Creep Fatigue Crack Growth in Advanced Martensitic Turbine Steels

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Abstract

The aim of this research project is to investigate sub-critical crack growth behaviour under creep-fatigue deformation conditions for advanced martensitic steam turbine rotor steels. The tasks are approached by studies concerning (1) the mechanical characterization of cyclic response and crack development; (2) the microstructural evolution as a result of creep-fatigue loading and crack propagation; and (3) the assessment of various crack growth models including new developments. The main focus is on short crack development for which a large proportion of component lifetime is spent.

For short crack growth testing, two series of isothermal strain controlled creep-fatigue tests on fully instrumented cylindrical specimens with shallow chordal crack starters have been conducted for advanced 9%Cr and 10%Cr turbine rotor steels at 600°C and 625°C. Cyclic/hold wave shapes involving a dwell period at peak strain in tension or compression have also been performed with crack development being monitored by means of electrical potential drop instrumentation. It is found that total strain range and hold period are the most influential factors controlling crack development, and the accelerated crack growth rates for dwelled specimens are caused by the effects of prior creep and strain enhanced oxidation damage ahead of the crack tip.

Subsequently, extensive work on post-test microstructural examination has been carried out by using optical microscopy and electron microscopy, and in particular the EBSD (electron backscattering diffraction) technique. Samples from short-crack creep-fatigue crack growth tests were systematically examined, with regard to the evolution of martensite morphology, crack propagation path, oxide layer, micro-cracks, etc. It is found that microstructural evolution took place during deformation, which was governed by the applied magnitude and duration of creep-fatigue loading. The characteristic values of micro-grain diameter have been quantified, and can be semi-empirically described as a function of crack depth, hold time and other test-related parameters.
In order to rationalize crack growth behaviour under diverse testing conditions, various models have been used to correlate different parameters with crack growth rates. The employed models for short-crack creep-fatigue crack development are: (1) the fracture mechanics based models (K type and J type); (2) the model based on SEDF (strain energy density factor); (3) the Tomkins model; and (4) the Skelton model (originating from Pineau). The SEDF model and Skelton model appear to be more effective than the other models, especially in their capability to incorporate the influence of hold periods. Similarities and differences between the evaluated models are also discussed and suggestions are given with regard to their practical application.

More importantly, with the support of microstructural analysis, it is possible to partially associate the acceleration of crack development to the change of micro-scale strengthening sub-structures. By defining a microstructural condition parameter (e.g. related to micro-grain diameter) and integrating it into the proposed crack growth models, the performance of crack growth prediction can be improved.

With this study on mechanical characterization, microstructural characterization and candidate crack growth models for short crack creep-fatigue experiments, a comprehensive view on the materials properties and damaging mechanisms for the tested steels can finally be obtained. The devised crack growth models can potentially improve the effectiveness of existing assessment procedures for steam turbine components.
Zusammenfassung

Ziel dieses Forschungsprojektes ist die Untersuchung des unterkritischen Risswachstums in hochentwickeltem martensitischem Turbinenläuferstahl unter Kriechermüdung. Dazu werden (1) die Rissentwicklung und das Verhalten unter zyklischer Last charakterisiert, (2) die Veränderung der Mikrostruktur als Folge von Kriechermüdung und Risswachstum beschrieben und (3) verschiedene Risswachstumsmodelle bewertet, einschließlich Neuentwicklungen. Der Schwerpunkt liegt dabei auf der Entwicklung kurzer Risse, ein Zustand, welcher einen Grossteil der Bauteillebensdauer einnimmt.


Um das Risswachstum bei variierenden Versuchsbedingungen zu erklären, wurden verschiedene Modelle verwendet, damit die unterschiedlichen Parameter mit dem Risswachstum in Beziehung gesetzt werden können. Die zur Untersuchung des Kurzrissermüdungsverhaltens eingesetzten Modelle sind: (1) Die bruchmechanikbasierten Modelle (\(K\) - und \(J\) -Typ), (2) das auf dem SEDF (strain energy density factor) basierende Modell (3) das Tomkins-Modell und (4) das Skelton-Modell (ursprünglich von Pineau). Das SEDF- und das Tomkins-Modell scheinen effektiver als andere Modelle zu sein, insbesondere wegen der Fähigkeit, den Einfluss der Halteperioden wiederzugeben. Ähnlichkeiten und Unterschiede zwischen den untersuchten Modellen werden ebenfalls diskutiert, und es werden Empfehlungen zur praktischen Anwendung gegeben.

Von Bedeutung ist vor allem, dass es durch die Analyse der Mikrostruktur möglich ist, die Beschleunigung der Rissentwicklung teilweise mit der Veränderung der mikroskopischen verfestigenden Strukturen in Verbindung zu bringen. Durch die Definition eines mikrostrukturbasierten Schadensparameters (beispielsweise bezogen auf den Mikrokorn-Durchmesser) und Einbeziehung in die vorgeschlagenen Risswachstumsmodelle kann die Leistungsfähigkeit der Risswachstumsvorhersage erhöht werden.

Durch diese Studie über die mechanischen Eigenschaften, die Mikrostruktur und über die in Frage kommenden Risswachstumsmodelle für Kriechermüdungsversuche mit kurzen Rissen ist es endlich möglich, die Materialeigenschaften und Schädigungsmechanismen des untersuchten Stahls umfassend in den Blick zu nehmen. Die entwickelten Risswachstumsmodelle haben das Potential, die Leistungsfähigkeit der bisherigen Beurteilungsverfahren für Komponenten von Dampfturbinen zu erhöhen.
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**Nomenclature**

- \( a \) Crack depth
- \( a_0 \) Initial crack depth
- \( a_f \) Final crack depth
- \( a_{temp} \) Temporary crack depth in the PD calibration process
- \( a_{f, temp} \) Temporary final crack depth in the PD calibration process
- \( A \) Constant in stress range-plastic strain range equation
- \( A' \) Constant in Ramberg-Osgood equation
- \( A_C \) Constant in the \( (C_t)_{avg} \) type crack growth model
- \( A_J \) Constant in the \( J \) type crack growth model
- \( A_K \) Constant in the \( K \) type crack growth model
- \( A_{pl} \) Area surrounded by load-displacement curve in a single loop
- \( A_w \) Constant in the SEDF type crack growth model
- \( b \) Half width of a centre cracked plate
- \( b_1, b_2 \) Constants in the micro-grain size model
- \( c_1, c_2 \) Exponents in the micro-grain size model
- \( B \) (Short crack development) a strain related function in the Skelton model
- \( B \) (Long crack development) specimen thickness for a CT testpiece
- \( B_N \) The net thickness at side grooves for a CT testpiece
- \( C_{LL} \) Load line compliance
- \( C^* \) Path independent line integral characterizing crack tip stress/strain rate fields
- \( C_t \) Parameter to extend \( C^* \) into small-scale and the transition creep regime
- \( (C_t)_{avg} \) The average value of the \( C_t \) parameter during the hold time of the cycle
- \( d_c \) Creep damage accumulated per cycle in the Skelton model
- \( d_m \) Average micro-grain diameter
- \( ds \) An element of arc length (contour path) around a crack tip
- \( D \) Diameter of a round bar specimen
- \( E \) Young's modulus (elastic modulus)
- \( E' \) Young's modulus for plane strain condition (i.e. \( E' = E(1 - \nu^2) \))
- \( E_{RT}, E_0 \) Young's modulus at room temperature and at the start of the test
- \( f \) Sampling frequency in testing/data recording
- \( F', F \) Geometrical associated factors for CT specimens
- \( g \) Material and geometry dependent function in \( J \) integral calculation
- \( G \) Strain energy release rate
- \( k \) Coefficient between friction stress and (cyclic) yield strength
- \( k_{HHP}, k_{HHP}' \) Original and modified Hall-Petch coefficient
- \( K \) Stress intensity factor
$K_{\text{max}}, K_{\text{min}}$  Maximum and minimum stress intensity factor (in a cycle)
$\Delta K$  Range of stress intensity factor in a cycle
$\Delta K_{\text{eff}}$  Effective stress intensity factor range in a cycle
$\Delta K_{\text{eq}}$  Equivalent stress intensity factor range in a cycle
$K_{\text{open}}$  Stress intensity factor for which the crack is open
$J$  Path independent line integral characterizing crack tip stress/strain fields
$\Delta J_t$  Total range of $J$ integral in a cycle
$\Delta J_e, \Delta J_p$  Elastic and plastic parts of $\Delta J_t$
$m_c$  Exponent in the $(c')_{avg}$ type crack growth model
$m_J$  Exponent in the $J$ type crack growth model
$m_K$  Exponent in the $K$ type crack growth model
$m_w$  Exponent in the SEDF type crack growth model
$n$  Strain hardening exponent
$n'$  Exponent in Norton's law
$N$  Number of cycles
$N'$  Reduced number of cycles (due to creep-fatigue interaction)
$N_f$  Number of cycles to failure
$N_{2\%}$  Number of cycles to 2% load drop
$P, \Delta P$  Load and load range
$P_{\text{max}}, P_{\text{min}}$  Maximum load and minimum load
$P_c, P_o$  Crack closure load and crack opening load
$q$  Effective load ratio
$q_o, q_c$  Fraction of the total strain range for which a crack is open or close
$Q$  Material dependent exponent in the Skelton model
$R_e$  Strain ratio ($\epsilon_{\text{min}} / \epsilon_{\text{max}}$)
$R_\sigma$  Load ratio ($P_{\text{min}} / P_{\text{max}}$, or $\sigma_{\text{min}} / \sigma_{\text{max}}$)
$t_h$  Hold time in a cycle
$t_R$  Creep rupture time
$T_i$  Traction vector on $ds$
$\bar{T}$  Equivalent tensile stress term in the Tomkins model
$T$  Temperature
$u_i$  Displacement vector at $ds$
$\dot{u}_i$  Displacement rate vector at $ds$
$U, U'$  Potential energy and potential energy rate
$V$  Load line displacement
$\Delta V$  Change in load line displacement
$\Delta V_c$  Difference in load line displacement during the hold period
\( \Delta V_{pl} \) The plastic part of load line displacement range
\( \Delta V_{total} \) Total load line displacement range
\( \dot{V} \) Load line displacement rate (i.e. \( dV/dt \))
\( \dot{V}_e, \dot{V}_p, \dot{V}_c \) Elastic, plastic and creep part of \( \dot{V} \)
\( \dot{V}_{ss} \) Steady-state load line displacement rate
\( \overline{V} \) Instantaneous voltage value
\( \overline{V}_{0.2} \) Initial voltage value (i.e. at \( a_0 = 0.2 \text{mm} \))
\( \overline{V}_a, \overline{V}_b \) Voltage values at the beginning and end of the hold period
\( \overline{V}_{\text{max}} \) Maximum voltage value (i.e. under tensile peak stress)
\( \Delta \overline{V}_{\text{mean}} \) Mean voltage change during dwell in a single cycle
\( \Delta \overline{V}_{\%} \) Relative voltage change (i.e. \( \Delta \overline{V}_{\%} = (\overline{V} / \overline{V}_a - 1) \times 100\% \))
\( w_{id} \) Hysteresis energy expended per cycle (cyclic plastic strain energy)
\( w_e \) Elastic strain energy expended per cycle
\( w_p \) Plastic strain energy expended per cycle
\( W \) Width of a CT specimen
\( \dot{W}_s \) Strain energy density rate
\( Y \) Geometry factor
\( Y_0 \) Half distance between the output voltage leads (in Johnson’s formula)
\( Y_{cc} \) Geometry factor for round tensile specimens with edge chordal cracks
\( Y_{scc} \) Geometry factor for round tensile specimens with edge semi-circular cracks

\( \alpha, \beta \) Damage coefficients in the proposed crack growth model modified with \( \Phi \)
\( \alpha' \) Constant in Ramberg-Osgood equation or Norton’s law
\( \alpha'' \) The Coffin constant
\( \alpha_N \) Constraint factor in Newman’s equation
\( \beta_0 \) Constant denoting the effect of time-independent damage
\( \beta_{\text{oxide}} \) Constant denoting the effect of time dependent oxidation damage
\( \beta_{\text{creep}} \) Constant denoting the effect of time dependent creep damage
\( \varepsilon, \Delta \varepsilon \) Strain and strain range
\( \varepsilon_{ij} \) Strain tensor
\( \varepsilon_{\text{max}}, \varepsilon_{\text{min}} \) Maximum and minimum strain
\( \varepsilon_{\text{in}}, \Delta \varepsilon_{\text{in}} \) Inelastic strain and inelastic strain range
\( \varepsilon_{\text{in}}^{\text{max}}, \varepsilon_{\text{in}}^{\text{min}} \) Maximum and minimum values of the inelastic strain
\( \varepsilon_0 \) Reference strain value (in Ramberg-Osgood type equation)
\( \Delta \varepsilon, \Delta \varepsilon_e, \Delta \varepsilon_p \) Total, elastic and plastic strain range
\( \Delta \varepsilon_{\text{in}} \) Inelastic strain range in each cycle
$\Delta_{\text{cum}}$ Cumulated inelastic strain

$\dot{\varepsilon}_{\text{ss}}$, $\dot{\varepsilon}_0$ Steady-state strain rate and reference strain rate

$\dot{\varepsilon}_c$ Creep strain rate

$\varepsilon_f(\dot{\varepsilon}_c)$ Creep ductility as a function of $\dot{\varepsilon}_c$

$\dot{\varepsilon}_d$ Instantaneous equivalent creep strain rate during the dwell

$\tilde{\varepsilon}_f(\dot{\varepsilon}_c)$ Equivalent creep ductility as a function of $\dot{\varepsilon}_c$

$\Phi$ Microstructural condition parameter

$\Phi_0$ Constant for $\Phi$

$\sigma$, $\Delta\sigma$ Stress and stress range

$\sigma_{ij}$ Stress tensor

$\sigma^\infty$ Remote tension stress

$\sigma'_0$ Reference stress value in Ramberg-Osgood type equation or Norton’s law

$\sigma_0$ Material’s inherent friction strength

$\sigma_B$, $\sigma_F$ Back stress and friction stress

$\sigma_R$ Relaxation stress during the hold period

$\sigma_f$ Flow stress term in Newman’s equation

$\sigma_{\text{hold}}$ Stress at the end of hold period

$\sigma_{\text{hold}}^{\text{cl}}, \sigma_{\text{hold}}^{\text{mic}}$ Stress at the end of hold period at the 1st cycle and midlife cycle

$\sigma_{\text{max}}$, $\sigma_{\text{min}}$ Maximum and minimum stress values

$\sigma_{\text{max}}^{\text{cl}}, \sigma_{\text{min}}^{\text{cl}}$ Maximum and minimum stress values at the 1st cycle

$\sigma_{\text{max}}^{\text{mic}}, \sigma_{\text{min}}^{\text{mic}}$ Maximum and minimum stress values at the midlife cycle

$\sigma_{x}^{\text{max}}, \sigma_{x}^{\text{min}}$ Maximum and Minimum stress values in the linear elastic regime

$\sigma_o$, $\sigma_c$ Opening stress and closure stress

$\sigma_y$ Yield strength

$\sigma_p$, $\sigma_s$, $\sigma_p$ Strengthening from precipitation hardening, solid solution hardening and dislocation hardening

$\Gamma$ Path surrounding the crack tip

$\nu$ Poisson’s ratio

BCC Body centred cubic structure

BCT Body centred tetragonal structure

BSE Backscattering electron

CI Confidence index

COST The Cooperation in Science and Technology program

CT Compact tension (specimen)

CTOD Crack tip opening displacement
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<td>DCPD</td>
<td>Direct current potential drop (technique)</td>
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<td>EBSD</td>
<td>Electron backscattering diffraction</td>
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<td>EBSP</td>
<td>Electron backscattering pattern</td>
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<td>EDM</td>
<td>Electrical discharge machining</td>
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<td>EM</td>
<td>Electron microscopy</td>
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<td>EMF</td>
<td>Electromotive force</td>
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<td>EPFM</td>
<td>Elastoplastic fracture mechanics</td>
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<td>EPRI</td>
<td>The Electric Power Research Institute</td>
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<td>FCC</td>
<td>Face centred cubic structure</td>
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<tr>
<td>IPF</td>
<td>Inverse pole figure</td>
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<td>IQ</td>
<td>Image quality</td>
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<tr>
<td>KAM</td>
<td>Kernel average misorientation</td>
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<tr>
<td>K-S</td>
<td>Kurdjumov-Sachs (orientation relationship in martensitic transformation)</td>
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<td>LCF</td>
<td>Low cycle fatigue</td>
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<td>LEFM</td>
<td>Linear elastic fracture mechanics</td>
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<td>Orientation imaging microscopy</td>
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<td>PSB</td>
<td>Persistent slip band</td>
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<td>RB</td>
<td>Round bar (specimen)</td>
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<td>ScopeM</td>
<td>The Scientific Centre for Optical and Electron Microscopy (ETHZ)</td>
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<td>SE</td>
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<td>SEDF</td>
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<td>SEM</td>
<td>Scanning electron microscopy</td>
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<tr>
<td>SENT</td>
<td>Single edge notched tension (specimen)</td>
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<td>SFE</td>
<td>Stacking fault energy</td>
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<td>TEM</td>
<td>Transmission electron microscopy</td>
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Packet, block, lath and subgrain are microstructural features in martensitic microstructures. Phases $\alpha$, $\alpha'$ and $\gamma$ denote ferrite, martensite and austenite, respectively.
Introduction

Fossil-fuel power plants (typically consisting of steam/gas turbines) have proven to be a reliable source of energy conversion to generate electricity, which currently makes them indispensable in the modern world. However, they are a major contributor to excessive CO₂ emission, which can cause a negative environmental impact (e.g. global warming). Moreover, the fact of only limited resources on this planet has brought about the demand of wiser, more sustainable developments. In view of these challenges, technological innovation in the power generation industry has never stopped.

Based on a fundamental thermodynamic principle, the most direct way to improve the output/efficiency in a thermal cycle is to increase the maximum (inlet) temperature. This concept has been well applied in the power generation industry. For example, in steam turbines currently produced in Europe, the maximum temperature can be as high as 600-620°C (ultra-supercritical), whereas for many years steam turbines typically operated with a steam inlet temperature of 540-565°C (supercritical). The principle reason behind the leap from supercritical to ultra-supercritical is the successful employment of new types of material (e.g. 9-12%Cr martensitic steels).

As the key component to produce rotational motion, turbine rotors normally carry a heavy load in steam turbines. Under high temperature/pressure application, they are mainly subjected to creep and fatigue loading, which can potentially lead to various kinds of failure. It is absolutely crucial to be able to establish a safe, efficient and reliable product defect assessment procedure in the design stage, and to predict the remaining life at any time during operation with high confidence by taking different types of potential defect into consideration. The related topics are of great interest to many organizations and researchers, and there are already relevant standards or codes of practice available as general references, such as a number of ASTM standards, the British R5 and BS 7910 codes, and the RCC-MR French A16 code.¹⁻⁶

The major target in this PhD project is to understand and represent sub-critical short-crack creep-fatigue crack growth behaviour, in particular for two ultra-supercritical candidate rotor steels. One of them is FB2 (9CrMoCoVNbNB), and the other is 10CrMoVNbN (or 10Cr for short). Although the current existing common assessment procedures comprise some of the important aspects related to this study, the value of this PhD project relates to at least three aspects:

1) The materials are two of the most advanced martensitic creep resistant 9-12%Cr steels, whereas the existing short-crack creep-fatigue assessment procedures have been developed mainly for materials of the previous generation. This means that the trend lines and boundary values may not be the same for the newly investigated materials, thus the effectiveness of them is examined.
2) The focus of this study is on the period of short crack development, which takes up most of the component lifetime. However, there are comparatively few codes of practice relating to this topic. It is advantageous that this study can take a step forward to optimize the existing short-crack creep-fatigue crack growth assessment procedure for the advanced martensitic steels.

3) There is not much literature covering the systematic microstructural evolution during creep-fatigue deformation for this type of steel. This study also tries to fill this gap by an extensive campaign of microstructural analysis, and this in-depth examination provides an added value in the understanding of dynamic recovery in this class of steel. More importantly, the effectiveness of suggested crack growth models can further be improved by taking microstructural evolution into consideration.

A round-bar type specimen geometry (with chordal crack starter) for strain controlled short-crack creep-fatigue crack growth tests was adopted in mechanical testing. The main control variables in the experiments were the strain/displacement range, temperature and dwell period, which then led to comprehensive test matrices.

In the subsequent stage of microstructural investigation, traditional optical microscopy on some of the creep-fatigue cracked samples was carried out first, by following the steps of classical sample preparation. Afterwards, a significant effort was made towards characterizing the evolution of martensitic morphology/strengthening sub-structures in these samples at the micro-nano scale by EBSD (electron backscattering diffraction) measurements.

Finally, with the information of mechanical response, crack development and microstructural evolution at hand, it was then possible to validate and establish different kinds of models to describe or predict short crack growth behaviour under creep-fatigue deformation conditions. The effectiveness of these models was compared, and possible adjustments for further improvements were also discussed.

The main body of this PhD thesis is divided into six chapters, which are outlined as follows:

**Chapter 1** introduces some of the basic concepts and background knowledge related to this study. Mechanical aspects such as the typical cyclic response of creep-fatigue loading, short (to long) crack development, creep-fatigue-oxidation interaction, crack closure and crack growth modelling are briefly reviewed at first. After that, microstructural aspects of three related topics (martensitic structure formation, strengthening mechanisms, recovery and recrystallization) are shortly considered. Finally, principles of the two most useful experimental approaches in this study, namely the DCPD (direct current potential drop) technique and the EBSD technique are also addressed.

**Chapter 2** introduces the experimental procedure for short crack growth tests, which includes material details (manufacturer, composition, heat treatment, etc.), creep-fatigue test details (specimen design, test set-up, experimental variables, test matrices, etc.) and microstructural investigation details (sample preparation process, testing facilities, operating parameters, test matrices, etc.).
Chapter 3 covers the major results from short crack growth tests. First, the cyclic stress-strain response is displayed, including the methodology of extracting essential information from the recorded experimental data. Afterwards, the calibration process of DCPD values is elaborated. In this chapter, further discussions on some of the critical issues for short crack development (e.g. strain-life correlation, characteristic crack depth/crack growth rate values, crack closure, DCPD evolution during the dwell and a stress partitioning method) are also presented.

Chapter 4 presents the results from microstructural analysis, which are composed of two parts: optical microscopic/scanning electron microscopic observation and EBSD analysis. The first part focuses on conventional issues, such as martensitic morphology, crack propagation paths, oxides, precipitates, and micro-cracks. More importantly, the EBSD technique enables a closer look on the martensitic sub-structures. Evolution of morphology and misorientation across different boundaries as well as the variation of grain size along the crack propagation direction are carefully assessed and documented.

Chapter 5 reveals the most critical results in this PhD project, which are related to short crack growth modelling. Different models are demonstrated one after another with parallel comparison between the tested materials. Discussions on the pros/cons of these models and the underlying inference with respect to their applicability are also given. By referring back to the microstructural analysis, a modification to the proposed models by incorporating a microstructural condition parameter is proven to be able to best represent short-crack creep-fatigue crack growth behaviour for the investigated materials.

Chapter 6 summarizes the most important findings of this study. Afterwards, the applicability of proposed short crack growth models is reviewed. At the end, suggestions are also given for the unsolved issues and possible future directions of this study.

Finally, Appendix A lists all the short-crack creep-fatigue crack growth models as well as the best-fit values for the investigated materials and test conditions. Appendix B illustrates the compliance check for the tested round bar specimens. Appendix C discusses the factors influencing DCPD variation during dwell.

In the meantime, the author has also participated in the round-robin on long-crack creep-fatigue crack growth testing for the standard ASTM E2760, which serves as a good supplement and acts as the starting point for future study. Details of this part of the work are documented in Appendix D.
Chapter 1

Basic concepts

This chapter reviews the fundamental theories, laws and methods with respect to the determination of mechanical and microstructural properties as well as the modelling of creep-fatigue crack development for advanced martensitic turbine steels. Through this rather broad-covered introductive chapter comprising the most relevant aspects to this study, a better understanding of the background, methodology and subsequent investigation can be achieved.

To be more specific, the first section briefly explains the reason for studying crack growth behaviour for turbine components, the origin of targeted creep-fatigue damage in turbine rotors, and how to simulate the actual operating conditions by means of laboratory tests.

The following section reviews the typical response of stress and strain during such feature tests, which can generally be represented by the evolution of hysteresis loops. In particular, two types of loading scenarios of creep-fatigue experiments are distinguished, in accordance with either primary or secondary loading conditions during practical application.

Section 3 reviews the theories of crack development. The focus is applied on short crack growth behaviour, which is the main concern in this study. The definition of short cracks is introduced first, and the difference between short and long crack development is explained afterwards. The influence of fatigue-creep-oxidation interaction on crack growth behaviour is also covered.

Section 4 summarizes (short) crack growth models utilized in the later analysis in Chapter 5. Starting with the two commonly adopted fracture mechanics based models, two semi-empirical models are also given. Then, an alternative model based on strain energy density factor is presented.

In order to fully comprehend the nature and changes of microstructural features in the investigated materials during creep-fatigue deformation, and how they can be related to macroscopic mechanical properties as well as to crack growth modelling, section 5 provides the necessary background of microstructural aspects for the discussion in Chapter 4.

Finally, section 6 introduces two most important techniques implemented in mechanical and microstructural experiments. A good knowledge of them helps to understand the experimental procedure and result analysis.
1.1 Thermal fatigue in turbine rotors

In the power generation industry, steam turbines play a crucial role in CCPP (Combined-Cycle Power Plant), with the purpose of producing a more flexible electricity supply than conventional single-cycle power plant at a lower cost. This can be greatly beneficial to nature resource conservation as well as to exhaust emission reduction.

In a typical three-stage steam turbine, the hot steam enters and is expanded through the high pressure turbine, gets reheated and travels (and is further expanded) through the intermediate pressure and low pressure turbines to maximize the energy transferred to the turbine rotors.\(^8\) For the sake of a further improvement of thermal efficiency with both economic and environmental advantages, an essential strategy is to develop new materials with improved creep-fatigue properties. Many efforts have been made in developing new materials to accommodate the increased operating temperature, which include the introduction of martensitic creep resistant 9-12\% Cr steels for ultra-supercritical steam turbines.

After the first advanced 9\%Cr steel grade (Grade 91) was developed in the 1980s, new stronger steels have been introduced such as the two main materials investigated in this study, which were the most favoured candidates developed within the collaborative European Cost 501 project started almost thirty years ago.\(^9\) One of them is FB2 (9CrMoCoVNbNB), and the other is 10CrMoVNbN (here referred to as 10Cr for brevity). The performance and characterization of these novel materials at the design operating temperatures need to be evaluated carefully, and therefore it is essential to establish an effective yet less overly-conservative defect assessment procedure for the components manufactured with such materials in new steam turbines.

For high temperature application, damage mechanisms such as creep, fatigue and oxidation are usually the most critical factors to cause life reduction or even total failure of components. Of particular concern is the turbine rotor itself, which commonly possesses a massive weight/thickness and is difficult to replace. One possible damaging situation which can occur in rotors is the excessive surface crack development at critical locations. Hence, of all the loads a rotor is subjected to in operation (thermal transients, self-weight bending, centrifugal loading, pressure load etc.), thermal transients are perhaps the most influential due to repetitive changes in temperature gradient during service duty. This is consistent with the increasing demand for the more flexible production of electricity, which inevitably leads to a larger number of start-up and shut-down cycles.

Although during service the actual cyclic thermal stress-strain evolution of a rotor is complicated, simplified representations can be relatively straightforward to understand.\(^10\),\(^11\) Examples are shown in Figure 1-1, illustrating the difference between two operating protocols on the surface material of rotors. During turbine start-up, a compressive stress is generated at features on the rotor periphery due to the constraint from subsurface bulk materials, to supress
the thermal expansion (OA in Figure 1-1). This thermal expansion can often be restrained by the cooler surroundings, i.e. there is a limit strain value at the compressive ramp (A’ in Figure 1-1). Subsequently, the stress can evolve into residual tension due to reversed plasticity when the temperature ultimately attains a stable distribution throughout the section (AB in Figure 1-1). Depending on the heating rate and temperature value, a relaxation stage can take place at peak strain in compression, i.e. dashed lines in Figure 1-1.\textsuperscript{12, 13}

During steady-state operation, the tensile residual stress undergoes relaxation while the thermal strain remains unchanged (BC in Figure 1-1). During turbine shut-down, two types of cooling should be distinguished, i.e. slow cooling and rapid cooling, depending on the location in the component. Under slow cooling conditions, material on the surface cools at a similar rate as in the bulk, therefore inducing a steady decrease of stress/strain (CO in Figure 1-1a). However, under rapid cooling conditions where the temperature is reduced suddenly, the rotor surface cools down much faster than the interior, resulting in the generation of an additional tensile stress at the rotor surface (the magnitude of this stress depending on the cooling rate, BD in Figure 1-1b. This thermal contraction may be restrained, i.e. at point D’). Finally, the stress gradually reduces to zero at ambient temperature when the turbine is at standstill (DO in Figure 1-1b).\textsuperscript{11, 14, 15}

![Figure 1-1 Typical thermal stress-strain response for the surface material of turbine rotors under: (a) slow cooling condition and (b) rapid cooling condition. (After Ref.\textsuperscript{11, 16})](image)

In both cases, thermal cycling has a constraint on strain. Accordingly these two types of operating cycle are typically represented in the laboratory by means of isothermal strain controlled low cycle fatigue tests with hold time(s) at different positions in the cycle. They may also be referred to as creep-fatigue tests.\textsuperscript{17, 18} Technically, a cyclic test should comprise a time-dependent portion to become a true creep-fatigue experiment. However, in traditional continuous-cycling low cycle fatigue tests, the strain rates are low whereas the temperatures are normally high enough to cause certain creep damage, although it might be limited. In addition, low cycle fatigue tests and true creep-fatigue tests are generally performed in a similar manner in the laboratory.\textsuperscript{7} Hence, the unified term ‘creep-fatigue test’ is adopted in this study, for both low strain rate continuously cycled tests and tests with hold periods.

