C_{s,\text{sat}} \) are the nominal concentration, and the saturation concentration of solute atom \( i \) on dislocation cores, \( C_{s,\text{sat}} = C_{s,0} \exp\left(\frac{U_{ni}}{kT}\right) \)
3) \( t_w \) is the average waiting time of dislocations at obstacles, Normally, the travelling time \( (t_{tr}) \) of mobile dislocations moving through discrete obstacles is negligible compared to the waiting time \( (t_w) \). According Orowan’s kinetic equation

\[
\gamma = \rho_{mo} b \bar{v} = \rho_{mo} b \frac{L}{t_{tr} + t_w} \approx \rho_{mo} b \frac{L}{t_w} = \rho_{mo} b \frac{\rho_i^{-1/2}}{t_w}
\]

Therefore,

\[
t_w = \rho_{mo} b \frac{\rho_i^{-1/2}}{t_w}
\]
4) \( K = \frac{3U_m}{b^2 kT} \) is constant which includes the solute-dislocation binding energy \( (U_m) \). \( U_m \approx 0.5 \text{eV} \)
5) \( D_s \) is vacancy-assisted diffusion coefficient.
\[ D_s = C_v a^2 \nu \exp\left(\frac{\Delta S_{vf} + \Delta S_{vm}}{k}\right) \exp\left(-\frac{G_{vf} + G_{vm}}{kT}\right) \]

Where: \( G_{vf} \) and \( \Delta S_{vf} \) are respectively activation energy to form a vacancy and the change in entropy when a vacancy is created.

\( G_{vm} \) and \( \Delta S_{vm} \) are respectively activation energy to move a vacancy and the change in entropy when a vacancy jumps.
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