As already suggested by the name, a creep-fatigue test can involve interactions between different mechanisms such as time-independent elastic-plastic strain, time dependent creep
strain and environmental factors. To fully understand the influence of those factors on cyclic stress-strain response and on accurate life prediction, some background knowledge will be shortly reviewed in the following text.

### 1.2 Cyclic stress-strain response

Cyclic stress-strain response is the most direct output from a creep-fatigue test, which not only gives information about mechanical deformation but also reflects the alteration in microstructure. During a fatigue test, hardening or softening as revealed by the cyclic stress response of the testpiece can appear, depending on the material's original metallurgical condition and its deformation history. For precipitation strengthened alloys at high temperatures, cyclic softening usually happens, with a rapid drop at first and then a steady decrease of the resistance. This is schematically shown in Figure 1-2: in a (total) strain controlled fully reversed continuous-cycling creep-fatigue test, the height of the hysteresis loops is reduced continuously, whereas the maximum and minimum stresses evolve symmetrically. In order to characterize the common cyclic stress-strain response of a certain material at a certain temperature, a steady-state saturated response should be employed. This is normally done by making use of values at the midlife cycle (0.5\(N_f\)) where the rates of change of maximum and minimum stress are at their lowest.

![Figure 1-2 Schematic representation of changes in hysteresis loop shape (left) and maximum/minimum stresses during a creep-fatigue test.](image)

One way to describe the cyclic elastic-plastic strain curve is to use a Ramberg-Osgood type equation (assuming power-law hardening), which is similar to the initial form for the monotonic case:

\[
\Delta \varepsilon_t = \frac{\Delta \sigma}{E} + \left(\frac{\Delta \sigma}{2A'}\right)^{1/n}
\]

where \(\Delta \varepsilon_t\) is the total strain range; \(\Delta \sigma\) is the total stress range; \(E\) is the Young’s modulus; \(n\) is the cyclic strain hardening exponent and \(A’\) is a constant.
For materials exhibiting Masing behaviour, the locus of stabilized hysteresis loops under different stress-strain levels should fall onto a single curve (connection through tips from a series of loops with fully reversed strain ranges), which characterizes the material’s cyclic stress-strain behaviour.\cite{21,23,24,26} The cyclic strain hardening exponent $n$, which reflects the material’s resistance to plastic deformation, can be obtained through this curve. An illustration is shown in Figure 1-3, where stress values are plotted against plastic strain values. Three midlife hysteresis loops are chosen to denote the stable responses for each testing condition, whereas the red dashed line passes through the maximum points perfectly. The cyclic strain hardening exponent can then be obtained for this material at this defined temperature.

![Figure 1-3 Stress-plastic strain midlife hysteresis loops and cyclic stress-strain curve showing the Masing behaviour.](image)

$$\Delta\sigma = A\left(\Delta\varepsilon_p\right)^n$$ \hspace{1cm} (1-2)

Here $n$ is the cyclic strain hardening exponent and $A$ is a material dependent coefficient.

In order to simulate practical operating conditions, generally two situations can be distinguished, regarding the involved dwell periods in laboratory experiments. Depending on the loading conditions, it is possible to attribute the creep damage to either primary (directly applied) loading, or to secondary (self-equilibrating) loading.

The laboratory representation for the first situation imitates steady-state operation with mainly primary loading (corresponding to Figure 1-4). A moderate constant maximum force normally leads to a small strain (usually in the elastic regime). This can generate a relatively more pronounced effect of dwell period (creep damage accumulation) compared with the fast ramping period. This type of stress-strain response is illustrated in Figure 1-4, with o-a-b-o showing the path of a single loop. Note that the abscissa is LLD (load line displacement), which is different but analogous to strain. As depicted in this figure, the ramping parts o-a and b-o are predominantly linear elastic (strictly speaking, the lower part is nonlinear due to the loading state and specimen geometry); whereas during the dwell period a-b, LLD starts to accumulate as a result of inelastic deformation, which cannot be fully recovered under a positive load ratio.
1.2 Cyclic stress-strain response

Therefore, along with an ongoing test, the position of the loops shifts to the right side owing to the accumulated LLD (ratchetting due to permanent creep strain accumulation). In fact, the compliance of the testpiece (slopes of o-a, b-o) also changes during test (mainly due to a propagating crack and the inelastic deformation). This type of operating cycle is commonly approached by long crack growth experiments, which will be explained in more details in Appendix D.

![Figure 1-4](image1.png)

**Figure 1-4** Stress-strain response in a stress-controlled creep-fatigue test with hold time in tension.

The laboratory representation for the second situation imitates a whole operating cycle subjected to the thermal transients (corresponding to Figure 1-5), where the total displacement is constrained (i.e. strain controlled ramping/relaxation). The hold time is included at peak loading state (where the strain is fixed at maximum), corresponding to the slow cooling case in Figure 1-1. A schematic representation is shown in Figure 1-5. In practice the hold time could be anywhere in the loop.

![Figure 1-5](image2.png)

**Figure 1-5** Stress-strain response in a strain-controlled creep-fatigue test with hold time in tension.

In a single cycle, the evolving path is o-a-b-o, which can be decomposed into three stages. During ramping up (o-a), the stress rises in a decelerating manner due to the accumulation of inelastic strain; during the hold time (a-b), stress relaxation occurs, leading to an initial quick descent and gradually approaches apparent saturation. Meanwhile, the inelastic strain (creep strain) slightly increases, depending on the magnitude of relaxation. During ramping down (b-o), stress and inelastic strain values get back to almost the starting values.
It should be pointed out that the isothermal testing condition is a simplified condition, whereas in reality the temperature also changes during the start-up and shut-down processes. It can be reasonably expected that the heaviest creep-fatigue damage occurs at the highest service temperature, thus leading to the shortest component life time.\textsuperscript{27} In the type of creep-fatigue test involved in this study (effectively cyclic relaxation tests, Figure 1-5), the contribution of creep deformation is mainly introduced through the hold periods, rather than during ramping.

\section{1.3 Crack development}

As a consequence of creep-fatigue deformation, cracks may initiate and develop to a size which is critical for the component/specimen life. When cracks are present in a structure, one approach for determining failure is by a predetermined maximum tolerant crack size, or a limited crack growth rate (damage tolerant approach).\textsuperscript{23} In any case, where and how the crack develops is a key issue in life assessment. Depending on the investigated length scale, the distinction between short crack propagation and long crack propagation is first introduced. One of the possible explanations for the abnormal crack growth behaviour of short cracks, i.e. crack closure, is then examined. In contrast to pure fatigue crack development, the interaction between fatigue, creep and oxidation generates additional features in practical circumstances, which are also briefly covered in the third and fourth parts of this section.

\subsection{1.3.1 Definition of short and long cracks}

In an idealized case, a natural fatigue crack usually starts from the surface, at a certain angle to its loading axis. This is due to the repeated intrusion and extrusion of persistent slip bands (PSB).\textsuperscript{28} The time for this crack nucleation depends not only on the stress-strain state, but also on other factors such as surface condition, microstructural condition and geometry condition (schematically plotted as red curves in Figure 1-6). In reality, a component contains flaws/defects, in addition to the geometrically stress-concentrated areas and harsh environment. Therefore, it might be legitimate to assume that the crack initiation sites already exist, and the focus is on how fast those cracks will propagate in this study.

During crack propagation, the direction changes from parallel to the maximum shear stress to perpendicular to the loading direction. Depending on the depth of the crack, it can be regarded as a microcrack/short crack (blue curves in Figure 1-6), or a long crack (black curves in Figure 1-6). Under a typical cyclic loading condition, it takes comparably longer time for the microcrack/short crack development, whereas the time for long cracks to fracture can be relatively short.\textsuperscript{28}
Linear elastic fracture mechanics (LEFM) was first adopted to describe the stress-strain fields of cracks in an isotropic material, and was found effective only for relatively long cracks. When the crack growth rate is correlated with stress intensity factor range $\Delta K$, small cracks often exhibit a large yet decreasing propagation rate when $\Delta K$ is under the threshold value determined in long crack tests. Only when these cracks have developed into long cracks, their growth rates finally converge with those from long cracks (also see Figure 1-12). This presumably indicates that $\Delta K$ is not the appropriate correlating parameter. This anomaly led to the development of theories concerning short cracks, where the mechanical and microstructural conditions are quite different from long cracks. This certainly has its physical significance, as short cracks can be of major importance for high temperature structures, since a large portion of their service life will be contained within the short crack regime.

As proposed by Suresh and Ritchie, the short crack regime can be divided into three parts: microstructurally short cracks, mechanically short cracks and physically short cracks (some other researches have similar but slightly different definitions), as conceptually shown in Figure 1-6. In the first part, the size of the crack is comparable to a characteristic microstructural feature, i.e. grain size. Due to the interactions with local obstacles such as grain boundaries and precipitates, crack propagation decelerates and accelerates alternately. When the crack length is larger than the size of several grains, the influence of microstructure is reduced, and the crack path changes from parallel to the maximum shear stress to perpendicular to the loading direction. Cracks at this moment are called mechanically short cracks. The third stage, the physically short crack regime, is a transitional phase from short crack to long crack, where the influence of plastic zone size ahead of the crack tip starts to fade. At the end of this stage, plastic zone size is finally not comparable to the propagated crack size (also see Figure 1-7), thus the theory of LEFM is valid. However, there is no clear distinction between all these stages (some regimes can overlap), and sometimes a short crack is simply defined as a crack that is ‘physically’ small, i.e. small compared to the dimensions of the testpiece/structure.
far as this study is concerned, a more appropriate definition is with respect to the size of the cyclic plastic zone.

Figure 1-7 shows the significance of distinguishing between short and long crack growth behaviour in a service-like component with a notch (e.g. a groove on a surface). When the crack depth is relatively small, it is fully embedded in the plastic zone. The corresponding laboratory reproduction is usually by cylindrical specimen with reversed elastic-plastic loading, with a smooth or slightly notched surface (Figure 1-7a) to represent fatigue crack initiation in the real component. Crack growth behaviour in this kind of experiment does not always stand for short crack, although the testpiece diameter is smaller than the gross plastic zone. This is because after certain propagation, the crack size is no longer ‘physically’ small compared with the uncracked ligament, whereas the normally adopted engineering stress-strain state deviates from the designed values. (The specimen compliance will significantly increase when the crack has propagated longer). One possible criterion to define short cracks is when the crack depth is smaller than about a quarter of the diameter in a feature specimen, i.e. $a / D \leq 0.25$. \cite{7,35,37} (Technically, even if the crack has propagated further through the cylindrical specimen, e.g. beyond 25% of the diameter, the growth behaviour will be different from that in a traditional long crack growth test with a CT specimen: the driving forces are not the same). In practice, such criteria could not be directly applied to a turbine rotor. Consequently, absolute crack size criteria are usually adopted.

When the crack has penetrated far enough beyond the (cyclic) plastic zone in the component, CT specimens are commonly adopted to assess crack growth behaviour at this stage (Figure 1-7b). The applied cyclic load is mainly within the elastic regime, whereas the generated plastic zone at the crack tip is very small compared to the crack depth.
1.3 Crack development

1.3.2 General concept of crack closure

Apart from the interaction with microstructural features and the role of crack tip plastic zone size, the effect of crack closure is another possible contributory factor associated with the abnormal growth behaviour of short crack growth, compared with traditional long crack growth.

Crack closure, which refers to the premature contact between coupling cracked surfaces, reduces the effective load range for which the crack tip is actually open. The mechanisms relevant to this study can be categorized into three origins: plasticity induced, roughness induced and oxidation (corrosion product) induced.\textsuperscript{29, 36, 38, 39} Figure 1-8 shows these three mechanisms.

Although the remote stress-strain state is in the elastic regime, the material located near the crack tip is usually subjected to plastic deformation (small-scale yielding). Along with increasing crack length, the size of the cyclic plastic zone ahead of the crack is also growing (shown in Figure 1-8a). During decohesion of the crack surface, the plastically deformed material at the previous crack tip (which is now broken open) can release its elastic loading, but the remaining residual inelastic deformation still exists. This results in a larger plastic wake than the idealized situation (elastic, sharp crack tip), which causes earlier crack closure upon unloading.

The second mechanism is mainly due to the roughness between contacting fracture surfaces, since the crack route often exhibits a wavy profile such that small displacements exist. When the crack closes, those asymmetric crack surfaces start to contact first, rather than the true crack tip (geometric mismatch, Figure 1-8b).

The third mechanism originates from oxidation or corrosion effects. Under high temperature conditions, Cr-rich oxide scale is produced on the surface as well as along crack paths (in fact,
the freshly opened crack tip is more susceptible to oxidation). This oxide layer effectively increases the volume of the fracture surface layers, which make contact before the true crack tip (Figure 1-8c). Even if some of the oxides are smashed during the compressive loading, the remaining debris still contributes to the closure effect.

All those mechanisms build up as short cracks propagate to become long cracks, which generally lead to a reduced effective load range (the first mechanism is dominant at larger crack lengths where CTOD is in the order of micrometres; whereas the other two are dominant at smaller crack lengths where CTOD is in the order of nanometres). This theory successfully explains the faster propagation behaviour of short cracks (when related to the same mechanical correlating parameter). The degree of crack closure is normally estimated by the remote load-displacement history (change of curvature on load versus displacement hysteresis loops), or by CTOD measurement.

![Figure 1-9 Schematic representation of typical hysteresis loop with crack closure effect.](image)

For a short-crack creep-fatigue crack growth test (bulk plastic deformation, strain controlled), the crack closure stress on the compressive-going ramp is usually closer to the minimum load than the crack opening stress on the tensile-going ramp. This is because the large residual tensile plastic deformation during previous ramping-up must be reversed at first, before crack closure can happen (the unloading process is mainly elastic at the beginning).

For a long-crack creep-fatigue crack growth test (small-scale yielding, stress controlled), the crack closure stress is more or less the same as the crack opening stress. This is because the ramping period is mainly in the elastic regime, and compliance values hardly change during a single cycle.

An example of a hysteresis loop showing a significant crack closure effect is displayed in Figure 1-9. In such a total strain controlled test, the maximum and minimum strain values are symmetrical, whereas the minimum (engineering) stress has a larger magnitude than maximum (engineering) stress when crack closure is present. (Note that the stress and strain values are nominal values, i.e. without considering the reduction of the area in the minimum
1.3 Crack development

cross-section. This terminology is strictly speaking not precise enough, but is widely adopted in practice).

In fact, cusps near the compressive peak stress can normally appear when cracks have developed beyond 25% of the cross-section area of the testpieces. For short crack growth tests, this usually means that the crack is already too deep to be a short crack.27

1.3.3 Creep-fatigue interaction

As introduced in section 1.1, steam turbine materials are frequently subjected to creep-fatigue loading during operation. Evaluation of creep-fatigue interaction is important for the failure/fitness assessment, because the superimposed cyclic deformation and creep/relaxation may lead to more critical damage accumulation (e.g. the enhanced crack propagation behaviour as shown later in this thesis).

Creep-fatigue interaction processes are inevitably complex, which are not only dependent on the properties of the investigated materials (e.g. creep ductility and creep strength), but also dependent on the testing parameters (e.g. temperature, stress or strain range/rate, hold periods and the environment).43, 44 Moreover, it may be essential to take the stability/evolution of microstructure into consideration, especially for the materials involved in this study (i.e. 9%-10%Cr martensitic creep resistant steels). It has been reported that apart from a high creep rupture strength, the creep damage characteristics of this type of steels can be different from conventional steam turbine materials, mainly owing to the distinctive tempered martensitic substructures.45, 46 For example, it was found that micron-size creep cavitation normally occurs only late in creep life. In addition, small cavities can be nucleated at inter-lath boundaries without excessive cavity growth.47, 48

Therefore, this section begins with a generalized description of creep-fatigue interaction with regard to the cracking mechanisms. Afterwards, the featured deformation-induced microstructural degradation in martensitic creep resistant steels is shortly explained.

Generalized creep-fatigue cracking mechanisms

In ductile materials, cracks can commonly initiate as a consequence of five mechanisms: (1) cyclic slip; (2) local oxide or corrosion product rupture; (3) void formation at second-phase particles; (4) grain boundary sliding (to form wedge cracks) and (5) grain boundary cavitation.36 In the first two cases, fatigue cracks can develop from the surface and, upon further loading, penetrate deeper into the subsurface. In contrast, deformation at elevated temperatures can cause local stress concentrations at internal irregularities/weak internal interfaces, which are responsible for the nucleation of voids/cavities or micro cracks. Subsequent growth/coalescence of these defects may lead to transgranular or intergranular fracture, which is influenced by the interaction between creep and fatigue.49, 50
Depending on the creep-fatigue testing conditions (as well as the material), crack initiation and growth can be fatigue-dominated, or creep-dominated, or due to creep-fatigue interaction, leading to an increased crack development rate.\textsuperscript{12,51-54} Schematic illustrations of four generic creep-fatigue cracking mechanisms are sketched in Figure 1-10.

![Schematic representation of creep-fatigue failure modes](image)

**Figure 1-10** Schematic representation of creep-fatigue failure modes: (a) fatigue-dominated case; (b) creep-dominated case; (c) creep-fatigue interaction showing consequential creep damage and (d) creep-fatigue interaction showing simultaneous creep damage. (After Ref.\textsuperscript{12})

Under creep-fatigue loading conditions at high strain rates/ranges without significant dwell periods, cracks often initiate from the surface and grow from short cracks to long cracks in a transgranular profile into the bulk material, representing fatigue-dominated cracking (i.e. Figure 1-10a). This crack propagation mechanism is similar to that in traditional low cycle fatigue tests, which is most probably due to irreversible dislocation motion at the crack tip.\textsuperscript{23,55}

In contrast, with long hold time involved and/or at smaller strain rates/ranges or at higher temperatures, creep cavities may initiate typically at grain boundaries. Usually, internal creep damage can develop on microstructural inhomogeneities, either as cavities on grain boundaries, or as wedge cracking at triple junctions, or as voids adjacent to particles in the matrix resulting from local stress concentrations. With on-going external force those defects may grow owing to the thermally activated diffusion process, or to the continuous dislocation movement. Strings of cavities may occupy a large fraction and form microcracks along grain boundaries, which are referred to as intergranular cracks.\textsuperscript{56-58} The accumulation of such
1.3 Crack development

damage, i.e. the nucleation, growth, and subsequent linkage of cavities can lead to a creep-dominated intergranular profile, as shown in Figure 1-10b. Finally, the coalescence of several internal microcracks may result in a merged major crack extending to the surface, by continuously absorbing adjacent cracked grain boundaries. During this process, the component may no longer be functional, either because of the loss of load bearing at net section ligament or because of the excessive deformation due to widespread creep.

Under intermediate conditions (e.g. intermediate strain range or hold time), two types of creep-fatigue cracking mechanisms can be distinguished: transgranular fatigue crack initiation followed by intergranular growth (i.e. consequential creep damage, Figure 1-10c) and simultaneous transgranular/intergranular initiation and growth (i.e. simultaneous creep and fatigue damage generation, Figure 1-10d).

Once a transgranular short crack propagates deeper into the material, the stress-strain state at the crack tip becomes more critical due to local stress concentration. At grain boundaries adjacent to the crack tip, cavities/microcracks are more likely to initiate due to boundary sliding/dislocation arrangement, e.g. during the hold periods. (It is found that backward dislocation motion and the transition of strain hardening mechanisms are the main reasons for cyclic creep acceleration). Therefore, further crack propagation may take place in those damaged grain boundaries, and the cracking profile transfers from transgranular to at least partly intergranular (e.g. Figure 1-10c). In addition, when the crack tip is exposed to the air, it is likely that oxidation effects will cause the loss of carbide precipitates on the grain boundary, due to the outflow of elemental Cr (i.e. oxygen reacts with M23C6 precipitates). This kind of weakened/embrittled grain boundaries is more prone to crack propagation.

On the other hand, when creep cavities/microcracks are finely distributed in the material (e.g. due to high temperatures or long hold periods), it is likely that the major fatigue crack will meet some of those already existing cracked grain boundaries on the way. In this condition, the crack propagates preferentially at these damaged boundaries. This certainly leads to a faster propagation of the fatigue crack, and the crack path changes from transgranular to intergranular (e.g. Figure 1-10d).

It should be noted that the above classification illustrates only the conventional cracking mechanisms under creep-fatigue deformation conditions (i.e. Figure 1-10a to d). In reality, for example, the dependence on creep ductility of the tested material has a big influence on creep-fatigue interaction. Creep cavities tend to form at grain boundaries in less creep-ductile materials, whereas creep voids tend to form at matrix-inclusion interfaces in highly creep-ductile materials.

In tempered martensitic steels

For the investigated materials in this study (i.e. 9%Cr and 10%Cr martensitic creep resistant steels), the development of creep damage is not necessarily the same as the classical interpretation. As briefly introduced in section 1.5.1, tempered martensites are composed of
organized sub-structures (i.e. packets, blocks, laths and subgrains). Strengthening comes from the dense boundaries/dislocation networks, the pinning effect from finely distributed precipitates and the presence of solid solution hardening as well as the interaction with each other (more details can be found in section 1.5.2). Microstructural changes are of great importance to martensitic steels, because the loss of effectiveness of these barriers can strongly affect the deformation condition, leading to a change in creep properties of the material.\textsuperscript{44, 48, 64-66} Therefore, a comprehensive investigation of the microstructural evolution during deformation at elevated temperatures is necessary, as demonstrated in Chapter 4 in this thesis.

Depending on the testing conditions (i.e. extent of creep-fatigue interaction), an important softening mechanism is dynamic recovery. In particular, the initial tempered lath martensite structures develop to a larger and more equiaxed sub-structure, associated with the decrease of dislocation density and the increase of subgrain size (more details on recovery can be found in section 1.5.3). When creep loading is more dominant, e.g. after thousands of hours at practical application temperatures, the coarsening of fine particles may also take place and deteriorate the creep resistance.

When cyclic loading is absent, significant recovery shows up normally after the onset of tertiary creep (i.e. fine lath/subgrain structures remain relatively unchanged in the early stages).\textsuperscript{64} However, when cyclic loading is present, the comparably faster-recovered microstructure reduces the material's resistance to creep deformation and consequently shortens creep rupture time. This behaviour is considered to be typical for cyclic softening materials, e.g. the materials adopted in this study.\textsuperscript{44} Consequently, it is recommended to use a material's creep properties after cyclic deformation to estimate the remaining component life (i.e. taking microstructural changes into account), to avoid premature failure in long term creep.

At the same time it should also be noted that strain-controlled cavity/void nucleation may occur during creep-fatigue deformation, and the preferential sites are not limited to prior austenitic grain boundaries. On the one hand, tempered martensitic steels consist of all sorts of boundaries associated with second-phase particles (e.g. $M_\text{23}C_6$).\textsuperscript{45} Due to high local stress concentration at these geometrical irregularities, small inter-lath cavities can often be observed. On the other hand, for such highly creep-ductile steels, particle-matrix decohesion may take place in the bulk material (e.g. due to high plastic strain in the vicinity of carbide particles).\textsuperscript{43} Therefore, the appearance of creep damage is not necessarily intergranular (e.g. strings of cavities on interior sub-boundaries can visually exhibit a transgranular profile). In this sense, the traditional way of separating the contributions of fatigue and creep in terms of percentage intergranular fracture on the cracking profile is not entirely appropriate for the martensitic 9-10%Cr steels.

In practice, the change of material's deformation condition and the occurrence of cavities/decohesion may take place simultaneously or separately, depending on the creep-fatigue loading. In either case, creep-fatigue interaction usually generates more critical conditions to facilitate crack development than pure fatigue, or pure creep.
1.3 Crack development

General idea of damage summation

Under creep-fatigue loading, both creep damage accumulation and a change of material microstructure/deformation condition may occur at the same time. According to the loading scheme in laboratory representations as illustrated in section 1.2, the contribution of creep loading during hold times to crack development can at least be twofold: (1) accelerated fatigue crack growth during ramping; and (2) creep crack growth during dwell. Further assessments require appropriate crack growth models, e.g. those for creep-fatigue short and long crack development (with respect to the crack depth and cyclic plastic zone size, see section 1.3.1).59

A simple yet widely used method is to evaluate creep crack growth during dwell and fatigue crack growth during cycles separately, and to sum them up to estimate total crack growth rate:67-70

\[
\frac{da}{dN}_{\text{total}} = \frac{da}{dN}_{\text{fatigue}} + \frac{da}{dN}_{\text{creep}}
\]

(1-3)

However, the total (creep-fatigue) crack growth rate may not simply be the linear summation of separate constituents. As discussed above, creep cavitation can influence fatigue crack propagation, and conversely, fatigue loading can influence creep cracking behaviour. Therefore, additional enhancement terms should be applied to the basic crack growth parts to account for the interaction (in a non-linear way). For example, the amount of physical damage such as creep cavities, cracked boundaries can be adopted as the index of creep damage accumulation during creep-fatigue loading.51, 61, 71, 72

As shown later in Chapter 4, no obvious voids/microcracks could be identified on prior austenite grain boundaries for the creep-fatigue deformed martensitic steel specimens examined in this study (the details concerning materials and tests can be found in section 2.1). This was at least partly due to the complex martensitic structures, i.e. hierarchic sub-boundaries. More importantly, a significant change of the microstructural condition was observed, which signifies an alternative mechanism of creep-fatigue interaction for this type of steel, i.e. change of material’s resistance to further deformation and crack propagation. As will be elaborated through the following chapters, this study has indicated that the enhancement of total crack growth rate could be caused by the effects of prior creep and strain enhanced oxidation damage ahead of the crack tip during previous hold times. More details of short-crack creep-fatigue crack growth modelling can be found in Chapter 5.

1.3.4 Oxidation interaction

In reality, turbine rotors are operated within a steam environment, which can be close to, or even more hazardous than ambient air. Generally, the gaseous environment is considered to be harmful, to reduce the fatigue life of metals, such as low carbon high strength steels. By comparing the results of experiments conducted under vacuum and those under ambient air conditions, the effect of air/oxygen can normally be explained by a typical \( da / dN - \Delta K \) curve
(which is schematically shown in section 1.4): (1) the threshold $K$ value changes for tests under vacuum, and (2) the absolute crack growth rate in the Paris regime (defined in Figure 1-12) is lower for tests under vacuum than those in air.\textsuperscript{73} Examples for a 9Cr1Mo steel can be found in the literature, which also elucidated the comprehensive influence of load ratio (i.e. the minimum load divided by the maximum load), temperature, crack closure on the magnitude on this environmental effect.\textsuperscript{74, 75}

On the one hand, the presence of oxide wedging can sometime slow the crack propagation process (e.g. Cotterill found that near-threshold growth rates in vacuum are higher than those in air at 525°C, as a consequence of oxide-induced crack closure).\textsuperscript{74, 76} On the other hand, in low cycle fatigue tests the oxidation interaction often leads to an oxidation-assisted fatigue crack development. Two possible explanation are the oxide-induced slip irreversibility and metal removal by oxidation. During the formation of intrusions and extrusions, oxygen can react with the freshly exposed metal surface at the crack tip, and/or cause dislocation tangling to hinder the reversibility of the slip process.\textsuperscript{77} This is shown in Figure 1-11a, with red lines denoting oxide scales on the surface. Under typical low cycle fatigue conditions, it is likely that crack tip blunting takes place together with an oxide layer build up on the virgin metals, as displayed in Figure 1-11b. The growth rate of this layer through diffusion decreases parabolically with time.\textsuperscript{78, 79} During cyclic deformation, these oxide scales are continually broken and re-formed owing to repeated oxide fracture at crack opening.\textsuperscript{80} In other words, in each cycle the crack can propagate further due to the destruction of oxides and removal of metal.\textsuperscript{81, 82} Therefore, owing to this interaction with oxidation, cracks can propagate faster than under pure fatigue loading conditions.

![Figure 1-11 Illustration of the effect of oxidation on fatigue crack growth: (a) oxide induced irreversibility of slip bands (b) oxide induced metal removal at the crack tip. (After Ref.\textsuperscript{73, 78, 82})](image)

In particular, the mechanisms of oxidation were found to be strongly dependent on the temperature, environment, strain range, test duration and the chemical composition in creep resistant ferritic/martensitic steels.\textsuperscript{76, 83-36} For example, Khanna et al. examined the oxidation behaviour of several 9Cr-1Mo steels and observed that the one with the highest Silicon contents had the best oxidation resistance. They also discovered an ‘inversion’ in oxidation rates (i.e. for the tested 9Cr-1Mo steels the oxidation rates were lower at 700°C than at 600°C)
1.3 Crack development

and a Cr-depletion mechanism proposed as the explanation.\(^8^4\) This abnormal temperature dependence of oxidation kinetics was also noticed by Zurek et al. for a number of 9-12%Cr steels, which was concluded to be due to the formation of a protective oxidized layer with higher Cr contents.\(^8^5\) Quadakkers et al. focused on the breakdown process of the oxide layers. He discovered that for 9-12%Cr steels, generally two oxidized layers could develop in the temperature range between 600°C and 650°C (in a steam environment), separated by a small gap. The breakdown of the initial (inner) protective layer (comprising of (Fe,Cr)\(_2\)O\(_3\) and/or Cr-rich (Fe,Cr)\(_3\)O\(_4\), covered by a hematite layer) took place under long time exposure, while a rapid formation/growing of thick magnetite scales occurred.\(^8^6\) Fournier et al. discovered that the morphology of oxide layers in cyclic tests could be different from the usual duplex layers in static oxidation tests. Two of their observations were: (1) a greater number of successive layers, i.e. Fe-Cr spinel and magnetite were formed alternatively. Repeated cracking of these localized oxide layers could induce a faster crack propagation. In this process, oxygen could be transported through the micro-cracks in oxides to further react with the base material. (2) in heavily oxidized testpieces, oxygen might penetrate along the microstructural boundaries (i.e. sub-boundaries, see section 1.5.1). Slip bands might not necessarily be the easiest cracking path, because cracks could preferentially propagate through the damaged sub-boundaries.\(^8^7\)-\(^8^8\)

As far as this study is concerned, another contributing factor is time dependent creep loading (or to be more exact, relaxation), which is evident during the hold periods at either peak strain in tension or compression. A general conception is that crack propagation can be accelerated due to a longer time of exposure to oxidation during creep/relaxation.\(^7^9\), \(^8^9\)-\(^9^3\) Although it is difficult to separate the contribution of oxidation from that of creep, comparative experiments under vacuum and in air with the same dwell periods have shown that creep-fatigue-oxidation interactions are of great significance to the acceleration of crack propagation. For example, Hecht systematically studied the influence of oxidation and dwell periods on the number of cycles to failure through strain controlled creep-fatigue tests. He found that for the 9Cr1Mo steel, compressive-hold tests failed earlier irrespective of the environment.\(^9^4\) Sugiura showed that for a modified 9Cr1Mo steel, creep-fatigue tests in air with a 600s compressive hold failed invariably earlier than those with a 600s tensile hold at 600°C.\(^9^5\) Kim and Weertman's finding on a modified 9Cr1Mo alloy also demonstrated compressive dwell sensitivity in air (but tensile dwell sensitivity in vacuum).\(^9^1\)

This was consistent with the findings of other researchers that for 9-12%Cr heat resistant steels, that compressive dwell can be more detrimental than tensile dwell.\(^9^6\)-\(^9^8\) Two main possible explanations have been proposed to account for the more damaging effect of the compressive hold.

The first explanation is based on the environmental interaction with oxygen and represents mainly the crack initiation stage. During compressive dwell, an oxide layer can be formed on the smooth surface and notch root of the specimen. Upon subsequent tensile loading, this relatively weak oxide layer is likely to be stretched open and can serve as sites for crack initiation (whereas in the compressive ramp after a tensile dwell, the removal of oxide spalls does not facilitate crack initiation much).\(^9^1\), \(^9^6\), \(^9^9\) This mechanism significantly accelerates the
initiation of a fatigue crack, more than the traditional mechanism coincident with intrusion/extrusion formation due to the generation of persistent slip bands (PSBs). (Another theory for 9Cr1Mo steel suggests that during tensile dwells crack tip blunting appears due to the formation of surface oxide coating, while the applied effective stress/strain range at the crack tip is much higher during compressive dwell).98

The second mechanism is based on the mean stress effect and represents mainly the crack propagation stage. Due to stress relaxation during the hold period, a tensile dwell generates a negative mean stress shift whereas a compressive dwell generates a positive mean stress shift, corresponding to a larger tendency to crack opening. In addition, 9-12%Cr steels are more resistant to creep/void formation compared with low alloy steels. Therefore, tensile dwell is normally reported to be not as influential as compressive dwell in creep-fatigue deformation for this class of steel.98-100

In all, when considering the influence of creep and oxidation (which corresponds to the steady-state running period in service), crack growth behaviour can be much more complex than pure fatigue crack development. The traditional way of predicting crack propagation (i.e. assuming LEFM) may not be sufficient under these conditions, especially for short crack development where the stress-strain state is also much different from the long crack situation. Therefore, adequate models should be established, taking oxidation/creep crack growth parameters also into consideration. (Although the influence of oxidation on fatigue crack growth is well acknowledged, few publications can be found to explicitly incorporate variables associated with oxidation into crack growth models for 9-12%Cr martensitic steels).73, 101

1.4 Life prediction and crack growth models

It is indeed possible to estimate creep or fatigue failure by a series of life curves, i.e. the maximum cycle number or time a component can sustain under defined stress/strain state. For example, the S-N curve/Wöhler curve which is mainly used under elastic fatigue loading; the Coffin-Manson plot which is employed under elastic-plastic fatigue loading; and the Larson-Miller curve which depicts creep rupture behaviour under the influence of stress and temperature.

Nevertheless, this life time is actually an integration of different stages, where the driving forces/ crack propagation mechanisms are different. The intermediate states during deformation cannot be fully revealed by such life curves. Therefore, crack growth laws have been developed within the scope of fracture mechanics, to give a more precise prediction of the growing cracks in damage tolerant design. However, due to the inherent complexity of creep-fatigue interaction especially for the short crack regime, pure fracture mechanics based models may yield less satisfying results. Hence, some empirical models have also been devised, which can be readily applied to practical usage.
In this section, different crack growth models relevant to this study will be shortly introduced. They are: two fatigue crack growth models, i.e. $da/dN$ correlated with stress intensity factor $K$ (based on LEFM) and $da/dN$ correlated with cyclic $J$ integral (based on EPFM); two semi-empirical models (the Tomkins model and the Skelton model); and finally the model with SEDF (strain energy density factor). Some modifications of these models to specifically incorporate creep contribution are also included.

1.4.1 Fracture mechanics based models

When the investigated materials contain cracks, traditional ‘strength based’ criteria for structural design are not sufficient. The early work of Griffith (later developed by Irwin and Orowan) targeted this problem by using an energy criterion approach, i.e. cracks start to propagate when the provided energy is large enough to overcome a material’s resistance to elastic deformation and energy dissipation in surface creation and plastic deformation. In practice, an essentially equivalent approach, i.e. the stress-intensity approach is perhaps more often used. It examines the stress distribution condition directly near the crack tip by the stress intensity factor $K$, assuming the concept of similitude (i.e. a concept to ‘scale’ the conditions and status of laboratory testing specimens to real components. Thus, the application of the model is not restricted to an identical crack size or geometry). Adopting this parameter in the current study is the starting point for creep-fatigue crack growth modelling.

Stress intensity factor $K$

Stress intensity factor $K$ is a commonly adopted parameter to indicate the driving force for crack development under LEFM conditions, especially for the long crack regime. The widely accepted Paris law depicts the continuum crack growth regime (linear part in a double-logarithmic plot), which is in the form of:

$$\frac{da}{dN} = A_K (\Delta K)^{m_K}$$ (1-4)

where $a$ is crack depth, $A_K$ and $m_K$ are material dependent constants, and

$$\Delta K = K_{max} - K_{min}$$ (1-5)

$$K = Y \sigma \sqrt{\pi a}$$ (1-6)

$K_{max}$, $K_{min}$ and $\Delta K$ are the maximum, minimum and the range of stress intensity factor, respectively; $Y$ is the geometry factor. A typical sigmoidal plot showing the variation of fatigue crack growth rate with $\Delta K$ (for long crack growth) is displayed in Figure 1-12.
Three stages can normally be distinguished, namely regimes A, B and C. In Regime A (non-continuum stage) discreet crack growth occurs, where the effects of microstructure, mean stress and environment on fatigue crack growth are large. In Regime B (continuum stage, Paris regime), these influences become less significant, and plastic zone size at the crack tip is relatively small compared to the crack depth. In Regime C (unstable stage), fast rupture appears (static fracture). For LEFM conditions, \(\Delta K\) is equally good in all regimes.

As briefly introduced in section 1.3.1, when the crack is small, the \(K\) similitude concept breaks down. This is also illustrated in Figure 1-12 by red dashed arrows. The reason for this ‘abnormal’ crack propagation even under the long crack threshold value may come from: 1. Microstructural aspects (i.e. continuum mechanics is not valid when the crack depth is of the order of the grain size); 2. Mechanical aspects (i.e. small-scale yielding conditions are not fulfilled, and crack closure is not fully developed); and 3. Chemical aspects (i.e. different environmental conditions at the crack tip). In any case, more appropriate correlating parameters rather than \(\Delta K\) can be adopted to describe short crack growth behaviour, e.g. \(\Delta K_{eq}\), \(\Delta J\), or \(\Delta J_p\).

Crack closure can reduce the effectiveness of total stress intensity factor range \(\Delta K\) to a smaller value \(\Delta K_{eff}\), where

\[
\Delta K_{eff} = K_{max} - K_{open} = q_o \Delta K = q_o (K_{max} - K_{min})
\]  

\(K_{open}\) is the stress intensity factors at the crack opening point (corresponds to the position of \(\sigma_o\) in Figure 1-9); \(q_o\) is the fraction of the total load range for which a crack opens. Under ordinary circumstances the opening point (\(K_{open}, q_o\)) should be used, based on the assumption that the crack develops only when the crack is open. However, one complication is that the opening points are sometimes too obscure to identify on hysteresis loops, whereas the closure points are still easily discernible. This will be explained in more details in Chapter 3.
1.4 Life prediction and crack growth models

In strain controlled creep-fatigue testing conditions, plastic deformation is often too significant to bypass. One proposed modified $K$ type parameter is the equivalent stress intensity factor (range). \(^{(35, 112)}\)

\[
\Delta K_{eq} = (E\Delta \varepsilon_p + q_0 \Delta \sigma)Y \sqrt{\pi a}
\]  

(1-8)

In this formula, $\Delta \varepsilon_p$ accounts for the contribution of plastic loading, and $q_0$ takes crack closure into consideration. There are also other definitions of $\Delta K_{eq}$, e.g. in Ref. \(^{11}\).

Path-independent line integral $J$

Another widely adopted method to incorporate plastic deformation at the crack tip is to use a path-independent $J$ integral. This concept was developed by Rice to analyse the strain concentration at notches and cracks. \(^{(113)}\) Per definition, it is an energetic contour integral along any path around a two dimensional crack tip:

\[
J = \int_{\Gamma} \left[ W_i dy - T_i \left( \frac{\partial u_i}{\partial x} \right) ds \right]
\]  

(1-9)

where $\Gamma$ is the path surrounding the crack tip; $T_i$ is the traction vector defined according to the outward normal along $\Gamma$; $u_i$ is the displacement vector and $ds$ is an element of arc length along $\Gamma$. $W_i$ is strain energy density given by:

\[
W_i = \int_0^{\varepsilon_i} \sigma_{ij} d\varepsilon_{ij}
\]  

(1-10)

with $\sigma_{ij}$ and $\varepsilon_{ij}$ the stress and strain tensors respectively.

Practically, $J$ integral can be viewed as the potential energy difference between two cracked bodies whose crack depths differ by an infinitesimally small amount:

\[
J = \frac{1}{B} \lim_{\Delta a \to 0} \frac{U(a + \Delta a) - U(a)}{\Delta a} = -\frac{1}{B} \left( \frac{dU}{da} \right)
\]  

(1-11)

Here $J$ is the rate of decrease of potential energy $dU$ with respect to a small increase in the crack depth ($da$), and $B$ is the specimen thickness. \(^{(24, 113)}\)

As originally devised for nonlinear elastic materials, one extreme case of $J$ integral is the linear elastic mode I loading. Then it equals to the energy dissipated during fracture per unit of newly developed crack surface, i.e. the strain energy release rate $G$:

\[
J_e = G = \frac{K^2}{E'}
\]  

(1-12)

with $E'$ equal to the Young’s modulus $E$ for plane-stress conditions and $E' = E(1 - \nu^2)$ for plane strain conditions, where $\nu$ is the Poisson’s ratio. Under elastic-plastic conditions the
application of $J$ integral is actually a kind of extrapolation, which is representative of crack tip stress-strain field.$^{24,42}$

The fully plastic solution of $J$ integral for a developing crack was first given by Shih and Hutchinson.$^{20}$ For materials with simplified plastic behaviour (pure power-law hardening), a Ramberg-Osgood type equation is:

$$\frac{\varepsilon_p}{\varepsilon_0} = \alpha \left( \frac{\sigma^\infty}{\sigma_0} \right)^{1/n}$$  \hspace{1cm} (1-13)

where $\varepsilon_0$ and $\sigma'_0$ are reference values (close to the monotonic true fracture ductility and true fracture strength); $\sigma^\infty$ is the remote tension stress; $\alpha'$ is a constant and $n$ is the strain hardening exponent (in one extreme being the linear elastic case, $n=1$; in the other extreme being perfectly plastic behaviour, $n \to 0$).

For a centre cracked plate (crack length $2a$, total width $2b$) under full plasticity for power-law hardening materials, the expression of $J$ integral can be written as:

$$J = a' \varepsilon_0 \varepsilon_0 (1 - \frac{a}{b}) g(a/b,n) \left( \frac{2b\sigma^\infty}{2(b-a)\sigma_0} \right)^{1/n+1}$$  \hspace{1cm} (1-14)

with the function $g(a/b,n)$ as a material and geometry dependent term of $a/b$ and $n$.

By taking equation (1-13) into equation (1-14) and rearranging, $J$ integral is:

$$J = \varepsilon_p ag(a/b,n)\sigma^\infty \left( \frac{b}{b-a} \right)^{1/n}$$  \hspace{1cm} (1-15)

Therefore, for a fatigue test with remote stress range $\Delta\sigma$ and local plastic strain range $\Delta\varepsilon_p$, the value of plastic $J$ integral range as a characterizing parameter for crack growth has the form of:

$$\Delta J_p = \left( \Delta \varepsilon_p \Delta \sigma^\infty \right) \cdot \left[ g(a/b,n)(1 - \frac{a}{b})^n \right] \cdot a$$  \hspace{1cm} (1-16)

With $g$ value already calculated and tabulated for various $a/b$ and $n$ situations, the total cyclic $J$ integral can readily be obtained by summing up the elastic portion and the plastic portion:

$$\Delta J_c = \Delta J_e + \Delta J_p = \frac{\Delta K^2}{E'} + \Delta J_p$$  \hspace{1cm} (1-17)

Note that $J$ integral should be only related to the part of the cycle for which the crack is open.

Hysteresis loops can be used to facilitate the calculation of $J$ integral, by defining $w_e$ and $w_p$ as elastic and plastic strain energy expended per cycle:
Figure 1-13 Representation of nominal stress and strain and corresponding energy terms in the determination of total cyclic J integral.

In terms of the cyclic stress-strain hysteresis loop shown in Figure 1-13, \( w_c \) is area \( E \) and \( w_p \) is area \( A+D \). Therefore, by integrating equations (1-6)(1-16)(1-17)(1-18)(1-19) and taking different crack front geometries into consideration, cyclic \( J \) integral can be written as:

\[
\Delta J_t = Y^2 \cdot 2\pi \cdot w_c \cdot a + Y^2 \left[ g(a/b,n) \cdot (n+1) \cdot \left(1 - \frac{a}{b}\right)^n \right] \cdot w_p \cdot a
\]  

(1-20)

Although the exact value of cyclic \( J \) integral requires full stress-strain history and crack geometry history, there are approximate solutions, e.g. for round bar specimens with a semi-circular surface defect (two major simplifications made in this case are omitting the variation of geometry factor \( Y \) and function \( g \)), \( \Delta J_t = 3.2 w_c a + 5.0 w_p a \) \( \) (1-21)

For other shapes of crack front, as long as the geometry functions are known, cyclic \( J \) integral can be calculated with reference to equation (1-21). For example, for a straight-front crack, the expression is:

\[
\Delta J_t = (3.2 w_c a + 5.0 w_p a) \cdot \left(\frac{Y_{cc}}{Y_{scc}}\right)^2
\]

(1-22)

with the \( (Y_{cc} / Y_{scc})^2 \) factor where \( Y_{cc} \) and \( Y_{scc} \) are geometry/compliance functions (of crack depth related to total diameter \( D \) of the specimen) for round tensile specimens with edge chordal cracks and edge semi-circular cracks.\(^{114}\)
Basic concepts

\[ Y_{cc} = 0.926 - 1.771 \left( \frac{a}{D} \right) + 26.421 \left( \frac{a}{D} \right)^2 - 78.481 \left( \frac{a}{D} \right)^3 + 87.911 \left( \frac{a}{D} \right)^4 \]  
(1-23)

\[ Y_{sec} = \frac{1.84}{\pi} \tan \left( \frac{\pi a}{2D} \right) \cos \left( \frac{\pi a}{2D} \right)^{0.5} \left[ 0.752 + 2.02 \frac{a}{D} + 0.37 \left( 1 - \sin \left( \frac{\pi a}{2D} \right) \right)^3 \right] \]  
(1-24)

It is then possible to express crack growth rates simply as a function of the total range of \( J \) integral

\[ \frac{da}{dN} = A_J \left( \Delta J \right)^{n_J} \]  
(1-25)

**C* type parameter for creep crack growth**

When it comes to steady-state creep crack growth, the crack tip parameters mentioned previously may still be used under specified conditions, i.e. \( K \) for creep brittle materials or shortly crept elastically-loaded creep ductile materials; \( J \) for shortly crept plastically-loaded creep ductile materials. However, when creep time is large, the influence of initial elastic or plastic strain weakens, and creep strain rate \( \dot{\varepsilon} \) becomes the corresponding controlling parameter. A commonly adopted relationship for stress and steady-state creep rate is Norton’s Law, which is similar to power-law hardening as in equation (1-13):

\[ \frac{\dot{\varepsilon}_{ss}}{\dot{\varepsilon}_0} = \alpha' \left( \frac{\sigma}{\sigma_0} \right)^{n'} \]  
(1-26)

where \( \dot{\varepsilon}_{ss} \) is steady-state strain rate, \( \dot{\varepsilon}_0 \) and \( \sigma'_0 \) are reference values, \( n' \) is the creep stress exponent. Therefore, an analogous line integral \( C^* \) was devised to characterize the redistributed crack tip stress-strain state:

\[ C^* = \int \left[ \dot{W}_i dy - T_i \left( \frac{\partial \dot{u}_i}{\partial x} \right) ds \right] \]  
(1-27)

with \( \dot{u}_i \) the displacement rate vector and \( \dot{W}_i \) the strain energy density rate related to point creep strain rate \( \dot{\varepsilon}_{ij} \), in the form of:

\[ \dot{W}_i = \int_{\varepsilon_0}^{\dot{\varepsilon}_{ij}} \sigma_{ij} d\dot{\varepsilon}_{ij} \]  
(1-28)

By comparing \( C^* \) and \( J \) integral, the concepts and structures of the equations are the same, with strain values replaced by strain rate values. This also leads to a similar energy rate interpretation of \( C^* \):

\[ C^* = -\frac{1}{B} \left( \frac{dU^*}{da} \right) \]  
(1-29)
where $U^*$ is the stress-power difference between two cracked bodies with identical creep behaviour but infinitesimal cracked areas at secondary creep regime. An illustration of this definition is shown in Figure D-14, with $P$ the (constant) creep load and $\dot{V}_{ss}$ the steady-state force line deflection (load line displacement) rate.

![Figure D-14](image-url)

Figure D-14 (a) schematic plot showing a cracked component under tensile creep load and (b) the energy rate interpretation of $C^*$.

The semi-empirical determination of $C^*$ can then be evaluated from experimental creep cracking data in the steady-state condition where power-law creep prevails. As $C^*$ is designed for extensive creep conditions (analogous to the total plasticity condition in the $J$ integral) at steady-state creep regime (power-law, equation (1-26)), a similar parameter $C_t$ was suggested to extend the $C^*$ integral concept into the small scale and the transition creep regime ($C_t$ reduces to $C^*$ after the transition time when steady-state extensive creep is achieved). $C_t$ is of more practical significance, because in a real component creep deformation often appears only in a region local to the crack tip, with the surrounding material being under linear elastic conditions.  

For compact tension (CT) specimens with constant force $P$, width $W$ and thickness $B$, the derived expression of $C_t$ is:

$$C_t = \frac{P\dot{V}_c}{BW} \frac{F'}{F}$$  \hspace{1cm} (1-30)

where $\dot{V}_c$ is the creep part of force line deflection rate:

$$\dot{V} = \dot{V}_p + \dot{V}_c + \dot{V}_e$$  \hspace{1cm} (1-31)

$F'/F$ is a geometrical associated factor ($F' = dF / d(a/W)$) determined from the finite element analysis. The value of this factor is available for typical laboratory loading type and testpiece geometries. Apart from this semi-empirical approach, the reference stress method is another approximate method to calculate $C^*$ type parameters. 

In creep-fatigue crack growth tests when the crack opening reaches a critical value (i.e. during dwell at maximum load), small scale creep at the crack tip plastic zone changes the local stress...
state and strain rate distribution and possibly causes an extension of the major crack. In such cases, an averaged value of the $C_t$ parameter during dwell, i.e. $(C_t)_{avg}$ was found to be the most effective crack tip parameter,\textsuperscript{119} which was defined as:

$$
(C_t)_{avg} = \frac{1}{t_h} \int_0^{t_h} C_t \, dt
$$

where $t_h$ is the hold time at peak load in one cycle.

Then equation (1-30) can be written as:

$$
(C_t)_{avg} = \frac{\Delta P \Delta V_e}{(BB_N)^{0.5} W t_h} \cdot \frac{F'}{F}
$$

with $B_N$ the thickness at side grooves.\textsuperscript{2}

The average time-dependent crack growth rates during hold time $(da / dt)_{avg}$ is expressed as:

$$
\left( \frac{da}{dt} \right)_{avg} = A_c (C_t)_{avg}^{nc}
$$

A widely used method is to evaluate creep crack growth during dwell and fatigue crack growth during cycles separately, and to sum them up to estimate the total crack growth behaviour:\textsuperscript{67, 68}

$$
\left( \frac{da}{dN} \right)_{total} = \left( \frac{da}{dN} \right)_{fatigue} + \left( \frac{da}{dN} \right)_{creep} = \left( \frac{da}{dN} \right)_{fatigue} + \frac{1}{f} \left( \frac{da}{dt} \right)_{avg}
$$

There are also alternative representations of creep-fatigue crack growth models based on this damage summation method, i.e. in Ref.\textsuperscript{11}. The application of these models will be illustrated in more details in Appendix D.

\subsection*{1.4.2 Semi-empirical short crack growth models}

Apart from these fracture mechanics based parameters, two semi-empirical models are also widely used for (short) crack growth behaviour under elastic-plastic loading conditions. Total or plastic strain is considered as the controlling parameter and $da / dN$ increases almost linearly with crack depth.

The model proposed by Tomkins assumes that the amount of crack decohesion at the crack tip (crack development in a single cycle) depends on the size of deformation zone where the critical shear stress is reached. The length of this deformation zone is a function of applied stress, material-dependent flow stress and the crack depth. For power-law hardening material, the model can finally be expressed as:
1.4 Life prediction and crack growth models

\[
\frac{da}{dN} = \frac{\pi^2 \Delta\varepsilon_p \Delta\sigma^2}{8 \left(2\bar{T}\right)^2} \times \left[1 + \frac{\pi^2}{8 \left(2\bar{T}\right)^2} \left(\frac{\Delta\sigma}{2\bar{T}}\right)^2\right] \tag{1-36}
\]

where \(\bar{T}\) is an equivalent tensile stress term in the range between the ultimate tensile stress and true tensile fracture stress.\(^{120}\) The direct evaluation of \(\bar{T}\) is difficult because its value varies with applied stress range in the deformation zone.\(^ {101}\) Alternatively, it can be approached through regression on known experimental data. The validity of Tomkins model is also self-explanatory as, by integration, equation (1-36) gives a form of Coffin-Manson type relation:

\[
\Delta\varepsilon_p N^\alpha' = \text{Const.} \tag{1-37}
\]

where \(\alpha'\) is the Coffin constant (slope value).

Another model was devised by Skelton (originally from Pineau) for short crack growth behaviour when creep damage is not so significant:

\[
\frac{da}{dN} = B d^{Q} \tag{1-38}
\]

with \(Q\) as a material dependent exponent which is usually close to unity. The value of \(B\) is directly related to the total strain range \(\Delta\varepsilon_t\) and environment, which can be determined experimentally.\(^ {121}\) At very small crack depths this equation may break down and crack growth rates seem to be independent of crack depth. This critical minimum crack depth was found to be 140\(\mu m\).\(^ {27}\)

Since crack growth rate (\(mm/cycle\)) generally increases as the dwell time is extended, a rather conservative upper bound of \(B\) is given for a number of alloys as:\(^{35}\)

\[
B = 2.61 \times 10^4 \Delta\varepsilon_t^{1.85} \tag{1-39}
\]

Although this upper bound was originally designed for continuous-cycling low cycle fatigue tests under 550\(^{\circ}\)C in ferritic steels, its application can well be extrapolated for austenitic steels at higher temperatures and thus is included in the R5 assessment code.\(^5,27\)

In creep-fatigue tests, a term \(d_c\) can be added into equation (1-38) to account for the creep damage in each cycle.\(^7,122\) This concept is based on a damage summation approach:

\[
\frac{1}{N'} = \frac{1}{N} + d_c \tag{1-40}
\]

where \(N\) denotes the number of cycles involved in a continuous-cycling test and \(N'\) represents the reduced number of cycles in a similar creep-fatigue test with hold time. Then the crack growth relation can be written as:

\[
\frac{da}{dN}_{\text{total}} = \left(\frac{da}{dN}_{\text{fatigue}}(1 + N d_c)^2\right) \tag{1-41}
\]

By integrating equation (1-38):
Basic concepts

\[ N = \frac{1}{(Q-1)B} \left( a_0^{1-Q} - a^{1-Q} \right) \]  

(1-42)

If \( Q \) equals unity then

\[ N = \frac{1}{B} \ln \left( \frac{a}{a_0} \right) \]  

(1-43)

These two equations can be applied in beach mark/striation counting where the spacing normally increases with propagating cracks. Corresponding to equation (1-38), the modified creep-fatigue crack growth relation can be expressed as

\[ \left( \frac{da}{dN} \right)_{\text{total}} = B a^Q \left\{ 1 + \frac{d_c}{(Q-1)B} \left( a_0^{1-Q} - a^{1-Q} \right) \right\}^2 \]  

(1-44)

and when \( Q = 1 \)

\[ \left( \frac{da}{dN} \right)_{\text{total}} = B a^Q \left\{ 1 + \frac{d_c}{B} \ln \left( \frac{a}{a_0} \right) \right\}^2 \]  

(1-45)

Values of \( d_c \) have been determined for a wide range of alloys.\(^{122}\)

Provided that material creep data is at hand, the estimation of \( d_c \) is also possible though life fraction rules. For example, in the ductility exhaustion method:

\[ d_c = \int_0^{t_b} \frac{\tilde{\varepsilon}_c}{\tilde{\varepsilon}_f \left( \tilde{\varepsilon}_c \right)} \, dt \]  

(1-46)

where \( t_b \) \((0 \leq t \leq t_b) \) is the creep dwell period in each cycle; \( \tilde{\varepsilon}_c \) is the instantaneous equivalent creep strain rate during the dwell and \( \tilde{\varepsilon}_f \left( \tilde{\varepsilon}_c \right) \) is the creep ductility at that strain rate, accounting for the stress state. The strain rate can be evaluated from the instantaneous stress relaxation data during the dwell period.\(^{123}\)

Likewise, \( d_c \) may also be determined by time fraction method as

\[ d_c = \int_0^{t_b} \frac{1}{t_R(\sigma,T)} \, dt \]  

(1-47)

where \( t_R \) is the creep rupture time calculated as a function of the stress \( \sigma \) and temperature \( T \) during the hold time. The application of these formulas can be found in section 5.2.

1.4.3 The strain energy density model

Alternatively, a model based on hysteresis strain energy density can be used for creep-fatigue testing conditions to account for the effect of both the plastic strain during ramping and creep damage during the hold period.
The concept of using strain energy density factor to correlate with fatigue crack development was proposed by Sih as a more general treatment for ductile materials with plastic deformation than using the stress intensity factor $K$. He views the fatigue process as a quasistatic series of discrete crack growth steps. When the material dependent strain energy density reaches a threshold value at the most favoured direction, crack extension will occur. According to Sih’s definition, SEDF can be computed by multiplying a limit radius of the core region with the strain energy density, which can be derived from the finite element stress-strain analysis at the crack tip.\textsuperscript{124-126}

Instead of such a complex calculation scheme for strain energy density, a simplified scheme may be adopted in practice. It is generally accepted that the area contained within the stress-strain hysteresis loop of a cyclic loaded structure containing a crack represents the total plastic work done for the fracture process at the crack tip.\textsuperscript{127-129} A large portion of this energy is dissipated as heat, whereas the rest is expended mainly in microstructural evolution (development of dislocation structures, formation of voids, etc.) and creation of new surfaces, i.e. development of crack fronts.\textsuperscript{130, 131} In other words, the cyclic plastic strain energy (hysteresis energy), $w_p$, is proportional to the extent of crack propagation, as well as the microstructural alternation.

Irrespective of the different mechanisms of creep and fatigue, $w_p$ should be able to account for their effects. Since the crack tip plastic strain energy is proportional to the far field plastic strain energy and the crack depth ($w_p \cdot a$),\textsuperscript{127} this integrated parameter (the strain energy density factor, SEDF) can represent the driving force for crack extension, i.e.

$$\frac{da}{dN} = A_w (w_p \cdot a) ^ {w_M}$$  \hspace{1cm} (1-48)

![Figure 1-15 Representation of hysteresis loop shape with hold time at peak strain in tension.](image)

Note that the definition of SEDF in this study is different from the one used by Sih, because the radius of the core regime is in fact implicitly comprised as a length term correlated by crack depth $a$ and the constants $A_w$ and $M_w$ (thus the unit doesn’t change).
For continuous-cycling hysteresis loops, $w_d$ is only related to the area $A$ as shown in Figure 1-15. It is calculated by subtracting complementary parts from the gross rectangular area:

$$w_d = \Delta \sigma \Delta \varepsilon_p - 2 \int_0^{\Delta \sigma} \varepsilon_p d\sigma = \frac{\Delta \sigma \Delta \varepsilon_p}{1 + n}$$

(1-49)

This differs from the previous definition in $\Delta J_p$, where the plastic strain energy $w_p$ is determined as the total minus the elastic area beneath the tensile-going loading branch of the stress-strain hysteresis loop (A+D in Figure 1-13). For loops with dwell time, the total energy dissipated should be the sum of all three areas (A, B and C in Figure 1-15), i.e.

$$w_d = \frac{\Delta \sigma \Delta \varepsilon_p}{1 + n} (1 - n) + \frac{\sigma_{\text{max}}^2 - \sigma_{\text{hold}}^2}{2E} + \frac{\Delta \sigma (\sigma_{\text{max}} - \sigma_{\text{hold}})}{2E(1 + n)}$$

(1-50)

### 1.5 Microstructural aspects

For a comprehensive crack development assessment under creep-fatigue conditions, it is not only essential to have a good understanding of mechanical behaviour based on stress-strain response, but is also necessary to obtain information regarding the microstructural evolution. The raw materials involved in this study are tempered ferritic-martensitic steels, which undergo significant change in microstructure after severe creep-fatigue deformation. Therefore, it is worthwhile to understand why the microscopic parameters change and how they are related to macroscopic mechanical parameters. In order to fully understand how exactly the microstructure changes during creep-fatigue deformation, background knowledge of three related topics will be shortly emphasized in this section (namely martensitic structure formation, strengthening mechanisms, recovery and recrystallization), which are all closely related to this study.

#### 1.5.1 Formation of martensitic microstructure

There are mainly two reasons to briefly review martensitic structure in this section. The first is certainly because the investigated materials are tempered martensitic steels. The evolution of their microstructure is almost directly associated with the martensitic morphology, which should be introduced in the first place. The second point of reviewing crystallographic features is because that's the foundation of using EBSD technique to capture the evolution of microstructure during creep-fatigue deformation. Owing to the distinctive crystallographic orientation relationships, it is finally possible to distinguish between different sub-structures (e.g. laths and subgrains).

The martensitic transformation is probably the most common and important phase transformation process in steel production, typically for the sake of an improvement of several
Austenite (fcc, face centred cubic structure) formed at higher temperature is cooled above a certain rate, to below a material dependent critical temperature (transformation starting temperature), and a non-diffusional shear process happens very quickly which distorts the original lattices into new lattices (bct, body centred tetragonal structure). This process is schematically shown through the Bain model in Figure 1-16.

The transformation from fcc austenite to intermediate bct structure and finally bcc martensite.

The axis system $a_1$, $a_2$ and $a_3$ represents the three-dimensional directions in original cubic cells (constructed with black lines), whereas $b_1$, $b_2$ and $b_3$ are their counterparts in new cells (constructed with red lines). The formed intermediate bct cell is represented by red circles indicating iron atoms, with larger edge length in the $b_3$ direction than the others. Under Bain strain (associated with an appropriate rigid body rotation), the lattice parameter in $b_3$ can be compressed to achieve a practical bcc structure. Crystal slipping or twinning occurs at the same time to accommodate the distortion of lattice (preservation of original shape). Finally, the martensitic structure can be formed.\textsuperscript{135-137}

There are several unique features in this process, such as its athermal nature (rapid transformation at low temperature) and the conservation of its chemical composition (trap of interstitial carbon atoms in octahedral sites). But perhaps the most prominent feature is a defined crystallographic orientation relationship between parent lattice and distorted
martensitic cells. One widely used orientation relationship is the Kurdjumov-Sachs (K-S) relationship,\textsuperscript{138} which is in the form of

\[
\{111\}_\gamma // \{011\}_{\alpha'} \quad <10\bar{1}>_{\gamma} // <11\bar{1}>_{\alpha'}
\]  

(1-51)

This relationship is also illustrated in Figure 1-16, where the (111) plane in austenite (shown in blue colour) is parallel to the (011) plane in martensite (shown in brown colour). The subscript $\gamma$ represents austenite, whereas $\alpha'$ indicates martensite (should be distinguished from $\alpha$, which is ferrite).

![Figure 1-17 Illustration of six crystallographic variants for a habit plane according to the K-S relationship.](image)

The planar interfaces between parent austenite and produced martensite are called habit planes, which are preferred locations where transformation is initiated.\textsuperscript{139} In the K-S relationship, there are six crystallographic variants for each habit plane. In Figure 1-17, the blue triangle represents a (111) habit plane surrounded by three edge directions indicated by...
1.5 Microstructural aspects

green arrows (in accordance with the (111) plane in austenite in Figure 1-16). The brown rectangles designate the parallel plane (011) in martensite, whereas their diagonals are also parallel to the corresponding edge directions in austenite.

In total six configurations can be formed for the same habit plane. Due to the symmetry of cubic crystals four types of habit planes exist, i.e.:

\[
(111)_{\gamma} // (011)_{\alpha'} \\
(\overline{1}1\overline{1})_{\gamma} // (011)_{\alpha'} \\
(\overline{1}1\overline{1})_{\gamma} // (011)_{\alpha'}
\]

which results in 24 martensite variants each with a different orientation created from a single austenite crystal. There are other relationships similar to but slightly different from the K-S relationship, such as the Nishiyama-Wassermann (N-W) relationship and the Greninger-Troiano (G-T) relationship. The application of those models depends on the composition of the material, e.g. the content of carbon. The K-S relationship was found to be appropriate for low carbon steels and will be considered within the scope of this study.\textsuperscript{140,141}

Owing to the rotation/shear deformation during phase transformation, martensite has some distinctive features in its morphology. Along with the progressing detection technique, the theory of the morphology of martensites in low carbon steels has been developed since the last century and the present interpretation is as follows: a prior austenitic grain is partitioned into a number of packets, in which the laths (martensite variants) are with the same habit plane; each packet is subdivided into several parallel blocks, where the laths are with organized orientations.\textsuperscript{142} An illustration of typical lath martensite structure is shown in Figure 1-18. (In some low carbon steels, laths tend to arrange themselves into two sets of groups as variant pairs in one block, separated by sub-block boundaries.\textsuperscript{140} But this is not a common law for all lath martensitic structure thus not represented in Figure 1-18).
As mentioned above, there are four types of habit planes according to the K-S relationship. Therefore, a maximum of four crystallographically different packets can appear in a single austenite grain. Within each packet, a maximum of six different types of blocks on the same conjugate parallel close packed planes can exist (in reality only three types of blocks due to the preferred arrangement of variant pairs).

Table 1-1 Typical misorientation values for different boundaries in martensite.

<table>
<thead>
<tr>
<th>Boundary types</th>
<th>Packet</th>
<th>Block</th>
<th>Sub-block</th>
<th>Lath</th>
</tr>
</thead>
<tbody>
<tr>
<td>Misorientation angles</td>
<td>&gt;15°</td>
<td>&gt;10.53°</td>
<td>5°~10° (avg. 7°)</td>
<td>&lt; 5° (avg. 3°)</td>
</tr>
</tbody>
</table>

Table 1-2 Typical dimensions of different units in martensite.

<table>
<thead>
<tr>
<th>Structure unit</th>
<th>Packet</th>
<th>Block</th>
<th>Sub-block</th>
<th>Lath</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size/width (μm)</td>
<td>&gt;100</td>
<td>10~20</td>
<td>5~10</td>
<td>&lt; 1</td>
</tr>
</tbody>
</table>

Thus the corresponding boundaries also have more or less defined crystallographic configurations. Generally, the misorientations across block boundaries are of high angles, whereas the lath boundaries are of low angles. The typical values for various boundaries are shown in Table 1-1. Accordingly, the dimensions of those microscopic structures are also different, with typical values shown in Table 1-2. The knowledge of the martensite morphology is the basis for examining microstructural evolution under microscopes, as will be discussed later in Chapter 4.

1.5.2 Strengthening mechanisms in tempered martensitic steels

After the martensitic transformation, steels are normally reheated for some time at moderate temperatures below A1. A tempering stage will improve some mechanical properties of the initial microstructure and achieve an optimized combination of both strength and ductility/toughness. From the metallurgical aspect, two processes taking place during tempering are the rearrangement of dislocations and the precipitation of carbides, which corresponds to the two most important strengthening principles for creep-resistant tempered ferritic-martensitic steels: sub-boundary hardening and precipitation hardening.

Owing to the martensitic transformation, a significant amount of dislocations are generated as a compensation for the large shear deformation in the matrix. These dislocations are stored within the lath structure and upon the relieving tempering process, will be partly annihilated or agglomerated to form the equiaxed subgrain structure. Most of the structure units are retained as before tempering, i.e. block and lath boundaries, whereas the intermediate bct martensite (if there is any) is fully transformed into a bcc structure. When subjected to an external force, great numbers of sub-boundaries (including subgrain boundaries, lath boundaries and block boundaries) act as internal obstacles to dislocation motion. The finer the sub-structure is, the larger the resistance can be expected.
The role of precipitation hardening can be viewed as twofold. The first strengthening mechanism is through direct interaction with free dislocations (Orowan strengthening); the second is through the interaction with sub-boundaries (dislocation network), which is the most important strengthening mechanism for creep deformation.\textsuperscript{145,147,148}

After tempering, two types of precipitates can be formed, namely $\text{M}_{23}\text{C}_6$ (M=Fe, Cr) carbides and MX (M=V, Nb, Ta and X=C, N) carbonitrides. $\text{M}_{23}\text{C}_6$ particles are usually larger and their preferential locations are lath/block boundaries. They exert pinning forces against boundary coarsening to maintain hindrance against dislocation movement. On the other hand, MX particles are smaller and dispersed finely in the matrix. Free dislocations need to bypass or cut through MX precipitates in order to deform the matrix, which acts as a back force to deformation. A schematic illustration showing subgrain structure and precipitates is displayed in Figure 1-19. (Although most features are inherited from the martensitic transformation, the emergence of subgrain boundaries and dispersed particles suggests that this new structure should be termed as tempered martensitic structure rather than martensitic lath structure).\textsuperscript{144}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{subgrain_structure.png}
\caption{Schematic drawing of subgrain structure and precipitates in tempered martensitic steel (after Ref. 145, 149).}
\end{figure}

In addition to those two hardening mechanisms, solid solution hardening from elements like W and Mo also contributes to the strength of materials at elevated temperatures.\textsuperscript{149}

In the later stage, creep-fatigue loading may deteriorate the microstructure, such as the widening/vanishing of martensitic lath structure; the growth/disappearance of fine precipitates; and the formation of new coarse particles (such as intermetallic Laves-phase and Z-phase). For example, Fournier et al. have found that for 9\%Cr martensitic steels, the mean subgrain size increased strongly after creep-fatigue deformation. More specifically, the creep-fatigue loaded samples exhibited larger subgrain dimensions than both the fatigue loaded and creep loaded samples.\textsuperscript{132,134} It was also concluded by others that the growth of sub-structures is strongly dependent on the accumulated inelastic strain, whereas the growth of carbides is mainly dependent on time.\textsuperscript{150} Ultimately, the effectiveness of strengthening is greatly reduced and the microstructure is regarded as not stable anymore.
1.5.3 Recovery and recrystallization in creep-fatigue deformation

As introduced in previous sections, martensite is in fact a metastable phase which will evolve under thermal activation. A large amount of stored energy (in the form of lattice defects such as dislocations) are relieved upon the transformation toward a more favoured microstructure. During this process, the self-rearrangement of dislocations leads to the formation of subgrains with low angle grain boundaries (this usually happens during the tempering process, e.g. at 570°C~700°C for the tested materials in this study). However, when creep-fatigue loading is subsequently applied on tempered martensitic structures, a further decrease of dislocation density and subgrain coarsening tends to appear (e.g. at 600°C and 625°C for the specimens in this study). This is generally referred to as (dynamic) recovery, which includes only the motion of low-angle grain boundaries. (Notable recovery can normally takes place at the intermediate temperature range, e.g. at one third of the material’s melting point). In extreme cases such as at high temperatures above a critical value (typically 0.5T_m) or high plastic strain, (dynamic) recrystallization can happen, along with the movement of high-angle grain boundaries. A finished recrystallization process can change the morphology and mechanical properties of the initial microstructure completely. Therefore, it is important to understand the mechanisms of recovery and recrystallization, as these two phenomena can influence a material’s resistance against fatigue and creep considerably.\(^{151}\)

For tempered martensitic steels subjected to creep-fatigue deformation, sub-boundaries made from dislocation arrays continue their polygonization process by the further absorption of thermally activated dislocations and the progressive integration of adjacent sub-boundary pairs. Those characteristic processes of dynamic recovery originate from the reduction of internally stored defect/boundary energies (decrease of total grain-boundary area per volume). Upon the coarsening of sub-boundaries, the misorientation associated with neighbouring subgrains also increases (sub-boundaries become sharpened). As long as the accumulated misorientation is still within the low-angle range, the growth of subgrains is still regarded as a recovery phenomenon.\(^{152}\)

Normally, recovery is considered to be a rival to recrystallization, as strong recovery can diminish the remaining driving force until a critical value where recrystallization is suppressed, especially for high SFE (stacking fault energy) materials such as ferritic iron. However, recovery can also be viewed as a nucleation stage for recrystallization (provided that the driving force for subsequent recrystallization is still sufficient). Here the term ‘nucleation’ is not interpreted in the traditional way as purely from thermal fluctuation in a homogeneous structure, but from the existing inhomogeneity due to excessive recovery, namely, high-angle sub-boundaries and distorted lattices by large precipitates. These newly formed high-angle sub-boundaries have higher mobility (15°~45° misoriented boundaries have the fastest migration rate) compared to low-angle boundaries and migrate under the assistance of the external plastic deformation.\(^{151, 152}\)
It has been generally accepted that dynamic recovery takes place as a steady ongoing process in strengthened alloys at elevated temperatures, characterized by dislocation annihilation and sub-boundary formation. However, the occurrence of dynamic recrystallization requires three critical values to be reached: temperature, peak/total strain and strain rate. With a proper combination of those three parameters, dynamic recrystallization can also happen for low carbon martensitic steels.\textsuperscript{153-155}

1.6 Experimental approaches

In order to successfully carry out creep-fatigue crack growth experiments, guidance and advice from existing testing procedures are normally worthwhile to follow. With regard to the current study, one code of practice is important, which mainly focuses on short-crack creep-fatigue crack growth.\textsuperscript{7} It gives recommendations on the scope of usage; definition of terminology; requirement for testing equipment; design of testpieces; test procedures as well as data analysis methods. In particular, this code of practice places the emphasis on the monitoring techniques for short crack development (e.g. potential drop methods). Certainly not all aspects are comprised in this code of practice, because the common details are already documented elsewhere, e.g. in Ref.\textsuperscript{1, 2, 41, 115}. Test specifics in accordance with these standards will be described in Chapter 2, and the purpose of this section is to briefly introduce two techniques that are particularly crucial for this study.

1.6.1 DCPD technique

In order to predict the instantaneous crack depth accurately, non-destructive real-time monitoring techniques should be applied during test, without interference from the experiment itself. Three commonly utilized procedures for cracked laboratory specimens are the optical method, the compliance method and DCPD (direct current potential drop) method. However, the previous two methods have certain limitations which are not suitable for creep-fatigue tests at elevated temperatures. For example, the optical method relies on a legible reading of external crack length, which is typically not possible either due to an enclosed testing environment (e.g. in a closed furnace), blurred surface conditions (oxidation scales) or complex crack geometry (sub-surface crack development). As for the compliance method, the inevitable plastic deformation (at least around the crack tip) throughout creep-fatigue deformation brings another factor to influence the relationship between load and displacement, leading to a less reliable crack depth interpretation. On the other hand, the DCPD method is based on simple yet robust physical laws, that the resistance of a conductive body is inversely proportional to the surface area of the minimum cross section. The good stability of the DCPD technique has made it a preferred in-situ crack monitoring procedure not only in the laboratory, but also in real components.\textsuperscript{7, 41, 156}
In practice, a constant direct current passes through the specimen to generate an electrical field of homogenous current flow. When a crack propagates, the growing crack depth brings about a reduced ligament section in the cracking plane, which changes the current streamline (i.e. electrical resistance increases) and the voltage difference between two measurement points across the crack. When voltage/electrical potential values are continuously recorded for a crack growth test, instantaneous crack depth can be deduced provided that a suitable calibration curve is available. In fact, an adequate calibration curve is usually the most essential and difficult part in this technique.

One possibility is to use the conformal mapping technique to really calculate the change of current flow, as done by Clark and Knott for various kinds of edge cracks from notches. Alternatively, finite element techniques can be adopted to provide a relatively rapid and inexpensive method to acquire the calibration curve. Other similar efforts have indicated that crack front shape (e.g. different aspect ratios) has a predominant influence on the development of PD values. Example of three crack calibration curves for short cracks is illustrated in Figure 1-20 to give a general idea of the range and shape of these theoretically derived conversion curves. The reported accuracy of numerical calculation is 100µm. Depending on the notch shape and crack front (even reproduced with the same notch depth, i.e. 0.2mm in the case of Figure 1-20), the calibration curve can be so different. Therefore, it is necessary to establish a conversion scheme in the first place, as will be discussed in more detail in section 3.2.

Figure 1-20 Example of PD calibration curves for short cracks reproduced from the literature (after Ref.157-159).

In practice, for complex geometries and crack paths it is difficult to apply numerical analysis on the electrical field. Therefore, the calibration curve may be formulated by an empirical relationship, generated from an interrupted trial test where sequential PD values can be associated with precise crack depth via interpolation. For generally standardized specimen geometries and crack fronts, analytical relationships between normalized potential values and normalized crack depths can be relatively easy to deduce (e.g. linear interpretation is usually adopted for CT specimens with a straight-front long crack).
1.6 Experimental approaches

In the experiments, two basic setup parameters in the DCPD technique are the input current value and the displacement between two output leads. A proper choice of input current should be capable of providing detectable PD change when the crack propagates, while avoiding overheating at crack tip area. Commonly employed currents are between 5 and 50A, depending on material and specimen geometry. (In combination with the voltage resolution of ±0.1µm, resolution in crack size is better than 0.1% of the specimen width). A similar methodology exists for the spacing between output leads. They should be placed as close to the cracking plane as possible for higher sensitivity, while an over-close arrangement can lead to excessive noise to signal ratio especially when the crack front is irregular. The recommended separation distance is 4 to 12 notch depths (a typical distance is 4mm, i.e. 2mm above and below the cracking plane).

There are also some uncertainties in the DCPD technique, e.g. crack closure induced short circuit electrical paths in cracked faces; temperature fluctuation induced resistivity change; temperature difference induced thermoelectric EMF at welding spots; plasticity induced resistivity change and microstructural evolution induced resistivity change. For the first uncertainty, it can only be mitigated (not excluded) by taking measurements at peak load where the closure effect is minimum. For the second uncertainty, it has been concluded from the literature that a 1° temperature fluctuation generates a change of the PD value of a few µV (compared with the change of PD value during a cycle with almost an order of magnitude higher). As long as the temperature is maintained within a valid range during testing (resistance heating furnace is normally stable and generates less noise), its fluctuation has only a small influence on material resistivity. In fact, this problem can be minimized by applying a reference probe on the same testpiece. The third uncertainty comes from the thermocouple effect of dissimilar metals. This problem can be alleviated by using a suitable material for output leads, and making use of a modern DCPD monitor which has an auto correction function (the displayed PD value is actually corrected through pulsed measurement with zero or reversing current to eliminate thermally induced PD). As for the last uncertainty, it is related to the inherent resistance of the tested material to plastic/creep deformation. The resistance change due to microstructure can be corrected by adding reference probes attached to the same specimen, although experience has shown that for the materials of interest in this project the change of resistance is not that significant.

It is suggested that sometimes the initial PD value at the start of the test (e.g. during the crack incubation period) first decreases until stabilization and subsequently increases with true crack extension. Apart from the possible influencing factors discussed in the previous paragraph, other reasons can be the initial loading condition, plasticity, creep damage and crack tip oxidation. (In this case, the minimum PD value should be adopted as the initial PD value for crack depth calibration process). In other circumstances PD can increase at the start of the test, exhibiting an apparent primary behaviour which is not due to crack extension, but is related to the development of a critical creep damage zone. For experiments involved in this study, although a certain degree of scattering of PD values existed, such initial PD transients were not experienced. This will be considered in more detail in section 3.2.1.
To prevent input current from flowing into machine components, either the testing machine should be properly grounded to provide a sufficiently high resistance (several orders of magnitude larger than specimen resistance), or the specimen should be electrically insulated from the loading grips.  

1.6.2 SEM imaging by EBSD

Traditional post-test microstructural analysis involves optical microscopy (OM) by bright field optical microscope or electron microscopy (EM) by scanning electron microscope. The advantage of optical microscopy comes from the relative easier sample preparation stage as well as microscope handling, while the main drawback is the limited resolution down to the micrometre scale. Therefore, SEM (scanning electron microscopy) is often utilized to achieve a better resolution in the nanometre scale, especially to identify the fine microstructure features in complex structures. The signal in SEM is mainly from secondary electrons (SE) emitted from the beam interacted volume close to the sample surface. Hence, SE imaging has high spatial resolution and strong topographic contrast, which can serve as a good measurement for interior crack or void identification as well as fractography.

![Figure 1-21 Schematic view of an EBSD system.](image)

At the same time as an incoming electron beam hits the sample (70° tilt), some electrons are backscattered due to the interaction with much heavier nuclei. When the escaping path fulfils Bragg’s law, a characteristic diffraction pattern associated with local lattice parameters can be formed. Since the electron beam bombards a certain volume of crystalline structures, a number of bands (from different diffraction planes) form the so called Kikuchi lines, which can then be captured by a detector (CCD camera). This pattern is termed electron backscattering pattern.
(EBSP), and this technique is referred to as electron backscattering diffraction (EBSD). A schematic illustration of the EBSD technique is shown in Figure 1-21.\textsuperscript{165, 166}

With the assistance of modern analysis software, EBSD can be evaluated by an optimized Hough transformation and indexed with a pre-knowledge of candidate phase parameters. Due to the unique relationship between crystallographic characteristics and EBSD, phase and orientation information for each measured point can be obtained. (In reality, several possibilities will be assessed and usually the best fit will be chosen by the software). In this way, a connection between crystallography and local microstructure can be established.\textsuperscript{167}

As will be shown in Chapter 4, the microstructure of investigated materials under creep-fatigue deformation conditions will evolve, which means that the associated mechanical properties may also change. This has a big influence on the crack growth behaviour, thus obtaining information of instantaneous microstructure can be the key issue to improve the effectiveness of applied crack growth models (Chapter 5). Thus the idea of employing EBSD analysis is exactly because as a powerful tool, EBSD can give quantitative information about phase identification, grain boundary misorientation, grain dimension, etc., which are indices in microstructural characterization.

Here the definition of a grain is not identical with the traditional metallurgical interpretation, but purely from measured crystallographic orientations: a group of measurement points which are within a user-defined orientation range (this is effectively a grain reconstruction method). The quality/accuracy of EBSD mainly depends on its spatial resolution and angular resolution. Spatial resolution is affected by the testing material (backscattering is more pronounced for materials with larger atomic numbers) and probe current (an intermediate accelerating voltage generates sufficient signals, while keeps the interaction volume as small as possible). With a modern facility, a spatial resolution of 10nm can be achieved for ferrites. Angular resolution represents the uncertainties of measured orientation values compared with the true values, which relies on the operation condition of microscope as well as sample qualities. Typically it is limited to 0.5° at best, but to be conservative a 2° minimum misorientation angle for low angle grain boundaries is often chosen to exclude inaccuracies due to angular resolution.\textsuperscript{168}

The practical application of the EBSD technique and corresponding data analysis will be presented in Chapter 4, where different types of figures and parameters utilized in the EBSD supporting OIM software are presented. To better understand the analysis, it is necessary to first introduce some terminology:\textsuperscript{169}

a) **Confidence Index (CI):** this parameter quantifies the level of confidence in the orientation identification. During the scanning process, automated indexing of the diffraction pattern is done by the software itself, provided that the correct phase (associated with lattice parameters) is selected. For each diffraction pattern, several possible orientations may be found, which fulfill the diffraction condition according to the analysis routines. The software uses a voting system and ranks all these possibilities. The bigger the number of votes, the more accurate the detection process is. The confidence index is then calculated by dividing the vote number difference between the first and second solutions by the total votes. Therefore CI ranges from 0 to 1. (For unresolved points, CI=−1).
b) Image quality (IQ): this parameter reflects the sharpness of the detected EBSP, and is influenced by both external (such as sample surface finish and microscope condition) and internal factors (such as the imperfection in crystal lattice). By plotting IQ values in a grey scale, an image quality map can be obtained. With a properly tuned microscope and a well prepared sample surface, what an IQ map denotes is effectively the defects within the sample matrix, e.g. precipitates, boundaries, dislocations.

c) Tolerance angle: the minimum value a line segment can be regarded as a ‘grain’ boundary (user defined). Note that the meaning of a ‘grain’ in the OIM software is not the same as in the traditional metallurgical respect, but is purely dependent on the misorientation between neighbouring scanning points. In other words, the software automatically checks the orientation difference in every adjacent grid pair, and when this value is larger than the tolerance angle, the boundary is regarded as a ‘grain’ boundary; when smaller, this grid pair is said to be within the same ‘grain’.

d) The coloured map of the inverse pole figure (IPF): an inverse pole figure shows the stereographic projection of the sample position (RD, TD and ND) relative to the crystal direction. Due to cubic symmetry, a unit triangle (confined by \([001]\), \([110]\) and \([111]\)) can be used for simplicity. It is then possible to assign colour gradation to represent how close the sample position is to these three axes. By default, colours of red, green and blue are assigned to \([001]\), \([110]\) and \([111]\) axes, respectively. Therefore, it is possible to form a coloured map of an inverse pole figure for each EBSD scan, in which the displayed colours represent orientations of every scanning point. This is the most important type of image in OIM, because it directly visualizes lattice orientation information.

e) Step size: the displacement between adjacent scanning spots. Normally the smaller the step size, the finer and more accurate the result will be. However, scanning at a smaller step size also takes much more time.
Chapter 2

Material and test details

The most important purpose of this project is to investigate short-crack creep-fatigue crack growth behaviour in advanced martensitic turbine steels. Two different types of steel employed both belong to the 9-12%Cr creep resistant steel group. The designated names for them are 10Cr (short for 10CrMoVNbN) and FB2 (short for 9CrMoCoVNbNB). A round-bar type specimen with chordal crack starter (effectively a SENT type geometry) was adopted for the strain-controlled creep-fatigue tests. Three main governing variables in the experiments were the strain (displacement) range (controlled by a side-contact extensometer), testing temperature (controlled by a resistance heating furnace) and dwell period (in terms of duration and position in the cycle), which then led to comprehensive test matrices. The crack development was continuously recorded by a DCPD monitoring system. After creep-fatigue deformation, some of the tested samples were carefully prepared for further microstructural analysis under optical and electron microscopes. In this way, materials’ degradation associated with the deformation history as well as crack propagation could be mutually related. In this chapter, a more detailed description of the testing materials and experimental procedures are presented.
2.1 Material details

The group of 9-12%Cr steels was originally developed for power generation applications, which typically involve thermo-mechanical loading. A good alloy design philosophy starts with a proper selection of elements. Apart from the base element iron, the second most important element is Cr, which essentially provides oxidation/corrosion resistance and forms Cr$_{23}$C$_6$ strengthening particles. Some researchers believe 9% of Cr is the optimal amount because creep rupture strength with 9%Cr is the highest, whereas in reality a better oxidation resistance can be achieved with a further increase of Cr content to 10-12% (but at the expense of creep strength). Other typical alloying elements are molybdenum, vanadium, cobalt and niobium, which either offer solid solution hardening or form dispersed precipitates to increase creep resistance. In particular, the carbon content is decreased so as to achieve a uniform dispersal of M$_{23}$C$_6$ particles.

Apart from high creep (and/or oxidation) resistance, other unique properties include low thermal expansion, high heat conductivity and good ability in welding. By adjusting the weight percentage of the main elements or by adding small quantities of additional strengthening elements, different steel grades sharing similar microstructures but with altered properties can be obtained (certainly an appropriate heat treatment procedure is mandatory, whereby the optimal combination of strength and ductility is reached).

The designated names for the two tested steels in short-crack creep-fatigue crack growth tests are 10Cr and FB2. The corresponding chemical compositions are shown in Table 2-1.

<table>
<thead>
<tr>
<th>Steels</th>
<th>C</th>
<th>N</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Nb</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>P</th>
<th>S</th>
<th>B</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>10Cr</td>
<td>0.13</td>
<td>0.046</td>
<td>9.94</td>
<td>1.45</td>
<td>0.17</td>
<td>0.054</td>
<td>0.1</td>
<td>0.44</td>
<td>0.52</td>
<td>0.03</td>
<td>0.009</td>
<td>0.0005</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>FB2</td>
<td>0.14</td>
<td>0.026</td>
<td>9.28</td>
<td>1.51</td>
<td>0.19</td>
<td>0.053</td>
<td>0.08</td>
<td>0.31</td>
<td>0.15</td>
<td>0.031</td>
<td>0.006</td>
<td>0.001</td>
<td>0.0091</td>
<td>1.33</td>
</tr>
</tbody>
</table>

10Cr (10CrMoVNbN) is a type of advanced 10%Cr rotor steel developed within the COST (COoperation in SCience and Technology) program in the mid-1980s (known as Steel F), which has been subsequently implemented as an HP/IP rotor steel. The material in this study was taken from a full-size production rotor provided by Böhler Edelstahl, with the heat treatment methodology as: 5h austenitizing at 1000°C; water-air quenched; two-stage aging at 570°C and 675°C for 20h each and controlled cooling down to room temperature. Afterwards, the rotor was cut into blocks, from which the testpieces for this project were taken.

Another main testing material is FB2, which is regarded as one of the best candidate steam turbine rotor steels to emerge from the COST program. The most prominent difference in chemical composition is a relatively high amount of Co (1%~5%) and the addition of B (~86ppm). Co reduces the diffusivity of atoms to improve the long term microstructural stability; whereas B is able to slow down the coarsening rate of M$_{23}$C$_6$ carbides. The material in this
2.2 Short-crack creep-fatigue crack growth tests

The tensile properties of these two materials are listed in Table 2-2 for reference.

Table 2-2 Tensile properties of the tested 10Cr and FB2 materials.

<table>
<thead>
<tr>
<th>Steels</th>
<th>0.2% proof strength</th>
<th>Ultimate tensile strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>At room temperature</td>
<td>At 600°C</td>
</tr>
<tr>
<td>10Cr</td>
<td>760MPa</td>
<td>387MPa</td>
</tr>
<tr>
<td>FB2</td>
<td>686MPa</td>
<td>469MPa</td>
</tr>
</tbody>
</table>

2.2 Short-crack creep-fatigue crack growth tests

In order to investigate the mechanics and mechanisms of short-crack creep-fatigue crack growth, two series of tests were performed, on FB2 and 10Cr, with single edge-notched round-bar specimens. For each experiment, the specimen was first heated to the test temperature, cyclically deformed until the pre-defined terminating condition was reached, and then cooled down to room temperature and broken open by high cycle fatigue loading. The final crack depth was measured by an optical microscope.

Figure 2-1 (a) Details of the single edge-notched round bar specimen for FB2 and (b) set-up of the testpiece.
Testpieces were extracted from close to the periphery of two full-size production rotor forgings and machined into a cylindrical profile with shoulders, threaded ends and shallow crack starters. The parallel gauge length was 20 mm with a diameter of 8 mm, whereas the straight-front crack starter was located at the middle cross-section of the specimen with a depth of 0.2 mm and a width of about 0.3 mm for FB2 testpieces (Figure 2-1a). The diameters of the EDM (electrical discharge machined) wires used for the crack starters of FB2 and 10Cr were 0.3mm and 0.15mm, respectively. The width of the crack starter for 10Cr testpieces was 0.15mm. A high sensitivity Class 0.5 side contacting extensometer with an initial gauge length of 15 mm was used for axial displacement control.

Combined with a fully reversal servo-controlled testing machine and a resistance heating furnace, conditions for high temperature creep-fatigue loading could be achieved. Temperature was controlled in accordance with the requirements of the ASTM creep-fatigue standard, with three type-N thermocouples attached to the top, middle and bottom of the specimen gauge section to ensure a homogeneous temperature distribution throughout the test. The test set-up is schematically shown in Figure 2-1b.

Short crack growth rates were determined at 600 and 625°C in two series of displacement (nominal strain) controlled continuously cycled and cyclic/hold tests for FB2 and 10Cr. (The definition of short cracks can be found in section 1.3.1, i.e. with crack depth/total width ratio \(a/D \leq 0.25\)). Nominal strain across the specimen gauge length was controlled with a strain ratio of \(R_e = -1\) (i.e. \(\varepsilon_{\min}/\varepsilon_{\max} = -1\)). Continuously cycled tests ramped between \(\varepsilon_{\max}\) and \(\varepsilon_{\min}\) by means of a triangular waveform, mainly with a strain rate of 0.001s\(^{-1}\). (In fact, after the analysis of testpiece FB2-RB02 as shown later in Chapter 3, it was found that reducing the strain rate to \(10^{-4}\) didn’t alter stress-strain response, nor crack development much. Other factors such as dwell period and strain range are far more influential. Therefore, the rest of the tests were all done with the same strain rate of \(10^{-3}\)).

To simulate the loading condition in the targeted applications as introduced in sections 1.1 and 1.2, creep damage is mainly considered to accumulate during the hold periods rather than from a lower strain rate. Cyclic hold tests involved the same wave form, but with a 30/60 minute hold time at either peak strain in tension (\(\varepsilon_{\max}\)) or compression (\(\varepsilon_{\min}\)). Conditions for two test campaigns are shown in Table 2-3 (for FB2) and Table 2-4 (for 10Cr), with the notation ‘RB’ standing for round bar specimens. (It should be noted that the round bar testpieces were all machined with chordal crack starters, therefore they were technically SENT specimens. But in order to differentiate from the traditional SENT testpiece geometry, ‘RB’ was used here to denote the cylindrical profile). The internal designated numbers were also listed in these two tables alongside. (The final crack sizes after testing are tabulated in Table 3-5 for FB2 and Table 3-6 for 10Cr in section 3.4).

In order to accurately monitor crack development from the initial crack starter, a constant electric direct current was introduced into the specimen shoulders and passed though the testpiece. The direct current potential drop (DCPD) across the chordal crack was measured by two output wires symmetrically spot-welded to either side of the crack starter, with a spacing of 4 mm. (Details of this technique can be found in section 1.6.1). High quality ceramic parts
2.2 Short-crack creep-fatigue crack growth tests

were mounted between both specimen ends and the loading grips to provide good electrical insulation of the specimen from the test machine.7

Table 2-3 Creep-fatigue testing conditions for FB2 round bar specimens (with chordal crack starters).

<table>
<thead>
<tr>
<th>Testpiece No.</th>
<th>Internal No.</th>
<th>TEMP(°C)</th>
<th>Strain rate (s⁻¹)</th>
<th>Tension hold (min)</th>
<th>Compression hold (min)</th>
<th>Total strain range (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB2-RB01</td>
<td>HT1134</td>
<td>600</td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB02</td>
<td>HT1193</td>
<td></td>
<td>0.0001</td>
<td>0</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB03</td>
<td>HT1137</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.75</td>
</tr>
<tr>
<td>FB2-RB04</td>
<td>HT1136</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.50</td>
</tr>
<tr>
<td>FB2-RB05</td>
<td>HT1192</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.40</td>
</tr>
<tr>
<td>FB2-RB06</td>
<td>HT1138</td>
<td></td>
<td>0.001</td>
<td>30</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB07</td>
<td>HT1262</td>
<td></td>
<td>0.001</td>
<td>60</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB08</td>
<td>HT1194</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>30</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB09</td>
<td>HT1139</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB10</td>
<td>HT1140</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.75</td>
</tr>
<tr>
<td>FB2-RB11</td>
<td>HT1141</td>
<td>625</td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.50</td>
</tr>
<tr>
<td>FB2-RB12</td>
<td>HT1191</td>
<td></td>
<td>0.001</td>
<td>30</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>FB2-RB13</td>
<td>HT1195</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>30</td>
<td>1.00</td>
</tr>
</tbody>
</table>

*See Table 2-5 for conditions of interrupted tests and Table 2-6 for conditions of microstructural analysis.

Table 2-4 Creep-fatigue testing conditions for 10Cr round bar specimens (with chordal crack starters).

<table>
<thead>
<tr>
<th>Testpiece No.</th>
<th>Internal No.</th>
<th>TEMP(°C)</th>
<th>Strain rate (s⁻¹)</th>
<th>Tension hold (min)</th>
<th>Compression hold (min)</th>
<th>Total strain range (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10Cr-RB01</td>
<td>HT1349</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB02</td>
<td>HT1352</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.75</td>
</tr>
<tr>
<td>10Cr-RB03</td>
<td>HT1354</td>
<td>600</td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.50</td>
</tr>
<tr>
<td>10Cr-RB04</td>
<td>HT1355</td>
<td></td>
<td>0.001</td>
<td>30</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB05</td>
<td>HT1491</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>30</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB06</td>
<td>HT1351</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB07</td>
<td>HT1353</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.75</td>
</tr>
<tr>
<td>10Cr-RB08</td>
<td>HT1357</td>
<td>625</td>
<td>0.001</td>
<td>0</td>
<td>0</td>
<td>0.50</td>
</tr>
<tr>
<td>10Cr-RB09</td>
<td>HT1356</td>
<td></td>
<td>0.001</td>
<td>30</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB10</td>
<td>HT1358</td>
<td></td>
<td>0.001</td>
<td>60</td>
<td>0</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB11</td>
<td>HT1492</td>
<td></td>
<td>0.001</td>
<td>0</td>
<td>30</td>
<td>1.00</td>
</tr>
</tbody>
</table>

*See Table 2-7 for conditions of microstructural analysis.

With the aim of determining the final crack depth for in-test DCPD calibration, each test was interrupted before final fracture and cooled down to room temperature. Afterwards, the testpieces were broken open by means of high cycle fatigue crack growth in the remaining ligament at room temperature, which led to a flat crystalline unoxidized fracture surface. A clear
distinction could therefore be observed between oxidized crack extension at high temperature and unoxidized crack extension at room temperature. (See section 3.2 for more details). In addition to final crack depth measurement, the broken specimen halves were also used to provide samples for post-test metallurgical inspection.

2.3 Post-test examination

Some of the testpieces after short-crack creep-fatigue crack growth experiments were further cut into smaller samples to be examined under light and electron microscopes. For the already broken-open half round bar, a first sectioning in the transverse direction and a second sectioning in the axial direction were made. Due to the contained initial crack starter, the direction of crack development can easily be identified. For some other supplementary experiments where accurate values of final crack depth were not required, testpieces were directly cut in the transverse direction symmetrical to the cracking plane. In this case, a full view of the crack path could be clearly acquired. This sectioning scheme is illustrated in Figure 2-2.

In the later stage, those cut samples were mounted with resin, mechanically ground by following a stepwise sequence and polished with fine diamond slurry. Final polishing was carried out with 0.02μm colloidal silica suspension. In this way, a flat, clean, bright, scratch-free and slightly etched surface could be achieved. For the optical examination, an etching process was employed using ‘Vilella’s Reagent’ (designated as etchant 80 in ASTM E407), which was prepared with 5ml HCl, 1g picric acid and 100ml ethanol (95%). Samples were immersed and gently stirred in the etchant for about 10 to 20s until a homogeneous thin yellow
corroded layer was observed. After purging with deionized water and blowing off, the prepared samples were ready for light microscopic examination.

In order to examine those samples under the SEM, several additional steps (without etching) were applied. These included: removal of the non-electrical conductive resin substrate (to avoid excessive charging); flattening of the round back face (to avoid unwanted inclination); and pasting of the silver paint (to fix the sample onto a standard holder). The cleanliness of samples was maintained at a high level to ensure a quality surface as well as an SEM chamber free from contamination.

The EBSD measurements were made at ScopeM (Scientific Centre for Optical and Electron Microscopy), ETH, with the FEI Quanta 200 FEG microscope. This modern microscope was set-up in 2006, equipped with a TEAM Pegasus Hikari XP EBSD camera and a supporting OIM (Orientation Imaging Microscopy) software suite.

EBSPs were acquired by using a 20kV acceleration voltage, number 4 electron beam spot size, number 4 aperture size and standard 15mm working distance. Depending on the degree of recovery in microstructural evolution, different sets of scanning parameters were applied, i.e. step size from 0.01~0.8μm; and sampling area from 4x4 μm² to 20x20 μm². In special cases, some other values were adopted, while still obeying the principle that the maximum step size should be able to resolve the smallest microstructural feature, whereas the minimum sampling area should be able to include the largest targeted structural feature.

The FB2 samples in the EBSD test campaign were all selected from creep-fatigued specimens tested at 600°C with hold period in tension. The as-received sample was actually cut from the same specimen FB2-RB01, but in the threaded end far away from the deformation zone. Therefore, the microstructure there could still be regarded as being in the as-received state. In addition, some complementary samples were also taken from the sequentially interrupted specimens tested under the same condition as FB2-RB07, but terminated at the cycle numbers listed in Table 2-5. These interrupted specimens were intended to reveal an evolving microstructure in conjunction with the creep-fatigue deformation/crack propagation.

<table>
<thead>
<tr>
<th>Testpiece No. (FB2)</th>
<th>Internal No.</th>
<th>TEMP(°C)</th>
<th>Strain rate (s⁻¹)</th>
<th>Tension hold (min)</th>
<th>Total strain range (%)</th>
<th>Cycle number</th>
</tr>
</thead>
<tbody>
<tr>
<td>RB07*</td>
<td>HT1266</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>50</td>
</tr>
<tr>
<td>RB07**</td>
<td>HT1265</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>117</td>
</tr>
<tr>
<td>RB07***</td>
<td>HT1268</td>
<td>600</td>
<td>0.001</td>
<td>60</td>
<td>1.00</td>
<td>157</td>
</tr>
<tr>
<td>RB07****</td>
<td>HT1267</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>313</td>
</tr>
</tbody>
</table>

*, **, *** and**** designate the four interrupted tests with the same testing condition as for FB2-RB07.
The detailed test campaign for microscopic inspection is shown in Table 2-6 for FB2 and Table 2-7 for 10Cr. Most of the EBSD measurements for FB2 specimens were performed to find out the influence of hold periods (i.e. at the same temperature of 600°C).

Table 2-6 FB2 samples investigated by optical microscopy and electron microscopy.

<table>
<thead>
<tr>
<th>FB2</th>
<th>HT1134¹</th>
<th>HT1134</th>
<th>HT1137</th>
<th>HT1136</th>
<th>HT1138</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-received¹</td>
<td>RB01</td>
<td>RB03</td>
<td>RB04</td>
<td>RB06</td>
</tr>
<tr>
<td>Optical microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Electron microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

(1) The as-received sample was taken from the threaded end, which was subjected to a very small load.

In contrast, 10Cr samples in the EBSD test campaign were taken from specimens at both 600°C and 625°C. The as-received sample was also cut from a threaded end. Moreover, a compressive dwelled sample was measured to find out possible differences in hold time position; and a long crack growth test sample from a CT specimen was also examined to explore the microstructural variation along the crack path. (Test details of complementary long crack growth tests can be found in Appendix D).

Table 2-7 10Cr samples investigated by optical microscopy and electron microscopy.

<table>
<thead>
<tr>
<th>10Cr</th>
<th>HT1349¹</th>
<th>HT1349</th>
<th>HT1351</th>
<th>HT1353</th>
<th>HT1357</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-received¹</td>
<td>RB01</td>
<td>RB06</td>
<td>RB07</td>
<td>RB08</td>
</tr>
<tr>
<td>Optical microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Electron microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

<table>
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<tr>
<th>10Cr</th>
<th>HT1356</th>
<th>HT1358</th>
<th>HT1492</th>
<th>HT1732</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>RB09</td>
<td>RB10</td>
<td>RB11</td>
<td>CT04</td>
</tr>
<tr>
<td>Optical microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Electron microscopy</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

(1) The as-received sample was taken from the threaded end, which was subjected to a very small load.
Chapter 3

Experimental results from creep-fatigue tests

As described in Chapter 2, two materials have been tested by using instrumented cylindrical testpieces with shallow crack starters (effectively SENT type specimens). This chapter gives the testing results of these short-crack creep-fatigue crack growth experiments, focusing on the macroscopic mechanical response such as stress/strain evolution and simultaneously the crack development.

In the first section, typical stress response with ongoing cycles and hysteresis loops under different conditions are illustrated. A method of rationalizing sequential stages in stress evolution is also introduced, based on which some common features of all experiments can be found. Due to the inherent resemblance in microstructure as well as in mechanical properties, the changes in cyclic stress-strain response were quite similar for FB2 and 10Cr. However, the crack propagation paths were rather different, which is discussed in the second section. Depending on different crack front geometries, customized PD to crack depth conversion schemes were derived for each material.

In addition, several issues regarding the strain-life relation, characteristic crack depth/crack growth rate values, cyclic strain hardening exponent, crack closure, DCPD evolution during the dwell and stress partitioning are presented. Through this relatively wide-ranging investigation on the experimental results, a comprehensive view on the mechanical properties as well as the crack growth behaviour of the tested materials can be established.
3.1 Cyclic stress-strain behaviour

3.1.1 Stress evolution with ongoing cycles

All the creep-fatigue experiments in this part were conducted in nominal strain (displacement) control. Therefore, when engineering/nominal stress is plotted versus the cycle number, an expected decreasing trend of peak stresses could be observed for all tests, despite different temperatures, strain ranges, or the presence of dwell periods. A selection of graphs showing stress evolution with elapsed cycles under various testing conditions (from FB-RB01 to FB-RB08, all at 600°C) is illustrated in Figure 3-1.

Black and red curves denote the evolution of the maximum stress and the minimum stress respectively with the increasing cycle number, whereas the blue curve is the average value, e.g. \(0.5(\sigma_{\text{max}} + \sigma_{\text{min}})\). For tests with dwell, the cyan curve represents the relaxed stress value at the end of the hold period.

For all testing conditions, the maximum and minimum stresses were almost symmetrical to each other for the majority of testing life, which inferred that the net section yield in tension and compression was similar. Close to the end of experiment, the average stress value evolved toward negative, due to the fast unsteady crack development (thus a smaller remaining cross-section).

Under displacement control, the maximum stress value represents the resistance of the remaining ligament to elastic/plastic deformation, while the minimum stress further takes crack closure into consideration, leading to a more negative value. Therefore, by looking purely at peak stress evolution, some information regarding crack development could already be obtained.

The effects of total strain range, strain rate and dwell period could be seen by comparing different plots in Figure 3-1. For example, FB2-RB01 (Figure 3-1a) and FB2-RB02 (Figure 3-1b) had different strain rates (0.001s\(^{-1}\) and 0.0001s\(^{-1}\)), but the stress evolutions were quite similar. Of course, due to the 9 times longer testing period the stress dropped slightly more quickly in FB2-RB02 at the same cycle number, but this effect was not significant compared to other testing parameters, e.g. total strain range or hold period. (The detailed testing conditions are listed in Table 2-3).

Taking FB2-RB01 (Figure 3-1a), FB2-RB03 (Figure 3-1c), FB2-RB04 (Figure 3-1d) and FB2-RB05 (Figure 3-1e) as examples, the total strain ranges were 1%, 0.75%, 0.5% and 0.4% respectively. The maximum and minimum stress values were respectively smaller with a smaller total strain range, while the number of cycles to reach unstable crack development increased considerably (by an order of magnitude).
3.1 Cyclic stress-strain behaviour

Figure 3-1 Stress evolution for short-crack creep-fatigue crack growth experiments of FB2 under various conditions.
Experimental results from creep-fatigue tests

Plots of FB2-RB06 (Figure 3-1f), FB2-RB07 (Figure 3-1g) and FB2-RB08 (Figure 3-1h) show creep-fatigue tests with 30min tensile dwell, 60min tensile dwell and 30min compressive dwell. One interesting feature for them is that the initial peak values at the beginning of tests are shown as individual dots, which illustrate a very strong softening effect right from the start. Apart from the beginning and last stages, the cyan curves develop nearly parallel to the black curves. This inferred that in a single experiment, the degree of stress relaxation during each cycle was almost the same. In addition, the effect of the 60min hold time on further reducing the number of cycles was apparent.

3.1.2 Partitioning scheme for $\sigma_{\text{max}}$-N curves

Since the testing duration and terminating criteria were different for various experimental conditions, a partitioning approach was devised to characterize each stress-cycle curve and provide a unified baseline for comparison of hysteresis loops and further discussion of crack development. This concept is illustrated by the endurance curve from FB2-RB01, as shown in Figure 3-2a.

For strain-controlled creep-fatigue crack growth tests, precipitation strengthened alloys at high temperatures usually exhibit a common cyclic softening behaviour. As expected, the investigated FB2 (9%Cr steel) showed a relatively rapid softening regime at first, and then a steadier softening regime before the unstable load drop (Figure 3-2a). The initial rapid softening in the first regime was mainly due to strong dynamic recovery, as a result of reorganization of the dislocation microstructure and the development of a subgrain structure. The stress response then entered a stable decreasing regime, resulting from a combination of damage development and the recovery process. After that, the stress drop increased as a consequence of the fast increasing compliance (due to the increased extent of crack development).

![Figure 3-2](image)

Figure 3-2 Schematic representation of typical stress-strain response recorded during short-crack creep-fatigue crack growth tests: (a) characteristic points and (b) corresponding hysteresis loops at 600°C.

It was then possible to represent the cyclic response by dividing the peak stress curve with characteristic points (red dots in Figure 3-2a): (Point 1) the beginning of the steady softening
3.1 Cyclic stress-strain behaviour

regime; (Point 2) the mid-life cycle (i.e. 0.5 \(N_{2\%}\)); (Point 3) the end of the steady softening regime; and (Point 4) the number of cycles to 2% load drop (i.e. \(N_{2\%}\)), which is a commonly adopted creep-fatigue crack initiation criterion.\(^{22,174}\) The corresponding hysteresis loops are plotted in Figure 3-2b, which also exhibit a gradual softening of peak stresses.

Comparing the loops inside Point 1 to Point 4 and outside (from first cycle to Point 1 and from Point 4 to the end of test), it is apparent that the largest softening effect and change of compliance occurred in those unsteady regimes, although their number of cycles is less than that in the steady regime. This potentially infers that if the stress-strain response is adopted to describe crack development behaviour, the most effective regime (i.e. stable short crack growth) should lie within the range between Point 1 to 4.

In order to get a clear picture of how different testing conditions have influenced the shape and evolution of stress-strain response in a single cycle, hysteresis loops for characteristic cycles are plotted in Figure 3-3. Although the main interest is within Point 1 to Point 4, loops from the last cycles are also given as a reference. Three prominent phenomena could then be observed:

(1) The first phenomenon is the influence of testing strain range on the hysteresis loop shape. At \(\Delta \varepsilon_t = 1\%\), the large plastic deformation was associated with a wide plastic strain range (interception distance at \(\sigma = 0\)), together with an elastic stress range of around 350MPa. When the total strain range was decreased, e.g. \(\Delta \varepsilon_t = 0.75\%\) for FB2-RB03 (Figure 3-3c), \(\Delta \varepsilon_t = 0.5\%\) for FB2-RB04 (Figure 3-3d) and \(\Delta \varepsilon_t = 0.4\%\) for FB2-RB05 (Figure 3-3e), a significant reduction of plastic deformation could be observed, whereas the elastic stress range remained almost the same. The shift of the driving force (for crack development) from plasticity to elasticity was responsible for the dissimilar crack growth behaviour for different \(\Delta \varepsilon_t\) (to be shown in a later stage). In addition, when the total strain range was reduced, the degree of stress softening from characteristic Point 1 to Point 4 also decreased, which inferred that the reversed plasticity was strongly linked to the mechanism behind softening, i.e. microstructural evolution.

(2) The second phenomenon is the change of hysteresis loop shape while a hold period was introduced. The maximum and minimum plastic strain values were no longer symmetrical, due to the accumulated visco-plasticity during the hold period. This led to an increase of the internal area of hysteresis loops, which was further related to the dissipated energy per cycle. (This difference can also be illustrated with the schematic plots of Figure 1-13 and Figure 1-15 in Chapter 1. Later on it will be shown in Chapter 5 that the SEDF model takes this difference into consideration to model crack development under different testing conditions). Apart from this bulged area, the shapes of loops are analogous to the test without hold period at the same \(\Delta \varepsilon_t\) (FB2-RB06, 07 and 08 compared with FB2-RB01), which exhibits a good consistency of the stress-strain response before 2% load drop.
Experimental results from creep-fatigue tests

Figure 3-3 Evolution of hysteresis loops for (SENT) creep-fatigue testpieces of FB2 under various conditions.
(3) The third phenomenon is related to the crack closure effect, which can be reflected by a cusp at the compressive loading ramp in a single cycle. As shown in Figure 3-3, almost all of the final cycles had distinct cusps, whereas before the 2% load drop point, crack closure was almost invisible. This is also consistent with the literature that for short crack development, crack closure generally occurs after 25% of the cross-section area is consumed.\textsuperscript{27} (As will be shown in section 3.2.2, 2mm crack development is approximately equal to a 2% load drop for the investigated specimens). Therefore, it was legitimate to affirm that at least in the steady softening regime, the influence of crack closure on crack growth behaviour could be neglected. Only after the 2% load drop point, this effect began to build up to cause a significant change in crack opening/closure stress. In other words, compliance was not an effective method of detecting the closure effect for very small cracks in this study.

Creep-fatigue tests were also conducted at 625°C for FB2, with similar cyclic stress-strain responses to these displayed in Figure 3-1 and Figure 3-3.

For the other test material, 10Cr, the same testing scheme was applied, as simply illustrated by the two examples in Figure 3-4. When the temperature was increased from 600°C to 625°C, a further decrease of endurance was expected, as shown in the case of 10Cr-RB06 (Figure 3-4c). However, the difference was not significant.
One distinction could be noticed here by comparing Figure 3-4 with Figure 3-1. As introduced in Chapter 2, 10Cr-RB01 was the experimental counterpart of FB2-RB01 (Table 2-3 and Table 2-4). However, 10Cr-RB01 (Figure 3-4a) had a longer steady softening stage than FB2-RB01 (Figure 3-1a), which potentially meant that either the microstructure evolved or the crack grew more slowly in 10Cr. In fact, since the notch root radius was 0.15mm for 10Cr specimens (whereas it was 0.3mm for FB2), the true fatigue crack might have initiated even sooner (if there was any notch root sensitivity on crack initiation) in 10Cr. It might be already possible from the analysis on the endurance curves to infer that 10Cr had slower crack propagation than FB2. Nevertheless, the influences of crack closure effect on hysteresis loop shapes were similar for testpieces from both materials, despite the different notch radii.

Characteristic stress values can be found in Table 3-3 for FB2 and Table 3-4 for 10Cr in section 3.4.

3.1.3 $\Delta \varepsilon_t$-N relationship

For traditional low cycle fatigue tests using unnotched specimens, the $\Delta \varepsilon_t$ – $N$ curve is usually a good indication of a material’s resistance to crack initiation under various conditions. Likewise, the common criterion as a 2% load drop from the endurance curve was also adopted within the scope of this study, where the ‘crack initiation’ period (short crack propagation) was defined (characteristic point 4 in Figure 3-2a).

![Figure 3-5 Influence of total strain range, temperature and hold time on the number of cycles to 2% load drop in FB2 SENT specimens with $a_0=0.2mm$ (data points for $\Delta \varepsilon_t=1%$ strain are offset for clarity, and a reference line from tests with smooth specimens is also given).](image)

The influence of total strain range, temperature and hold time on the number of cycles to 2% load drop in FB2 specimens with short edge chordal crack starters is shown in Figure 3-5.
3.1 Cyclic stress-strain behaviour

(Several experiments were conducted at $\Delta \varepsilon_r = 1\%$ and that led to some overlapping of symbols. For clarity those points are offset to a certain extent).

1) Total strain range $\Delta \varepsilon_r$ seemed to have a predominant influence on this ‘crack initiation’ period, as the number of cycles to 2% load drop was significantly larger that for lower strain ranges. For example, the $\Delta \varepsilon_r = 0.4\%$ specimen had a $N_{2\%}$ by almost an order of magnitude larger than that of the $\Delta \varepsilon_r = 1\%$ specimen.

2) By comparing hollow circular symbols (denoting tests at 625°C) and hollow squared symbols (denoting tests at 600°C), a deterioration effect of temperature can be observed, as hollow circular symbols are all located to the left side of hollow squared symbols.

3) The addition of 30min hold time largely reduced the number of cycles until 2% load drop, as shown by those black non-hollow symbols at $\Delta \varepsilon_r = 1\%$. Besides, the increase of 30min hold period to 60min brought about an even shorter time to crack initiation (the red symbol in Figure 3-5), which was apparently due to the fact that more creep damage was accumulated in each cycle when the hold time was longer.

4) It is also apparent in Figure 3-5 that the reduction of $N_{2\%}$ in compressive dwell was more prominent than in tensile dwell. As explained in sections 1.3.3 and 1.3.4, two factors might have contributed to this effect. Indeed, from the stress evolution (illustrated in Figure 3-1) it can be observed that the shift of mean stress took place in tests with hold time, where a compressive dwell generated a positive mean stress (although this mean stress was comparatively small). In addition, as shown from the microscopic investigation in Chapter 4, thick oxidized layers were also observed on specimen surfaces. Therefore, it is speculated that both mechanisms could lead to the fact that the number of cycles to 2% load drop was smaller in compressive hold specimens than that in tensile hold specimens.

5) In addition, a reference $\Delta \varepsilon_r - N$ curve for smooth FB2 specimens at 630°C is also plotted in Figure 3-5 (from Ref.175), which situated well beyond all other symbols. This was expected, since the testpieces within this study were already manufactured with crack starters (to act like crack initiation sites). Evidence here exhibits an immense impact of crack starters on actual crack initiation stage (even though the testing temperature is higher).

The $\Delta \varepsilon_r - N$ curve for the 10Cr specimens is plotted (Figure 3-6). Despite the similar influence of temperature, total strain range and hold time, there were in fact some differences in the magnitude of the influence. For example, although a rise of temperature from 600°C to 625°C indeed shortened the number of cycles to 2% load drop, the degree of this reduction was much smaller for 10Cr than for FB2. This presumably indicates that 10Cr is less susceptible to the temperature change from 600°C to 625°C (as far as creep-fatigue deformation is concerned).

One anomaly that could be observed in Figure 3-6 is that for the two continuously cycled tests with $\Delta \varepsilon_r = 1\%$, the one at 600°C (10Cr-RB01) had a slightly smaller number of cycles to reach
Experimental results from creep-fatigue tests

$N_{2\%}$, than its counterpart at 625°C (10Cr-RB06). The reason behind this was probably a variation in material properties. As will be shown later in Figure 4-27 in Chapter 4, the substructure of 10Cr-RB01 actually evolved faster than 10Cr-RB06.

By comparing the numbers of cycles to 2% load drop in both materials (Figure 3-5 and Figure 3-6), it is obvious that 10Cr specimens had overall longer life time than FB2 specimens. As explained in the previous context, this could either be caused by a slower microstructural evolution, or a shorter crack initiation/propagation phase.

3.2 Crack development

The basics and guidelines of DCPD method have been briefly introduced in section 1.6.1. By applying this technique, DCPD values were continuously recorded during the experiments. In order to transform voltage values into crack depth values, a calibration process was carried out for this particular specimen geometry with different crack front shapes. It was then possible to systematically analyse the pattern of crack development under various testing conditions.

3.2.1 Calibration of DCPD to crack depth

In FB2 testpieces, post-test inspection indicated that the developed crack front remained approximately straight and parallel to the root of the starting chordal crack. (A summary of final crack depths for FB2 testpieces can be found in Table 3-5 in section 3.4). A particular example is shown in Figure 3-7a, which had the longest propagated crack depth in all experiments (testpiece FB2-RB06). Even in this case, the final crack front was almost straight, and the fracture stages could be distinguished from different surface roughness and colours. As
3.2 Crack development

schematically displayed in Figure 3-7b, slow creep-fatigue crack propagation at elevated temperature occupied the largest fraction. During the break-open process at room temperature, high cycle fatigue loading was imposed, which generated a relatively smooth bright fracture surface (i.e. the ‘white ribbon’ in Figure 3-7a). Ultimately, the remaining ligament could not sustain the load and fast rupture took place.

Figure 3-7 Example showing a straight final crack front developed from a chordal crack starter in a FB2 specimens: (a) actual example fracture surface, (b) schematic representation.

This type of surface pattern applied for all FB2 specimens, thus a general representation of crack development path could be obtained, as depicted in Figure 3-8.

Figure 3-8 Schematic representation of crack front development from a chordal crack starter for FB2 specimens. (Direction from bottom to top).

In fact, there were also other factors adding up to the change of crack tip electrical streamlines, apart from the crack front shape. Complication could come from oxidation, microstructural evolution (e.g. dislocation condition), crack branching etc. (to be further discussed in section...
Experimental results from creep-fatigue tests

3.2.2 and in Appendix C), but they were not as influential as the reduction of cross-section. As briefly introduced in section 1.6.1, it was reported that a transient increase or decrease of initial PD value can occur at the beginning of creep-fatigue crack growth experiments for certain materials. However, in this study neither behaviour was observed. It was thus legitimate to assume that $\bar{V}_{0.2}$ corresponded to $a_0$ (examples are also given in Figure 3-10 and Figure 3-13).

It was therefore decided to use a two-stage calibration method to improve the effectiveness of DCPD to crack depth calibration in accordance with the adopted specimen geometry and crack fronts (e.g. 10Cr specimens had rather complex crack development paths, which is shown later in this section) in this study. The first stage was to derive a calibration formula based on the fact that the increase of PD value (material resistance) came from the decrease of the cross-section (due to crack propagation). For FB2 specimens the crack front was assumed to be always straight (i.e. evidence from Figure 3-7). It was therefore relatively convenient to deduce the relationship based on Figure 3-8:

$$\left(1 - \frac{0.9933\bar{V}_{0.2}}{\bar{V}}\right) \pi = \theta - \frac{\sin 2\theta}{2}$$

(3-1)

where

$$\theta = \arccos(1 - \frac{a_{\text{temp}}}{r})$$

(3-2)

$\bar{V}_{0.2}$ and $\bar{V}$ denote the initial and instantaneous PD values, $\theta$ is the included angle in Figure 3-8. The value of 0.9933 came from the ratio of area fraction excluding the crack starter divided by the full cross-section. In this way, the temporary depth, $a_{\text{temp}}$ was obtained purely from the change of electrical resistance.

In a second stage, this temporary value $a_{\text{temp}}$ needed to be revised by breaking specimens open after testing and accurately measuring the experimental final crack depth $a_f$. (The initial and final crack depths were unambiguous values. Final crack depth can be found in Table 3-5 for FB2 in section 3.4). Thus, evolution of the genuine crack depth $a$ could be determined with the following correction formula:

$$a = (a_{\text{temp}} - a_0) \cdot \frac{a_f - a_0}{a_f - a_{\text{temp}}} + a_0$$

(3-3)

Here $a_0$ corresponds to the initial crack depth, i.e. $a_0 = 0.2\,\text{mm}$; $a_{\text{temp}}$ is the temporary final crack depth calculated from equation (3-2). This correction formula can scale up and down the converted crack depth values according to the two fixed boundary values (i.e. $a_0$ and $a_f$), and modify each intermediate value in a linear scale.

For FB2 specimens, the effectiveness of the stage I calibration is shown in Figure 3-9a. The red curve is from equation (3-1) and (3-2), which displays the derived temporary crack depth $a_{\text{temp}}$. This plot also demonstrates that the final crack depth values $a_f$ obtained from post-test observation lay well along this conversion curve, while the relatively small scatter could be
3.2 Crack development

accommodated by a further correction step (i.e. equation (3-3)). In Figure 3-9b, two examples showing upscaling and downscaling of the conversion curve are also presented (final crack depth marked with arrows from Figure 3-9a). By employing this stage II calibration, a higher flexibility as well as accuracy could be achieved for each specimen.

After this two-stage calibration process, DCPD voltages could be converted to crack depth values. Examples are given here to show the general pattern of DCPD development and converted crack depth values. In Figure 3-10a, normalized PD values for FB2-RB01 (continuously cycled) and FB-RB07 (with 1h hold time) are plotted against cycle numbers. (In each cycle, only the maximum PD value, i.e. at the peak stress when the crack was fully open was utilized in this process). It can be seen that PD increased almost immediately and monotonically after the start of the test. There were some scattering of the raw data, thus a smoothing scheme was also applied, as shown by the red curves in Figure 3-10a. By employing the above mentioned two-stage calibration, crack depth at each intermediate cycle was obtained, which is illustrated in Figure 3-10b.

Figure 3-10 (a) Comparison of final crack depth observations made during post-test inspection with those predicted from stage I DCPD calibration and (b) further correction/scaling with stage II calibration for FB2 specimens.

Figure 3-10 Examples showing the evolution of (a) DCPD values; and (b) converted crack depth for two FB2 specimens.
Due to the fact that the recorded change of electrical streamline (i.e. change in PD) was strongly dependent on the experimental set-up of the voltage output leads, the variation of measured absolute PD values could have different growth spans. (In practice, errors such as welding position could have such an influence). Therefore in Figure 3-10, although the difference of PD values between two specimens is large, the discrepancy of crack depths is smaller.

Different from FB2 testpieces, circumstances in 10Cr specimens were rather complicated. Most of the observed final crack fronts were part-elliptical rather than straight, and the degree of curvature was testpiece-dependent (although some of the specimens still had quasi-straight final crack fronts). Cracks developed from the initial straight crack starters, with increasingly bulging frontiers in the middle section which finally led to the part-elliptical shape. This scenario was schematically illustrated in Figure 3-11a. However, there was no unified crack evolution path or unique aspect ratio. (Despite differences in crack development path/shape between FB2 and 10Cr specimens, a unified criterion of using the largest crack depth, i.e. maximum cracked displacement from the surface was employed to indicate the most dangerous situation).

In order to keep a consistent conversion formula for this batch of 10Cr testpieces, all the experimental final depths/shapes of final crack fronts in 10Cr specimens were statistically processed. Characteristic values for determining cracked area fraction (shown as shaded region in Figure 3-11b) are listed in Table 3-1.

Final (maximum) crack depths and $S_2$ values were measured directly, whereas the values of $S_1$ and $\theta$ were further calculated. In this manner, it was possible to derive a function as an average indication of cracked area with respect to crack development, by curve fitting. Thus, equation (3-1) and (3-2) could be rewritten into equation (3-4) and (3-5) for 10Cr.
### 3.2 Crack development

Table 3-1 Characteristic parameters to determine final crack front shapes in 10Cr specimens.

<table>
<thead>
<tr>
<th>Testpiece No.</th>
<th>Final crack depth (a_f) (mm)</th>
<th>(S_2) (mm)</th>
<th>(S_1) (mm)</th>
<th>Ratio (S_1/S_2)</th>
<th>(\Theta) (rad)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10Cr-RB01</td>
<td>3.48</td>
<td>3.55</td>
<td>1.32</td>
<td>0.37</td>
<td>1.09</td>
</tr>
<tr>
<td>10Cr-RB03</td>
<td>2.36</td>
<td>2.98</td>
<td>1.03</td>
<td>0.34</td>
<td>0.84</td>
</tr>
<tr>
<td>10Cr-RB04</td>
<td>1.96</td>
<td>2.38</td>
<td>1.17</td>
<td>0.49</td>
<td>0.64</td>
</tr>
<tr>
<td>10Cr-RB05</td>
<td>3.80</td>
<td>3.80</td>
<td>1.05</td>
<td>0.28</td>
<td>1.25</td>
</tr>
<tr>
<td>10Cr-RB06</td>
<td>3.34</td>
<td>3.37</td>
<td>1.49</td>
<td>0.44</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB07</td>
<td>3.04</td>
<td>3.37</td>
<td>1.19</td>
<td>0.35</td>
<td>1.00</td>
</tr>
<tr>
<td>10Cr-RB08</td>
<td>2.72</td>
<td>3.22</td>
<td>1.08</td>
<td>0.34</td>
<td>0.94</td>
</tr>
<tr>
<td>10Cr-RB09</td>
<td>2.83</td>
<td>2.93</td>
<td>1.55</td>
<td>0.53</td>
<td>0.82</td>
</tr>
<tr>
<td>10Cr-RB10</td>
<td>1.69</td>
<td>2.86</td>
<td>0.48</td>
<td>0.17</td>
<td>0.80</td>
</tr>
<tr>
<td>10Cr-RB11</td>
<td>3.16</td>
<td>3.68</td>
<td>0.73</td>
<td>0.20</td>
<td>1.17</td>
</tr>
</tbody>
</table>

\[
\left(1 - \frac{0.9933V_{0.2}}{V}\right)\pi = \theta - \sin 2\theta \cdot \frac{9.021 - 15.697e^{-0/0.576}}{16}
\]  \hspace{1cm} (3-4)

where

\[
a_{\text{temp}} = 4(1 - \cos \theta) + (1.213 - 7.026e^{0/0.18})
\]  \hspace{1cm} (3-5)

The effectiveness of this conversion is shown in Figure 3-12, which exhibits some more divergence compared with Figure 3-9a. This scattering was mainly due to the inconsistency of crack front shapes in 10Cr specimens.

![Figure 3-12](image)

**Figure 3-12** Comparison of final crack depth observations made during post-test inspection with those predicted from DCPD measurements for 10Cr specimens.

By employing the stage II correction process (equation (3-3)), this discrepancy could be minimized. The calculated temporary initial crack depth \(a_{0_{\text{temp}}}\) and final crack depth \(a_{f_{\text{temp}}}\) were rescaled according to defined crack starter length \(a_0 = 0.2\,mm\) and measured final crack...
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depth \( a_f \). An example of this process for 10Cr specimens is shown in Figure 3-13a and Figure 3-13b, by the testpieces 10Cr-RB01 and 10Cr-RB08.

Since the definition of crack depth was purely based on the maximum displacement the crack front has swept through, the influence of actual propagation path on DCPD→crack depth conversion was the key issue on the success of the calibration process. Thus, an estimation of the accuracy of this two-stage calibration scheme was necessary.

As discussed above, crack fronts for FB2 specimens were quite consistent, while those for 10Cr specimens exhibited some variations. Thus the main issue was to find out the boundary values of calibrated crack depth values for 10Cr testpieces. In other words, the dissimilarity should be compared, when the calibration curve for straight-front cracks (red curve in Figure 3-9) was used for 10Cr testpieces (which actually had part-elliptical front cracks, e.g. in Figure 3-11).

![Figure 3-13](image)

Figure 3-13 Examples of the influence of crack propagation path on (a) calibrated crack depth values and (b) crack growth rates; Influence of calibration process on (c) calibrated crack depth values and (d) crack growth rates. (Solid curves represent properly calibrated crack depth values; and dashed lines denote inappropriately calibrated values).

This is exemplified in Figure 3-13c, where the solid curves represent the properly calibrated crack depth values, and dashed lines denote those inappropriately calibrated values. The only unaffected points would be the crack depth values at the beginning and end of the tests with actual measured values (reference points in the correction process), therefore the largest deviation in crack depth occurs at intermediate cycles. In this example, the maximum deviation
3.2 Crack development

is as large as 0.19mm for 10Cr-RB01 and 0.11mm for 10Cr-RB08. (These values are not really the error bands, but the extreme cases to show how big the influence of crack front shape can affect the calibration process).

While the interpreted values of crack depth from these two calibration processes deviated up to about 0.2mm, the differences in slopes also caused apparent discrepancies in crack growth rates as can be seen in Figure 3-13d. In both cases, the largest difference occurred at the end cycle, causing a 1.2µm deviation for 10Cr-RB01 and 0.3µm for 10Cr-RB08.

In all, the most important conclusion from this assessment is that it is absolutely crucial to take the true cracking path into consideration during the calibration process.

3.2.2 Characteristic values of crack depth and crack growth rate

According to the concept defined in Figure 3-2a, cyclic stress evolution for creep-fatigue deformation could generally be characterized by four points with regard to the investigated specimen/test set-up. Similarly, it was necessary to find out whether these characteristic points could also accurately catch the pattern with respect to crack propagation. Therefore, discrete crack depth values are plotted at those characteristic points for both FB2 and 10Cr specimens, as shown in Figure 3-14. (Tests at 600°C are represented by red symbols, whereas tests at 625°C are represented by blue symbols).

For FB2 (Figure 3-14a), except for the test with $\Delta\varepsilon = 0.4\%$ at 600°C, crack depths propagated to ~0.5mm at the start of the steady softening regime (Point 1); to ~0.75mm at midlife (Point 2); to ~1.75mm at the end of the steady softening regime (Point 3); and to ~2mm at $N_{2\%}$ (Point 4). This illustrated a good consistency between cyclic stress response and crack propagation behaviour for the given geometry.

For 10Cr (Figure 3-14b), similar values could be observed at characteristic points, except that at Point 3 the average crack depth value was slightly smaller than that in FB2. In addition, a larger scattering of data points existed in 10Cr, primarily resulting from the complex propagation paths (shown in section 3.2.1).

In both graphs, there seems to be no systematic influence of temperature on crack depth at characteristic points, although it is shown in Figure 3-5 and Figure 3-6 that a higher temperature could lead to a shorter life to 2% load drop point. Another noteworthy point was that in line with the definition of ‘crack initiation’ (propagation of a relatively short crack), i.e. 2% load drop of the peak stress curve (dashed line in Figure 3-2a), crack depth values were all around 2mm. This is in accordance with the findings of others, where the range of short cracks is limited to the cracked ratio $a/D = 0.25$. Therefore, in the later stage of short crack growth modelling (Chapter 5), only the crack growth behaviour within the regime of $a \leq 2mm$ is considered.
Experimental results from creep-fatigue tests

By differentiating smoothed crack depth values against cycle numbers, crack growth rates were obtained and their values at characteristic points are shown in Figure 3-15 and Figure 3-16, which exhibit a general trend of relatively slow crack propagation at the rapid softening stage, followed by a faster propagation at the steady-state (saturation) stage.

Obviously, total strain range and hold time were the most dominating factors to influence crack propagation rate, while the effect of temperature was not that regular. Take the characteristic Point 3 in Figure 3-15 as an example. Despite the temperature and the position of dwell, the crack growth rates of the four dwell specimens had approximately the same value (0.014mm/cycle), which was almost two times the value under similar testing conditions without hold period. Similarly, when the total strain range was reduced, the crack growth rate also decreased.
The effect of the temperature change from 600°C to 625°C on crack growth properties was not straightforward from Figure 3-15. This was at least partly due to the wavy profiles of the actual continuous crack growth rate curves (e.g. those in Figure 3-13d), which were not shown in such a figure plotting only discrete values. Additionally, by comparing the number of cycles to reach a characteristic crack length, e.g. 2mm, it was clear that for most cases, increasing temperature (while keeping other test parameters the same) led to a reduced cycle number, which in turn indicated a faster crack propagation rate at a higher temperature.

In 10Cr specimens, the influence of dwell period and total strain range on crack growth rate was similar to that in FB2 specimens. However, by comparing the actual values of crack growth rates, 10Cr testpieces (Figure 3-16) had smaller values than FB2 testpieces (Figure 3-15),
especially after the characteristic point 2. This potentially demonstrated a high resistance in 10Cr against crack development under creep-fatigue deformation condition.

In Table 3-5 and Table 3-6 in section 3.4, crack growth rates at three different crack depth (i.e. at 0.5mm, 1.0mm and 2.0mm) can be found.

3.2.3 DCPD evolution during hold periods

Apart from the variation of crack depth with the increasing cycle number, it was also interesting to investigate what was happening during individual hold periods. As mentioned before in section 1.6.1, the DCPD technique is generally recognised as a robust method to monitor crack development as a function of cross-section variation. However, it has been realized that DCPD value cannot be directly associated with crack depth values on a small scale, because other factors could also influence the output voltage values, especially under creep-fatigue-oxidation interaction conditions. (A rather hypothetical and preliminary discussion on this issue is included in Appendix C).

This section is therefore concerned with the significance of voltage changes observed during hold times. In addition, what these changes meant with respect to the increasing voltage/crack depth in adjacent cycles is also studied.

One major difficulty in resolving this issue comes from the fact that, when using a DCPD device, the noise signals should be suppressed while the sensitivity should remain as high as possible. Therefore in this study, electrical insulation between the specimens/grips as well as thermal protection on input/output wires were carefully applied, which were supposed to exert a minimum external influence on voltage fluctuation. Meanwhile, no averaging technique was used in the data acquisition process to avoid any omission of the true signals. Thus the attained raw voltage values were instantaneous values with a relatively large scatter. The way to deal with this scatter was to employ a signal processing technique, i.e. Savitzky-Golay smoothing, to reduce the noise ratio to an acceptable level.

An example of stress and voltage evolution (slightly smoothed) during the dwell period of one cycle is shown in Figure 3-17. \( V_{\text{max}} \), \( V_a \) and \( V_b \) were the voltage values at the maximum load, the beginning of the dwell and the end of dwell, respectively. (In practice, only \( V_{\text{max}} \) in each cycle was converted into crack depth values by the calibration process, as explained in section 3.3.1).

This cycle came from the test with 30min hold time in compression (FB2-RB08), near the beginning of the steady softening stage. The horizontal axis denotes the lapsed time (seconds) in one cycle, with a break region omitting some fraction during the dwell in order to put the whole figure in a proper scale.
3.2 Crack development

Figure 3-17 Typical evolution of stress and voltage values during one cycle with 30min compressive hold.

The blue curve represented the stress evolution, which exhibited typical fast-to-slow relaxation behaviour as expected; the black curve demonstrated how voltage value varied during the ramping period as well as during the dwell. It can be seen that in general, DCPD value was proportional to stress/strain values. This is mainly due to the dislocation condition and the opening/closure of crack fronts.

In this example, there was a small increase of PD value during the hold time ($V_0 > V_a$). If this small increase could be interpreted directly into crack depth values, one might be able to attribute the enhanced crack growth behaviour in dwelled specimens to the hold period itself (in other words, real creep crack growth). However, this was found to be not true for short-crack creep-fatigue crack growth tests in this study.

By cautiously smoothing the raw data in the characteristic cycles, relative voltage change was calculated to be $\Delta V = (V / V_a - 1) \times 100\%$, which denoted the percentage change of voltage readings with respect to its initial value at the start of dwell (definition of terms was in accordance with Figure 3-17). Relative voltage changes for two compressive and two tensile dwelled tests are shown in Figure 3-18.

For each experiment, four characteristic cycles were chosen to represent the change of PD during dwell periods. Despite the wavy profile of the curves (which was owing to a complex interaction between different influential factors on voltage output values), the most obvious pattern could be observed: the variation of voltage values was small during the dwell periods, i.e. less than ±0.04% generally.

Figure 3-18a and Figure 3-18b demonstrate what happened during the compressive dwell: the change of voltage value was relatively large at the beginning of the steady softening regime (the instantaneous DCPD reading in Figure 3-17 corresponds to the black curve in Figure 3-18a), and continuously decreased from positive to negative under further deformation.
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On the other hand, Figure 3-18c and Figure 3-18d came from two tensile dwelled tests, and the common feature was that although almost all the voltage values were located in the negative regime, they seemed to evolve in a positive-going direction in the four characteristic cycles.

![Figure 3-18 Relative voltage change in characteristic cycles for two compressive dwelled tests: (a) FB2-RB08 (b) 10Cr-RB05; and two tensile dwelled tests: (c) FB2-RB06 (d) 10Cr-RB04.](image)

Due to the fluctuation of the voltage values, it was perhaps more reasonable to define a parameter to characterize the average change of instantaneous DCPD readings in each cycle. Therefore, the mean voltage change during the dwell $\Delta \bar{V}_{mean}$ was proposed, in the form of

$$\Delta \bar{V}_{mean} = \text{Avg.}(\bar{V} - \bar{V}_a)$$  \hspace{1cm} (3-6)$$

It was then possible to calculate $\Delta \bar{V}_{mean}$ for every cycle (hold period) along with the crack development, as shown in Figure 3-19 for FB2 and Figure 3-20 for 10Cr. The black upward triangles represent tests with tensile hold, whereas the blue downward triangles signify tests with compressive hold. The red curves were calculated from the voltage change between adjacent cycles (i.e. $\bar{V}_{\text{max}(i+1)} - \bar{V}_{\text{max}(i)}$) as illustrated in Figure 3-17.
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Note that the longitudinal coordinates in Figure 3-19 and Figure 3-20 are different from that in Figure 3-18. Absolute voltage values are displayed, without being normalized to their initial values.

![Figure 3-19](image1.png)  
**Figure 3-19** Mean voltage change during the dwell compared with the voltage change between adjacent cycles for FB2 specimens.

![Figure 3-20](image2.png)  
**Figure 3-20** Mean voltage change during the dwell compared with the voltage change between adjacent cycles for 10Cr specimens.

In accordance with the findings in Figure 3-18, three main observations could be made together with Figure 3-19 and Figure 3-20:

1. For compressive dwelled specimens, the mean voltage change $\Delta V_{\text{mean}}$ during the dwell was initially positive and evolved in the direction of continuous reduction. After approximately 1mm crack depth, the value was already negative (i.e. creep deformation became more significant at a larger crack depth).

2. For tensile dwelled specimens, the mean voltage change $\Delta V_{\text{mean}}$ was always negative in the short crack regime (i.e. until 2mm crack depth). More specifically, its value was lowest at the start of test, and constantly increased toward positive. It seemed that if this trend
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could be kept when crack has developed further (i.e. beyond 2mm), the mean voltage change during the dwell might finally be positive. This was indeed observed in two of the tensile dwelled tests where the crack had developed long enough.

(3) Irrespective of the position of hold period, the value of mean voltage change $\Delta F_{\text{mean}}$ during the dwell was mostly between $-1.0 \times 10^{-3}$ and $0.5 \times 10^{-3}$. Meanwhile, the absolute voltage change between adjacent cycles increased from zero up to $4 \times 10^{-3}$. This implied that the development of DCPD values (to a certain extent, crack growth) took place mainly in the ramping period rather than in the hold period.

Actually, this analysis also demonstrated that apart from the real crack growth, there were also other factors which could contribute to the voltage variation during dwell (i.e. the relationship between voltage value and crack depth was not one to one). And one future task is to identify which and how those factors influenced the acquired data. As a starting point, a preparatory discussion is enclosed to this thesis in Appendix C. Nevertheless, the most important conclusion in this section is that the voltage variations during hold periods were much smaller than those between respective cycles. This supports the conclusion that instead of direct creep crack growth during the hold time, the enhancement of apparent crack growth rate was due to accelerated fatigue crack propagation as a result of prior creep and strain enhanced oxidation at the crack tip (also shown later in Chapter 4).

3.3 Further discussion

3.3.1 Cyclic strain hardening exponent in the midlife cycle

As illustrated in Chapter 1, the cyclic strain hardening exponent can characterize a material’s resistance to cyclic plastic deformation, and assist crack growth modelling in the later stage.

![Figure 3-21 Illustration of the regression process for cyclic strain hardening exponents of (a) FB2 and (b) 10Cr.](image)

By plotting the gross section (load divided by the initial cross-section area) and the net section stress ranges (load divided by the instantaneous minimum cross-section area calculated from
3.3 Further discussion

the known crack depth) and plastic strain ranges at midlife cycles and carrying out power-law curve fitting, cyclic strain hardening exponents were obtained. The results for FB2 and 10Cr at 600°C and 625°C are shown in Figure 3-21.

The square and circular symbols came from continuous cycling tests under different total strain ranges. It could be observed from Figure 3-21 that the evolution in 10Cr was less steep than in FB2, especially at 625°C. Since the cyclic stress-strain response for this type of steel normally exhibits the so-called Masing behaviour (e.g. Figure 1-3), there should be some resemblance between the cyclic values and the monotonic values (i.e. the cyclic strain hardening exponent should be in principle proportional to the monotonic strain hardening exponent). To verify this assumption, monotonic strain hardening exponents for both materials were also calculated (from the tensile loading part in first cycles) and these values are listed in Table 3-2.

Table 3-2 Cyclic and monotonic strain hardening exponent for FB2 and 10Cr.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature (°C)</th>
<th>Cyclic strain hardening exponent (derived from the midlife cycle)</th>
<th>Monotonic strain hardening exponent (derived from the first cycle)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Net section</td>
<td>Gross section</td>
</tr>
<tr>
<td>FB2</td>
<td>600</td>
<td>0.095</td>
<td>0.106</td>
</tr>
<tr>
<td></td>
<td>625</td>
<td>0.094</td>
<td>0.099</td>
</tr>
<tr>
<td>10Cr</td>
<td>600</td>
<td>0.076</td>
<td>0.065</td>
</tr>
<tr>
<td></td>
<td>625</td>
<td>0.060</td>
<td>0.051</td>
</tr>
</tbody>
</table>

At 600°C, the monotonic strain hardening exponent values were very similar for both FB2 and 10Cr (i.e. \( n = 0.108 \) and \( n = 0.101 \)); while at 625°C, the values for FB2 specimens did not change much (\( n = 0.107 \)), but the values for 10Cr reduced distinctly (\( n = 0.088 \)). For the cyclic strain hardening exponents, their values at 625°C were smaller than those at 600°C for both materials. Moreover, as already displayed in Figure 3-21, much smaller values of cyclic strain hardening exponent were obtained in 10Cr. (This finding is analogous to the decrease of the nominal yield strength from 600°C to 625°C, as will be shown in Figure B-2 in Appendix B).

The interpretation of cyclic strain hardening exponent can be approximated as a ratio between fatigue strength exponent (slope of elastic asymptote of the \( \Delta \varepsilon_t - N \) curve, Basquin equation) and fatigue ductility exponent (slope of plastic asymptote of the \( \Delta \varepsilon_p - N \) curve, Coffin-Manson equation). A larger fatigue strength exponent (absolute value) represents a larger number of cycles to fatigue failure under elastic strain; whereas a larger fatigue ductility exponent (absolute value) denotes a larger number of cycles to fatigue failure under plastic strain. Therefore, a higher value of cyclic strain hardening exponent potentially indicates that the material is relatively more resistant to elasticity-dominated fatigue, but more vulnerable to plasticity-dominated fatigue (e.g. FB2).
3.3.2 Crack closure

As already demonstrated in Figure 3-3 and Figure 3-4, the effect of crack closure was significant only after the 2% load drop point for both FB2 and 10Cr. Although the main interest of cyclic stress-strain response is limited to this point as far as short crack growth behaviour is concerned in this study, it is still worthwhile to explore and understand the influence of crack closure at a later stage when cracks extended longer.

Consistent with Chapter 1, the method to identify the crack closure/opening point was purely from the characteristic deflection position on each hysteresis loop. Initially, an automatic detection routine was programmed in Matlab to carry out derivatives of stress-strain curves. The inflection point was supposed to represent the crack closure/opening point, where an abrupt change of slope took place. Later on, it was found that due to the unavoidable noise of the data acquisition process and inherent differences in turning radius on stress-strain curves, it was difficult to retrieve accurate values by a single program for various conditions.

Therefore, a manual recognition method was applied. Apart from the heavier work load, the logic was simple. For each testpiece, hysteresis loops were plotted at a defined interval of life cycles; crack closure/opening stresses were artificially recognized for those selected loops; a series of those identified discrete points could be obtained for every specimen; finally a regression process was performed to extrapolate and interpolate corresponding values for all other cycles. In addition to a higher credibility, an added benefit of this method was a smoothing process during regression, which suppressed most of the data noise.

Nevertheless, it must be acknowledged that two complications still remained within this process. The first was the subjectivity of individual observer on turning point determination. In most of the hysteresis loops, there were transitional deflection stages rather than sharp turning points, which led to some ambiguity of the identification process. This is shown by the short red curve in Figure 3-22 (denoted as ‘I’), linking the adjacent quasi-linear curves. A maximum error of ±5MPa could be generated during this stage. The author adopted the mean stress value (shown by the blue dashed line) to represent the crack closure stress.
3.3 Further discussion

The second complication resulted from the special characteristics of this sample geometry and loading conditions. No obvious opening point could be detected, but a quasi-linear loading regime as shown in Figure 3-22 (denoted as 'II'). Only for specimens tested under low strain ranges, was it possible to ascertain real opening points (e.g. in Figure 1-9). Therefore, it was decided to use both the crack closure stress and the opening stress to characterize the magnitude of crack closure effect within the scope of this study.

\[
q_c = \frac{(P_{\text{max}} - P_c)}{(P_{\text{max}} - P_{\text{min}})} \quad \text{and} \quad q_o = \frac{(P_{\text{max}} - P_o)}{(P_{\text{max}} - P_{\text{min}})}
\]  

(3-7)

Nonetheless, by utilizing the data from two specimens where crack opening points could be readily determined, the magnitude of crack closure behaviour could finally be displayed in classical effective load ratio graphs (i.e. Figure 3-23 and Figure 3-24). Here the effective load ratio \( q \) was defined as
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where $q_c$ corresponded to crack closure load $P_c$, and $q_o$ corresponded to crack opening load $P_o$. The load ratio $R_\alpha$ was defined as

$$R_\alpha = \frac{P_{\text{min}}}{P_{\text{max}}}$$  \hspace{1cm} (3-8)

For both FB2 and 10Cr, the evolution paths of the effective load ratio were similar. They dropped from the origin point $(R_\alpha = -1, q = 1)$ gently, along with a reduced load ratio. Values of the effective opening load ratio $q_o$ (red curve) were smaller than the effective closure load ratio $q_c$, because $P_o$ was always larger than $P_c$.

The three references curves in Figure 3-23 and Figure 3-24 correspond to the widely used prediction bounds for the crack closure effect. For example, equation (3-9) denotes the assumption that the crack front is completely closed during the compressive part of the load range.

$$q = 1 / (1 - R_\alpha)$$  \hspace{1cm} (3-9)

And equation (3-10) represents the scenario where the crack front is closed only for half of the compressive part of the load range, and is otherwise open.

$$q = (1 - R_\alpha / 2) / (1 - R_\alpha)$$  \hspace{1cm} (3-10)

These two equations normally provide reasonable upper and lower bounds, and a third proposed bound, equation (3-11), is able to decrease the overestimation of equation (3-10), especially when the load ratio expands toward negative (specifically for 1CrMoV steels at 550°C).

$$q = (1 - R_\alpha / 2.2) / (1 - R_\alpha)$$  \hspace{1cm} (3-11)

The effectiveness of these bounds is shown in Figure 3-23 and Figure 3-24. Both curves for FB2 and 10Cr fell into the bounds only after the load ratio dropped below -1.5. This was reasonable since at least until the 2% load drop point the influence of crack closure was negligible in the investigated experiments.

Moreover, while equation (3-11) indeed displayed a more favourable prediction than equation (3-10), the effective load ratio was still largely overestimated. It was acknowledged that the crack closure effect can be material and geometry dependent, therefore a generalized Newman’s equation could be employed to describe the crack opening stress for plasticity induced fatigue crack closure (later developed to the Forman/Mettu, or NASGRO equation)\textsuperscript{176-178}. According to Newman’s study, the opening stress for experiments with negative load ratio can be approximated by:

$$
\sigma_o = \sigma_{\text{max}} \cdot \left\{ (0.825 - 0.34\alpha_N + 0.05\alpha_N^2) \left[ \cos \left( \frac{\pi \sigma_{\text{max}}}{2 \sigma_f} \right) \right]^{1/\alpha} \right\} \\
+ \sigma_{\text{max}} \cdot \left\{ (0.415 - 0.071\alpha_N) \frac{\sigma_{\text{max}}}{\sigma_f} \right\} \cdot R_\alpha \hspace{1cm} (3-12)
$$
where $\sigma_f$ is the material dependent flow stress (between uniaxial yield strength and ultimate tensile strength), and the constraint factor $\alpha_N$ denotes the applied stress/strain state ($\alpha_N = 1$ for plane stress conditions and $\alpha_N = 3$ for plane strain conditions). The effective load ratio can then be calculated from the following equation:

$$q = (1 - \frac{\sigma_v}{\sigma_{\text{max}}}) \left(1 - \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}}\right)$$  \hspace{1cm} (3-13)

Unlike equations (3-9)~(3-11), the effective load ratio in equation (3-13) is actually expressed by stress values instead of load values. In other words, the net section stress rather than the gross stress should be taken into account. With the two best fit/predetermined variables (i.e. $\sigma_f$ and $\alpha_N$), the $q - R$ curve predicted from Newman’s equation can be adapted with more flexibility. One example is shown in Figure 3-25, where a better consistency between the predicted (with Newman’s equation) and experimental curves was demonstrated. In this case, plane strain condition was assigned ($\alpha_N = 3$) and $\sigma_f = 300\text{MPa}$. However, a precise prediction bound with high validity requires more experiments with different strain ratios and geometries.

![Figure 3-25 Variation of the effective load ratio as a function of $R_s$ ratio for 10Cr by using Newman’s equation.](image)

### 3.3.3 Partitioning of hysteresis loops

A stress partitioning method is usually a good way to understand the correlation between mechanical behaviour (e.g. cyclic hardening/softening effects) and microstructural behaviour (e.g. dislocation movement).\textsuperscript{179} The main purpose of this section is to provide an index to characterize material properties at any intermediate cycle. This index can then be integrated into crack growth models (in Chapter 5) to account for the instantaneous microstructural condition of the materials. (It is found that friction stress is such an index, see section 5.3.5).
Experimental results from creep-fatigue tests

For reversible hysteresis loops (i.e. the dislocation behaviour and stress/strain distributions are not substantially changed), the well-known partition scheme dated back to Cottrell to divide the total stress range $\Delta \sigma$ in a loop into a friction stress $\sigma_F$ and a back stress $\sigma_B$.\(^{180}\) This scheme was also applied to the current study, and one example is shown in Figure 3-26a by a loop from FB-RB03. However, for fatigue-relaxation type hysteresis loops, a common practice could not be found in the literature, and the one used here is shown in Figure 3-26b by a loop from FB-RB06 (with 30min hold period in tension).

![Figure 3-26](image)

Figure 3-26 Stress partitioning for (a) a continuously cycled hysteresis loop and (b) a hysteresis loop with hold period in peak tensile strain.

The corresponding equations could be written as:

\[
\sigma_B = \frac{\sigma_{e_{max}} - \sigma_{e_{min}}}{2} \\
\sigma_F = \frac{\sigma_{max} - \sigma_{e_{min}}}{2} \\
\sigma_R = \sigma_{max} - \sigma_{e_{max}}
\]  

(3-14)

where $\sigma_{e_{max}}$ and $\sigma_{e_{min}}$ are the maximum or minimum values in the linear regime on compressive-going ramp ($\sigma_{e_{max}}$ is also denoted as $\sigma_{\text{hold}}$ in Table 3-3 and Table 3-4); and $\sigma_R$ is the relaxation stress during the hold period. Note that the current expression for back stress is different from that used by Fournier et al.\(^{181}\) (For hysteresis loops like that in Figure 3-26a, the same back stress values could be obtained when $\sigma_{e_{max}}$ is identical to $\sigma_{max}$; for loops like the one in Figure 3-26b, Fournier’s scheme yielded a much lower value of $\sigma_B$, i.e. $\sigma_B = (\sigma_{e_{max}} + \sigma_{e_{min}})/2$. This is due to the normally positive values of $\sigma_{e_{max}}$ but negative values of $\sigma_{e_{min}}$. Nevertheless, the evolution paths of $\sigma_B$ are the same from both schemes, despite the different absolute values).

It has been suggested that the change of $\sigma_B$ and $\sigma_F$ can be best represented by plotting them versus the accumulated inelastic strain $\Delta \varepsilon_{\text{in}}^{\text{cum}}$ to take the influence of total strain range (and hold period as far as the testing conditions in this study) into consideration.\(^{179, 182}\) The expression for $\Delta \varepsilon_{\text{in}}^{\text{cum}}$ is:
3.3 Further discussion

\[
\Delta \varepsilon^{\text{cum}}_{\text{in}}(N) = 2 \sum_{i=1}^{N} \Delta \varepsilon_{\text{in}} = 2 \sum_{i=1}^{N} \left( \varepsilon_{\text{in}}^{\text{max}} - \varepsilon_{\text{in}}^{\text{min}} \right) \tag{3-15}
\]

where \( \Delta \varepsilon_{\text{in}} \) is the inelastic strain range in each cycle, \( \varepsilon_{\text{in}}^{\text{max}} \) and \( \varepsilon_{\text{in}}^{\text{min}} \) are the values of strain at the intercept points with zero stress (i.e. the maximum and minimum values of inelastic strain, shown in Figure 3-26).

Values of \( \Delta \varepsilon^{\text{cum}}_{\text{in}} \), \( \sigma_B \), \( \sigma_F \), and \( \sigma_R \) were computed for both FB2 and 10Cr. The evolution of back stress, friction stress and relaxation stress are plotted as a function of the cumulated inelastic strain in Figure 3-27, Figure 3-28 and Figure 3-29, respectively. (Although corresponding values at both temperatures have been calculated, only those at 625°C were shown here).

In Figure 3-27, a clear distinction in the absolute values of back stress from tests with hold period and from the continuously cycled tests could be observed. This was most probably due to the inherent difference according to the loop shape (Figure 3-26), which inevitably led to a smaller value of \( \sigma_B \). However, the trend of the curves resembled each other, which was similar to the softening behaviour in peak stresses (i.e. in Figure 3-1). This inference was also
consistent with the reported findings that the cyclic softening behaviour could mainly be related to the change of back stress.\textsuperscript{181,182}

In Figure 3-28, it could be seen that the absolute values of the friction stress remained almost the same (or only a slight decrease) for the three continuously cycled tests. But for those tests with hold periods (no matter in tension or in compression), there was also an apparent decrease in $\sigma_F$ values. This observation was different from the findings of Fournier et al., which stated that there was no influence of hold periods on the evolution of the friction stress.\textsuperscript{182}

This discrepancy could result from two aspects: (1) the change of the cross-section area. In dwelled samples, the crack propagated much faster, which brought about a quicker decrease of cross-section area. Since the adopted stress values were actually nominal values, the influence of crack development was not incorporated; (2) the change of microstructure. As demonstrated in a later section (Chapter 4), the microstructural evolution of creep-fatigue deformed specimens was significant, especially for those with long hold periods. Therefore, the interior resistance against plastic deformation (i.e. the movement of dislocations) would also decrease faster in tests with hold periods. Thus, it was no wonder that the behaviour of friction stress $\sigma_F$ was not the same for different testing conditions.

As for the evolution of relaxation stress, a uniform trend could be observed (Figure 3-29). This might prove the fact that the relaxation/creep behaviour was similar at the targeted temperatures (i.e. 600°C and 625°C) for each tested material, no matter with tensile hold or compressive hold. By comparing the relaxation stress $\sigma_R$ for FB2 (Figure 3-29a) and 10Cr (Figure 3-29b), larger values were found for the investigated 10Cr specimens. This inferred that the resistance against relaxation/creep was smaller in 10Cr than in FB2.

The last thing to point out is that in practice, the accurate determination of $\sigma_B$ and $\sigma_F$ relies also on the position of $\sigma_R^{\text{min}}$ (Figure 3-26). Due to data scattering, normally an offset parameter (strain displacement) is applied to estimate the first point deviated from linearity. In the current study, this value is $1 \times 10^{-4}$, which was found to be adequate to successfully suppress the noise,
while keeping the estimation as close as possible. If another offset value was assigned, the
acquired $\sigma_B$ and $\sigma_F$ would also be slightly different.

3.4 Summary

In the short-crack creep-fatigue crack growth test campaign for FB2 and 10Cr, primarily three
controlling variables were included, i.e. total strain range $\Delta \varepsilon$, temperature $T$, and hold time $t_h$
(in tension/compression). To be more exact, $\Delta \varepsilon$ mainly ranged from 0.5% to 1%, $T$ was
either 600°C or 625°C, and $t_h$ equalled zero, 30min, or 60min. The combination of these
parameters led to comprehensive experimental matrices. This chapter first explains the cyclic
stress-strain behaviour of all experiments, and the summary of characteristic stress values are
shown in Table 3-3 for FB2 and Table 3-4 for 10Cr.

### Table 3-3 Summary of stress values for short-crack creep-fatigue crack growth tests for FB2 specimens.
(Detailed testing conditions for FB2 can be found in Table 2-3).

<table>
<thead>
<tr>
<th>FB2</th>
<th>Internal No.</th>
<th>$E$ (GPa)</th>
<th>$\sigma_{\text{max}}^c1$ (MPa)</th>
<th>$\sigma_{\text{min}}^c1$ (MPa)</th>
<th>$\sigma_{\text{hold}}^c1$ (MPa)</th>
<th>$\sigma_{\text{max}}^\text{mlc}$ (MPa)</th>
<th>$\sigma_{\text{min}}^\text{mlc}$ (MPa)</th>
<th>$\sigma_{\text{hold}}^\text{mlc}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RB01</td>
<td>HT1134</td>
<td>146.1</td>
<td>457</td>
<td>-466</td>
<td>-</td>
<td>340</td>
<td>-345</td>
<td>-</td>
</tr>
<tr>
<td>RB02</td>
<td>HT1193</td>
<td>139.6</td>
<td>422</td>
<td>-425</td>
<td>-</td>
<td>300</td>
<td>-305</td>
<td>-</td>
</tr>
<tr>
<td>RB03</td>
<td>HT1137</td>
<td>154.7</td>
<td>433</td>
<td>-422</td>
<td>-</td>
<td>327</td>
<td>-333</td>
<td>-</td>
</tr>
<tr>
<td>RB04</td>
<td>HT1136</td>
<td>143.5</td>
<td>338</td>
<td>-343</td>
<td>-</td>
<td>292</td>
<td>-285</td>
<td>-</td>
</tr>
<tr>
<td>RB05</td>
<td>HT1192</td>
<td>144.2</td>
<td>284</td>
<td>-294</td>
<td>-</td>
<td>281</td>
<td>-279</td>
<td>-</td>
</tr>
<tr>
<td>RB06</td>
<td>HT1138</td>
<td>151.2</td>
<td>473</td>
<td>-547</td>
<td>251</td>
<td>293</td>
<td>-329</td>
<td>172</td>
</tr>
<tr>
<td>RB07</td>
<td>HT1262</td>
<td>146.8</td>
<td>456</td>
<td>-467</td>
<td>234</td>
<td>288</td>
<td>-318</td>
<td>158</td>
</tr>
<tr>
<td>RB08</td>
<td>HT1194</td>
<td>153.8</td>
<td>461</td>
<td>-463</td>
<td>-243</td>
<td>329</td>
<td>-303</td>
<td>-180</td>
</tr>
<tr>
<td>RB09</td>
<td>HT1139</td>
<td>140.2</td>
<td>431</td>
<td>-433</td>
<td>-</td>
<td>314</td>
<td>-321</td>
<td>-</td>
</tr>
<tr>
<td>RB10</td>
<td>HT1140</td>
<td>145.2</td>
<td>405</td>
<td>-411</td>
<td>-</td>
<td>299</td>
<td>-305</td>
<td>-</td>
</tr>
<tr>
<td>RB11</td>
<td>HT1141</td>
<td>136.8</td>
<td>323</td>
<td>-341</td>
<td>-</td>
<td>268</td>
<td>-275</td>
<td>-</td>
</tr>
<tr>
<td>RB12</td>
<td>HT1191</td>
<td>138.5</td>
<td>430</td>
<td>-427</td>
<td>-212</td>
<td>275</td>
<td>-257</td>
<td>-127</td>
</tr>
</tbody>
</table>

Here $\sigma_{\text{max}}^c1$, $\sigma_{\text{min}}^c1$ and $\sigma_{\text{hold}}^c1$ are the maximum stress, minimum stress and stress after relaxation
at cycle 1. $\sigma_{\text{max}}^{\text{mlc}}$, $\sigma_{\text{min}}^{\text{mlc}}$ and $\sigma_{\text{hold}}^{\text{mlc}}$ are the maximum stress, minimum stress and stress after
relaxation at the midlife cycle (corresponds to characteristic point 2 in Figure 3-2a).
Experimental results from creep-fatigue tests

Table 3-4 Summary of stress values for short-crack creep-fatigue crack growth tests for 10Cr specimens. (Detailed testing conditions for 10Cr can be found in Table 2-4).

<table>
<thead>
<tr>
<th>10Cr</th>
<th>Internal No.</th>
<th>$E$ (GPa)</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\sigma_{\text{min}}$ (MPa)</th>
<th>$\sigma_{\text{hold}}$ (MPa)</th>
<th>$\sigma_{\text{max}}^{\text{ml}}$ (MPa)</th>
<th>$\sigma_{\text{min}}^{\text{ml}}$ (MPa)</th>
<th>$\sigma_{\text{hold}}^{\text{ml}}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RB01</td>
<td>HT1349</td>
<td>142.4</td>
<td>464</td>
<td>-459</td>
<td>-</td>
<td>331</td>
<td>-341</td>
<td>-</td>
</tr>
<tr>
<td>RB02</td>
<td>HT1352</td>
<td>139.7</td>
<td>432</td>
<td>-423</td>
<td>-</td>
<td>322</td>
<td>-330</td>
<td>-</td>
</tr>
<tr>
<td>RB03</td>
<td>HT1354</td>
<td>145</td>
<td>341</td>
<td>-341</td>
<td>-</td>
<td>303</td>
<td>-294</td>
<td>-</td>
</tr>
<tr>
<td>RB04</td>
<td>HT1355</td>
<td>149.3</td>
<td>464</td>
<td>-479</td>
<td>243</td>
<td>286</td>
<td>-321</td>
<td>154</td>
</tr>
<tr>
<td>RB05</td>
<td>HT1491</td>
<td>149.1</td>
<td>468</td>
<td>-468</td>
<td>-227</td>
<td>317</td>
<td>-289</td>
<td>-159</td>
</tr>
<tr>
<td>RB06</td>
<td>HT1351</td>
<td>139.3</td>
<td>389</td>
<td>-376</td>
<td>-</td>
<td>296</td>
<td>-304</td>
<td>-</td>
</tr>
<tr>
<td>RB07</td>
<td>HT1353</td>
<td>154.0</td>
<td>408</td>
<td>-423</td>
<td>-</td>
<td>290</td>
<td>-294</td>
<td>-</td>
</tr>
<tr>
<td>RB08</td>
<td>HT1357</td>
<td>136.5</td>
<td>324</td>
<td>-330</td>
<td>-</td>
<td>272</td>
<td>-277</td>
<td>-</td>
</tr>
<tr>
<td>RB09</td>
<td>HT1356</td>
<td>148.6</td>
<td>429</td>
<td>-440</td>
<td>212</td>
<td>245</td>
<td>-274</td>
<td>107</td>
</tr>
<tr>
<td>RB10</td>
<td>HT1358</td>
<td>152.7</td>
<td>432</td>
<td>-442</td>
<td>202</td>
<td>235</td>
<td>-265</td>
<td>96</td>
</tr>
<tr>
<td>RB11</td>
<td>HT1492</td>
<td>138.5</td>
<td>430</td>
<td>-438</td>
<td>-207</td>
<td>265</td>
<td>-250</td>
<td>-108</td>
</tr>
</tbody>
</table>

After the test, specimens were broken open to obtain the final values of crack depth, which could then be employed in the DCPD calibration process as explained in section 3.2.1. In Table 3-5 and Table 3-6, true final crack depth values and crack growth rates at three different crack depth (i.e. at 0.5mm, 1.0mm and 2.0mm) were listed. Note that crack growth rates were given in micron instead of millimetre per cycle (i.e. $\mu$m/c).

Table 3-5 Summary of crack development for short-crack creep-fatigue crack growth tests for FB2 specimens. (Detailed testing conditions for FB2 can be found in Table 2-3).

<table>
<thead>
<tr>
<th>FB2</th>
<th>Internal No.</th>
<th>$a_f$ (mm)</th>
<th>$N_{2%}$</th>
<th>da/dN (0.5mm) ($\mu$m/c)</th>
<th>da/dN (1.0mm) ($\mu$m/c)</th>
<th>da/dN (2.0mm) ($\mu$m/c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RB01</td>
<td>HT1134</td>
<td>4.26</td>
<td>556</td>
<td>2.36</td>
<td>4.34</td>
<td>7.74</td>
</tr>
<tr>
<td>RB02</td>
<td>HT1193</td>
<td>4.96</td>
<td>528</td>
<td>2.54</td>
<td>4.30</td>
<td>7.51</td>
</tr>
<tr>
<td>RB03</td>
<td>HT1137</td>
<td>4.52</td>
<td>918</td>
<td>1.46</td>
<td>2.38</td>
<td>5.32</td>
</tr>
<tr>
<td>RB04</td>
<td>HT1136</td>
<td>3.83</td>
<td>3622</td>
<td>0.41</td>
<td>0.65</td>
<td>0.86</td>
</tr>
<tr>
<td>RB05</td>
<td>HT1192</td>
<td>6.07</td>
<td>5726</td>
<td>0.27</td>
<td>0.43</td>
<td>0.77</td>
</tr>
<tr>
<td>RB06</td>
<td>HT1138</td>
<td>6.16</td>
<td>398</td>
<td>3.02</td>
<td>6.49</td>
<td>15.8</td>
</tr>
<tr>
<td>RB07</td>
<td>HT1262</td>
<td>3.66</td>
<td>314</td>
<td>4.23</td>
<td>7.17</td>
<td>14.8</td>
</tr>
<tr>
<td>RB08</td>
<td>HT1194</td>
<td>4.87</td>
<td>320</td>
<td>4.80</td>
<td>7.09</td>
<td>13.1</td>
</tr>
<tr>
<td>RB09</td>
<td>HT1139</td>
<td>3.75</td>
<td>410</td>
<td>2.86</td>
<td>4.93</td>
<td>9.71</td>
</tr>
<tr>
<td>RB10</td>
<td>HT1140</td>
<td>3.52</td>
<td>822</td>
<td>1.46</td>
<td>2.57</td>
<td>5.08</td>
</tr>
<tr>
<td>RB11</td>
<td>HT1141</td>
<td>3.04</td>
<td>2678</td>
<td>0.54</td>
<td>0.80</td>
<td>1.20</td>
</tr>
<tr>
<td>RB12</td>
<td>HT1191</td>
<td>5.33</td>
<td>356</td>
<td>3.99</td>
<td>6.00</td>
<td>11.5</td>
</tr>
<tr>
<td>RB13</td>
<td>HT1195</td>
<td>4.34</td>
<td>368</td>
<td>2.99</td>
<td>5.96</td>
<td>15.4</td>
</tr>
</tbody>
</table>
3.4 Summary

Table 3-6 Summary of crack development for short-crack creep-fatigue crack growth tests for 10Cr specimens. (Detailed testing conditions for 10Cr can be found in Table 2-4).

<table>
<thead>
<tr>
<th>10Cr</th>
<th>Internal No.</th>
<th>( a_f ) (mm)</th>
<th>( N_{2%} )</th>
<th>( \frac{da}{dN} ) (0.5mm) (μm/c)</th>
<th>( \frac{da}{dN} ) (1.0mm) (μm/c)</th>
<th>( \frac{da}{dN} ) (2.0mm) (μm/c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RB01</td>
<td>HT1349</td>
<td>3.48</td>
<td>657</td>
<td>2.39</td>
<td>3.39</td>
<td>4.45</td>
</tr>
<tr>
<td>RB02</td>
<td>HT1352</td>
<td>3.79</td>
<td>1208</td>
<td>1.30</td>
<td>2.47</td>
<td>4.32</td>
</tr>
<tr>
<td>RB03</td>
<td>HT1354</td>
<td>2.36</td>
<td>3382</td>
<td>0.37</td>
<td>0.62</td>
<td>0.93</td>
</tr>
<tr>
<td>RB04</td>
<td>HT1355</td>
<td>1.96</td>
<td>447</td>
<td>2.99</td>
<td>4.69</td>
<td>(6.59+)</td>
</tr>
<tr>
<td>RB05</td>
<td>HT1491</td>
<td>3.80</td>
<td>438</td>
<td>3.96</td>
<td>5.69</td>
<td>8.20</td>
</tr>
<tr>
<td>RB06</td>
<td>HT1351</td>
<td>3.34</td>
<td>660</td>
<td>2.06</td>
<td>3.52</td>
<td>5.30</td>
</tr>
<tr>
<td>RB07</td>
<td>HT1353</td>
<td>3.04</td>
<td>1126</td>
<td>1.17</td>
<td>2.18</td>
<td>3.58</td>
</tr>
<tr>
<td>RB08</td>
<td>HT1357</td>
<td>2.71</td>
<td>3181</td>
<td>0.50</td>
<td>0.78</td>
<td>1.21</td>
</tr>
<tr>
<td>RB09</td>
<td>HT1356</td>
<td>2.83</td>
<td>420</td>
<td>4.37</td>
<td>6.41</td>
<td>9.43</td>
</tr>
<tr>
<td>RB10</td>
<td>HT1358</td>
<td>1.69</td>
<td>400</td>
<td>4.14</td>
<td>7.4</td>
<td>(11.6+)</td>
</tr>
<tr>
<td>RB11</td>
<td>HT1492</td>
<td>3.16</td>
<td>415</td>
<td>3.75</td>
<td>5.47</td>
<td>7.60</td>
</tr>
</tbody>
</table>

(1) The values in the brackets are expected to be smaller than real values, because in these two tests the final crack depth were smaller than 2mm. Therefore, the crack propagation rates at the last cycles are given here.

Similarities and differences between FB2 and 10Cr in mechanical response and the influences of the testing parameters on crack growth behaviour are briefly summarized as follows:

**Stress-strain response**

1. In strain controlled short-crack creep-fatigue crack growth tests, all the FB2 and 10Cr testpieces exhibited a cyclic softening behaviour (e.g. Figure 3-1). In a typical \( \sigma_{\text{max}} - N \) plot, this softening pattern could be viewed as a relatively rapid softening regime at first, followed by a steadier softening regime and finally a relatively unstable load drop. This common behaviour can be represented by the devised partition method, with four characteristic points (i.e. Figure 3-2). Point 1 denotes the beginning of the steady softening regime; point 2 denotes the mid-life cycle (i.e. \( 0.5N_{2\%} \)); point 3 marks the end of the steady softening regime; and point 4 marks the number of cycles to 2% load drop (i.e. \( N_{2\%} \)). This classification method laid the foundation for further comparison between specimens under various test conditions.

2. The applied strain amplitude (i.e. \( \Delta \varepsilon \)) and the hold period (i.e. position and \( t_h \)) had the largest impact on the number of cycles to 2% load drop (i.e. characteristic point 4). In particular, an increase from \( \Delta \varepsilon = 0.4\% \) to \( \Delta \varepsilon = 1\% \) almost reduced the cycle number by an order of magnitude. The presence of the 30min/60min hold periods brought about even smaller endurances at \( N_{2\%} \), whereas the effect of compressive dwell was more prominent
than that of the tensile dwell. Moreover, while the influence of temperature was apparent for FB2, it was not enormous for 10Cr. Under the same test conditions, the 10Cr specimens had an overall longer life time (until \( N_{2N} \)) than FB2 specimens (i.e. Figure 3-5 and Figure 3-6).

3. The occurrence of crack closure could be identified by the cusps on hysteresis loops in the compressive loading branch (e.g. Figure 3-22). For all the testpieces, crack closure was almost insignificant before 2% load drop (e.g. Figure 3-3). This infers that at least in the steady softening regime, the influence of crack closure can be neglected for the investigated materials and test set-up.

4. A stress partitioning method was employed to divide the total stress range into friction stress \( \sigma_F \), back stress \( \sigma_B \) and relaxation stress \( \sigma_R \) (Figure 3-26). It was found that the change of \( \sigma_B \) was similar to the softening behaviour in peak stresses (Figure 3-27), which is consistent with the literature. On the other hand, \( \sigma_F \) was almost constant for continuously cycled tests (Figure 3-28), but decreased apparently for dwelled tests. This could be due to the faster change of the cross-section area and/or the microscopic strengthening structures. In addition, the evolution path of \( \sigma_R \) was similar in both FB2 and 10Cr specimens (Figure 3-29), despite different hold periods. In particular, the values of \( \sigma_R \) were larger for 10Cr than in FB2, which inferred a higher resistance against relaxation/creep in the tested FB2 steel.

**Crack development**

1. For FB2 specimens, the propagating crack front was always approximately straight (Figure 3-7). For 10Cr specimens, the crack developed from the initial chordal crack starter, with an increasingly bulging frontier in the middle section which finally led to a part-elliptical shape (i.e. between chordal crack geometry and semi-circular crack geometry, Figure 3-11). Based on these two different propagation profiles, two calibration curves (DCPD \( \rightarrow \) crack depth) have been derived, in combination with an additional correction process (i.e. rescaled according to the measured final crack depth \( a_f \)).

2. By comparing the values of crack depth at characteristic points in all the tests (i.e. Figure 3-14), it has been observed that the total crack depth (including the crack starter) was \( \sim 0.5 \text{mm} \) at the start of the steady softening regime (characteristic point 1); \( \sim 0.75 \text{mm} \) at midlife (characteristic point 2); \( \sim 1.7 \text{mm} \) at the end of the steady softening regime (characteristic point 3); and \( \sim 2 \text{mm} \) at \( N_{2N} \) (Point 4). This illustrated a good consistency between cyclic stress response and crack propagation behaviour. In view of this evidence and in accordance with the findings of others, the short crack regime was limited to 2mm (i.e. \( 0.2 \text{mm} \leq a \leq 2 \text{mm} \)) in this study.

3. Total strain range \( \Delta \varepsilon_t \) and hold time \( t_h \) were the most dominating factors to influence crack growth rates (Figure 3-15 and Figure 3-16). Cracks in tests with \( \Delta \varepsilon_t = 1\% \) propagated
3.4 Summary

much faster than those with smaller strain amplitudes, whereas tests with $t_h \neq 0$ had even larger growth rates. For the investigated materials, cracks in 10Cr specimens generally propagated more slowly than in FB2 specimens under the same testing condition, which potentially demonstrated a higher resistance of the 10Cr steel to crack development under creep-fatigue deformation.

4. A systematic examination was performed on the evolution of DCPD values during the hold periods for various experiments. It was found that the mean voltage change during dwell $\Delta V_{mean}$ was initially positive and evolved toward negative for compressive dwelled specimens (e.g. Figure 3-18ab); whereas $\Delta V_{mean}$ was initially negative and evolved toward positive for tensile dwelled specimens (e.g. Figure 3-18cd). More importantly, the change of voltage during dwell was much smaller than that between adjacent cycles, which implied that the development of DCPD values took place mainly in the ramping period rather than in the hold period (Figure 3-19 and Figure 3-20). This observation supports the inference that instead of direct creep crack growth during the hold time, the enhancement of apparent crack growth rate is due to accelerated fatigue crack propagation as a result of prior creep and strain enhanced oxidation at the crack tip.
Chapter 4

Microstructural analysis

Along with macroscopic mechanical deformation response at elevated temperatures, the microstructures of tested specimens also changed. It is absolutely necessary to figure out how the interior crystallographic or morphological features altered as a consequence of creep-fatigue interaction, because the microstructural evolution would in turn influence the crack propagation behaviour considerably. In this study, although the raw materials (FB2 and 10Cr) were designed to be creep resistant, their true performance under severe loading conditions is worth inspecting.

In the first stage of microstructural investigation, traditional optical microscopy and secondary electron microscopy were carried out, by following the steps of classical sample preparation. The etched samples were able to reflect the shapes and dimensions of prior-austenite grains, the crack propagation path and oxide layers. Due to resolution limitations, this method was at most capable of identifying the directional sub-structures (martensitic blocks or laths, Figure 1-18). In SEM, the distribution of second-phase particles can be observed.

In the second/main stage of microstructural investigation, EBSD measurements were performed, with three essential purposes: (1) to distinguish between martensitic sub-structural features; (2) to assess the change of morphology and crystallographic orientation and (3) to quantitatively determine the evolution of micrograin size subjected to creep-fatigue deformation.

After elaborate measurement and analysis, the results indicated that there was a microstructural variation in each sample, and its magnitude was governed not only by the previous creep-fatigue testing conditions (sample dependent), but also by the measurement locations along the crack propagation path (crack depth dependent, or to be more exact, the accumulated plastic strain at the location when the crack got there). Finally, these results were supposed to improve the effectiveness of crack growth models by integrating a microscopic parameter, which will be explained in more details in Chapter 5.
4.1 OM and SEM observation

This part of the microstructural investigation was mainly designed for four purposes: (1) to observe the dimensions of the prior austenitic grain structure as well as the martensitic features; (2) to find out the profile of the crack propagation path; (3) to figure out the distribution of inclusions and/or precipitates; (4) to detect micro-cracks or voids, if there were any. In order to fulfil those tasks, different samples from various creep-fatigue testpieces were prepared and etched. Due to the composition/processing similarity between FB2 and 10Cr steels, most inspection was done on FB2 samples, and some conclusions were later re-confirmed by observations on 10Cr samples.

4.1.1 Martensitic morphology

Both FB2 and 10Cr are tempered martensitic steels, therefore they should exhibit a typical tempered martensitic morphology as introduced in Chapter 1, i.e. prior austenitic grains, packets, blocks, laths and subgrains (Figure 1-18 and Figure 1-19). After careful preparation,
the etched sample surfaces were able to reveal most of the tempered martensitic morphology, as displayed in Figure 4-1 and Figure 4-2. The majority of prior austenitic grain boundaries could be detected, as the austenitic grains were usually large and separated by different contrasts. Some of the packets could also be identified, since the interfaces (packet boundaries) divided different agglomerations of directional arrangements.

However, it was difficult to further distinguish between block and lath structures. Although elongated or lenticular features were clearly recognized, no information regarding the types of boundaries could be verified. This was because on one side the resolution of current optical microscopes was not sufficiently high, and on the other side the microstructure of martensite was very complex. (This problem can almost be solved by the EBSD technique, as will be shown in section 4.2.2).

Indeed, microstructures of the two tempered martensitic steels (i.e. FB2 and 10Cr) deviated from the ideal two-dimensional representation of lath martensites. Apart from a lot of interceptions of differently oriented clusters, the size and shape of martensitic features varied massively. Moreover, there seemed to be a certain degree of inhomogeneity especially in the deformed structure.

By comparing Figure 4-1 and Figure 4-2, another distinct feature could be noted: although FB2 and 10Cr are very similar steels, the prior austenitic grain sizes appear to be very different. For FB2 material in the as-received state, the grain size could be as large as 500μm; whereas for 10Cr, this value is typically 200μm. (The grain size here was estimated by manually measuring the diameter of every prior austenitic grain in the field of vision). This difference could come from chemical composition or from the heat treatment (or from the forging process). Two possible reasons were suggested as a first estimation.

By comparing the weight percentage of elements for both materials (in Table 2-1), it is clear that FB2 and 10Cr have similar alloying contents. However, the addition of Cobalt could result in the increased austenite grain size (as well as the formation speed). This can (partly) be the reason why FB2 exhibits larger prior austenite grains.

As introduced in section 2.1, the procedures of heat treatment for both steels are also analogous. However, FB2 was austenitized at 1100°C, whereas 10Cr was austenitized at 1000°C. Although more details in the process of heat treatment are needed for a doubtless explanation (such as time and heating/cooling rate in each stage, or even the size of rotor forging), higher temperatures usually generate larger austenitic grain sizes. This being the case, it is expected that the 10Cr steel exhibits a smaller prior austenite grain size than the FB2 steel.
4.1 OM and SEM observation

4.1.2 Crack initiation and propagation path

![Typical crack propagation path shown in the (a) polished state and (b) etched state.](image)

As introduced in Chapter 2, the formal short-crack creep-fatigue crack growth tests were all followed by a break-open process so as to determine the true final crack depth information. Therefore, in order to find out the crack propagation profile, several companion tests were also done without the break-open process to contain the complete crack propagation profile. One example (with the same testing conditions as for FB2-RB04) is shown in Figure 4-3.

![Magnified pictures showing (a) branch cracks at crack starter and (b) individual branch crack.](image)

From the pictures showing polished and etched states of the sample (Figure 4-3a and b), some distinct patterns could be examined:

1. The true fatigue crack started not necessarily from the deepest location within the initial crack starter, although theoretically speaking that was where the largest stress concentration existed. Instead, multiple cracks could originate at the same time (e.g. in this example, three candidate cracks were initiated, and two of them stopped propagating afterwards). This could be better illustrated in an enlarged picture in Figure 4-4a. Moreover,
it is clear that the specimen surface (root of the crack starter) as well as crack paths were covered or filled with oxides, which indicates that crack initiation was likely to have been assisted by oxidation.

In Chapter 3, it has been concluded that compressive dwelled specimens had a shorter life till the 2% load drop point than tensile dwelled specimens. One possible reason for this was oxidation assisted crack propagation, especially at the crack initiation stage. This is illustrated in Figure 4-5.

This example came from FB2-RB13, with 30min dwell in compression. One notable feature is that many oxide penetrations occurred on the surface of this specimen (even though they were far away from the cracking plane). An enlarged view in Figure 4-5 apparently exhibits that these intruded sites were not initiated on any prior austenitic grain boundary, nor were they 45° inclined to the loading axis. This type of stress-concentrated geometry is more favourable for crack propagation under creep-fatigue loading conditions.

2. Branch cracks were formed along the major crack path, with an inclined angle of approximately 45° to the main cracking direction (Figure 4-4b). There were actually two possible reasons for the branch crack propagation. Firstly, they could be formed at the instantaneous crack tip (tensile loading condition), with an equally concentrated stress state as the (later known) major crack, but stopped growing when the major crack propagated further. Secondly, they could be formed during the compressive loading condition, where the closing surfaces did not match each other thus inducing a high compressive force.

3. The major cracking plane was basically transverse to the loading direction, with a rather rough profile, resulting from the influence of locally concentrated plastic deformation. The etched sample surface could reveal the prior austenite grain boundaries and therefore, indicated that the cracking mode was transgranular. This can be better demonstrated by an enlarged view of the crack path, illustrated in Figure 4-6. (Figure 4-6b is a schematic representation of Figure 4-6a, with the prior austenitic grain boundaries highlighted). The major crack as well as three branch cracks penetrated directly through the prior austenitic grains, indicating a dominant effect of fatigue (transgranular) cracking. Similar pattern could
also be seen in branch cracks. As exhibited in Figure 4-4b, the branch crack was straight and penetrated at least through some martensitic sub-structure features.

![Image](image.png)

Figure 4-6 Enlarged view of part of the major crack path in Figure 4-3, showing a transgranular pattern by (a) etched surface and (b) schematic representation. (PAG refers to prior austenitic grain).

Even in the dwelled specimens, no obvious intergranular cracking path could be observed. This is illustrated in Figure 4-7, which was from the testpiece with 60min hold period (FB2-RB07). In this case, the specimen was already broken open, and therefore only half of the cracking plane could be displayed. Nevertheless, judging from the relatively flat fracture surface, it was more likely that this was due to transgranular cracking rather than intergranular cracking.
In fact, as will be shown in Appendix D, even in long crack growth tests with already obvious creep micro-cracks, the major cracking profile was still generally transgranular (Figure D-15). This proves that the investigated materials in this study are truly creep-resistant. However, it should be pointed out that for martensitic steels, it is possible that ‘intergranular cracking’ takes place on other boundaries rather than prior austenitic boundaries due to their complex substructures (i.e. packet boundaries, block boundaries or lath boundaries). Therefore, a more sophisticated examination technique should be used if necessary, e.g. the TEM technique. (In addition, the crack profile should be maintained, i.e. without the break-open process).

Owing to the high tendency to oxidize, the newly generated crack fronts were susceptible to a further propagation (oxide delamination during a subsequent tensile transient). This could also be seen in more details in Figure 4-4b, with the oxide thickness of around 3μm on each branch crack surface, while still leaving a thin gap for more oxygen to flow in. To have a clearer view on crack propagation paths, SEM measurements were performed in addition to optical microscopy.

In a conventional SEM, two types of detectors are normally adopted, namely, a secondary electron (SE) detector and a back scattered electron (BSE) detector. The SE signal has low energy, which means that the penetration depth is very small. Therefore, it is very sensitive to surface topography, as well as to contaminants (stain, dirt, etc.).

In contrast, the BSE signal has large energy and passes through a layer of typically tens to hundreds of nanometres (material and microscope dependent). Consequently, it can tolerate a certain quantity of surface defects, but is very sensitive to the density of matter which reflects electrons, as well as the lattice structure. Together with the acquired graphs using SE and BSE signals, a lot of features can be observed, which will be particularly discussed in the following section.

As illustrated before, when the true fatigue crack initiated from the crack starter, multiple sub-cracks could be generated at the same time, with one of them developing into the major crack. This is again displayed more clearly in Figure 4-8, showing two images at the crack starter location for two interrupted tests under the same testing condition.
4.1 OM and SEM observation

Due to the contrast difference between matrix metal and oxide, it is possible to distinguish between them easily—the oxide is always darker (fewer signals from secondary electrons). This made the observation of cracks straightforward, because the crack paths were filled with oxides. In the left image of Figure 4-8, at least five sub-cracks could be detected, with almost the same depth (0.1mm) from the crack starter. Compared with the crack opening (less than 2μm), the oxide layer thickness was quite large (about 30μm), which indicated that oxidation is likely to have played an important role in the crack initiation/propagation process. (Certainly some of the oxide could have been deposited after the crack was formed).

In the right image of Figure 4-8, the major crack had propagated for about 0.5mm. Although not many sub-cracks were initiated at the crack starter, there were a lot of branches along the
crack propagation path. An enlarged view of the final crack tip is displayed in Figure 4-9, showing a complex profile of branch cracks. Instead of a single sharp crack tip, the major crack broke down to two branch cracks, which subsequently further divided into even smaller branch cracks. Moreover, there were always angular relationships between lower and higher levels of branch cracks.

This structure reflected the fact that the fracture process at the crack tip was mainly by tearing open material along the two inclined slip planes (possibly also influenced by the microstructure). This additionally inferred that the plastic fraction of load had the dominant influence on crack propagation. Actually, Figure 4-8 and Figure 4-9 were from short-crack creep-fatigue crack growth tests, where the total strain range was large enough to induce severe plastic deformation throughout the testpiece, especially in the intensified crack tip region. It was no wonder that lots of branch cracks could be created.

Technically, the term ‘crack depth’ was used only as a nominal value, because of the existence of branch cracks especially in the crack tip region. It was hardly possible to uniformly define the true distance a crack has propagated. Moreover, the cracking plane was not always flat as in the ideal elastic case. Although macroscopically the major crack was normal to the loading direction, the local cracking path fluctuated along with the magnitude of inelastic deformation. Therefore, the crack depth values utilized in this study were in fact simplified nominal macroscopic values, without respect to local cracking configurations. (In this sense, crack tip parameters such as $K$, $J$, or SEDF adopted in this study were also calculated as ‘effective’ values, with no consideration of crack tip branching).

### 4.1.3 Second-phase particles

As displayed in Figure 4-3b, Figure 4-6a and Figure 4-7a, there seemed to be some left-over pits on the sample surface, which corresponded to the prior sites of second-phase particles which were plucked out during metallographic preparation. There were three reasons why they were less likely to be voids: (a) the testing time was too short to induce any significant creep damage in this specimen; (b) (following the previous point) the dimension of them was much larger than early voids; and (c) most of the pits were not observed in the unetched state. Therefore, it could be inferred that the raw material contains a certain amount of large second-phase particles. The formation/occurrence of them will be discussed in more details by using the SE signals.

During the last step of silica suspension polishing, the sample surface was slightly etched, with second-phase particles abraded away. Therefore in an SE image, these spots are shown in dark. Figure 4-10 illustrates two examples of different FB2 samples. Both of them demonstrated that the FB2 material contained a significant number of second-phase particles. (On the other hand, for the 10Cr steel, such big particles could only be occasionally encountered, e.g. maximum two to three spots in 1mm²). Although limited specific reports on
the formation of inclusions in FB2 steel could be found, there have been parallel studies on similar steels.\textsuperscript{185-187} Generally, the large second-phase particles (i.e. typically larger than 1μm) are most likely to be either non-metallic inclusions or boron nitrides, or complex inclusions (Gianfrancesco has found that for a trial FB2 rotor steel, BN particles exist in the form of joined particles with other inclusions/oxides, which have the size of several microns).\textsuperscript{172}

![Figure 4-10 Example of FB2 samples showing high amounts of second-phase particles. (Some of them are highlighted with red arrows).](image)

The precipitation of non-metallic inclusions occurs during the steel making process. According to their origins, they can either be the result of reaction (e.g. oxides, sulphides, nitrides, silicates or phosphides), or residual particles from the environment (e.g. slags, fluxes). Reducing the amount of non-metallic content (or at least reducing the dimension of inclusions) has always been the pursued objective in steel production (i.e. to make clean steels), because their existence can normally impose a negative influence on the mechanical properties of steels. Although no specific study on the inclusion content of the FB2 steel could be found, a related study on the CB2 steel (a cast steel of similar composition) demonstrated that the dimension of inclusions can be as large as 10μm (certainly cast steel and forged steel are different).\textsuperscript{187} As far as the creep-fatigue loading is concerned, these inclusions may cause early microcracks in the body.

On the other hand, the formation of boron nitrides can take place in high Cr ferritic heat resistant steels (which contain boron). The quantity and dimension of them are strongly dependent on the content of boron and nitrogen, as well as the heat treatment. For example, under very fast cooling, fine boron nitrides can precipitate in the same way as the strengthening MX particles (typically smaller than 0.1μm); but when the cooling rate is reduced, BN particles with the size of 1μm to 3μm can appear, or constitute agglomerated groups with even larger dimensions (e.g. 5~20μm).\textsuperscript{185, 186} Although the heat treatment for the FB2 steel involved water quenching, it was difficult to ensure a homogeneous fast cooling rate in the relatively large/thick component, therefore it was possible that such large boron nitride precipitates could be formed. (This speculation should be verified by chemical analysis in future work).
One example is demonstrated by a BSED image in Figure 4-11. This sample came from a continuously cycled test, which in principle should not exhibit any significant creep damage. The measured area was nowhere near the cracking plane, i.e. this location hadn’t experienced a lot of plastic deformation.

![Figure 4-11 Inclusion/boron nitride-induced microcracks within the sample.](image)

However, due to the existence of second-phase particles (5~10μm), microcracks appeared in this specimen. It seemed that the microcracks originated from a line of particles, and by linking several neighbouring microcracks a larger crack could be formed. In other words, these big second-phase particles were able to induce unexpected microcracks, even though the creep deformation was minimal. Due to the higher amount of large particles in the FB2 steel under investigation, its resistance against creep-fatigue deformation might be smaller than estimated. This was consistent with the finding that cracks propagated faster in the tested FB2 steel than in the tested 10Cr steel (see Chapter 3), although theoretically the designed creep resistance of FB2 should be larger.

It should be emphasized again that the cracking phenomenon in Figure 4-11 was assisted by the presence of inclusions rather than by creep. (True creep-assisted micro-cracking will be illustrated by long crack growth samples in Appendix D, i.e. Figure D-16 and Figure D-17). In fact, for short crack growth tests, even under the most severe loading condition (with 60min hold time), only a very limited number of microcracks could be detected. Certainly the primary reason behind this was that both 10Cr and FB2 are creep resistant steels. In addition, this could be due to an insufficient creep time, or due to the fully reversed loading condition (i.e. $R_c = -1$).

As can be already spotted in Figure 4-11, there were some brighter particles within the matrix, which were regarded as precipitates (carbides, carbonitrides, etc.). Because their ability to
scatter back electrons was stronger than the matrix (resulting from a higher atomic mass), more electrons were scattered back at these precipitates (thus they appeared to be brighter).

It has been introduced previously that both FB2 and 10Cr are tempered martensitic heat resistant steels, in which precipitates are designed to disperse along the grain boundaries and within the matrix. Therefore, it should be possible to observe a large amount of precipitates already in the as-received state. During subsequent deformation, a well-studied recovery process takes place, involving the competitive growth of these precipitates, or even the generation of new precipitates (normally at a higher temperature and in a much longer time scale).\textsuperscript{97, 188, 189, 190} Because BSE signals are especially sensitive to different phases as well as crystallographic orientations, they can serve as a useful tool to examine the tempered martensitic structure.

Figure 4-12 and Figure 4-13 present some examples of BSE images for FB2 and 10Cr steels in this study. Although the samples have not been etched, they could display a distinctive martensitic structure.

Figure 4-12 BSE images of different samples for the FB2 steel: (a) as-received (b) continuously cycled (c) with 30min tensile dwell (d) and with 60min tensile dwell.
Microstructural analysis

Figure 4-13 BSE images of different samples for the 10Cr steel: (a) as-received state (b) with 30min tensile hold (c) with 30min compressive dwell (d) with 60min tensile dwell.

By comparing images from different samples, some important findings could be revealed:

(1) Precipitates tended to locate on all sorts of boundaries, but the quantity and distribution of them were dependent on testing conditions. In the as-received samples (Figure 4-12a and Figure 4-13a), precipitates were mostly detected on large-scale boundaries, e.g. prior austenitic grain boundaries or packet boundaries. It was hardly possible to outline the fine lath structure through these particles. In addition, they did not distribute homogeneously in the as-received state (isolated precipitates could often be observed). On the other hand, fine precipitates were evenly spread throughout large-scale and small-scale (block, lath) boundaries in dwelled samples (Figure 4-12cd and Figure 4-13bcd). The dimension and distribution of these particles were more uniform than in the as-received state. As for the continuously cycled case (Figure 4-12b), it seemed that the appearance of precipitates was intermediate between the as-received state and the dwelled state.

(2) The morphology of the initial lath martensitic structure has evolved together with the creep-fatigue deformation. Because the BSE signal was capable of resolving agglomerations of
fine arrangements, different microstructural features could readily be identified. For the as-received and continuously cycled samples (Figure 4-12ab and Figure 4-13a), the block/lath shape was quite obvious and strong directional structures were perceptible. However, after heavy creep-fatigue deformation, this type of structure could be changed. As demonstrated in the other images, the quantity of lath structures was considerably decreased, while lots of equiaxed structures have emerged. This was in accordance with the recovery process, as micro-grains surrounded by boundaries tended to form equiaxed assemblies to minimize their energy due to the distorted configuration.

Note that the images in Figure 4-12 and Figure 4-13 were taken from the bulk area on the samples. Even though the variation along the crack path was not yet considered here, the evolution of microstructure under the influence of creep-fatigue deformation could already be witnessed. This directly led to the concern of how this change would affect the crack propagation behaviour. In order to answer this question, it was necessary to qualitatively and quantitatively characterize the change of microstructural features. In terms of creep-fatigue loading, the immediate targeted quantity is the micro-grain size, and analysis on this is elaborated in the next section by means of the EBSD measurement.

4.2 EBSD observation

For this grade of steel, there have been already some studies on the fine microstructure at the micro/nano scale. Most of them were done using TEM (transmission electron microscopy) (e.g. Ref.97, 132, 189, 191), whereas EBSD analyses specifically with respect to creep-fatigue were relatively few (e.g. Ref.134). In fact, for the observation of microstructural evolution near a propagating crack (just like in the current study), there are three major advantages of the EBSD technique over the TEM technique:

1. The targeted areas were situated immediately adjacent to crack propagation paths. Material in intensified deformation/oxidation zones should not be represented by microstructural investigation in the bulk. This means that the prepared samples preferably retained the whole crack fronts. This would be an extremely difficult sample preparation process for the TEM method, but caused not much trouble for the EBSD method.

2. The crack depth was normally over several millimetres, which implied that the inspected area should be large enough to reflect any microstructural evolution along the crack propagation direction. While TEM is good at resolving detailed features in a small area, its limitation on large-scale sampling is apparent. This is also connected to another issue, which is whether the results from TEM would actually represent the true characteristics in case of microstructural inhomogeneity in complex martensitic structures. In this sense, the EBSD method is more adequate (sample areas can be much larger).

3. One of the prominent features in martensite is that all its morphological features are associated with crystallographic orientations. While obtaining crystallographic information in TEM is not so straightforward, the unique advantage of the EBSD technique is an
automated detection of orientations in every sampling pixel. With this knowledge at hand, it is easier to determine the degree of evolution for relevant microstructural entities.

### 4.2.1 Proper representation of EBSPs

Before starting an official test campaign, it was necessary to find out the optimum scanning parameters (e.g. step size) for the materials under investigation. Thus, four EBSD measurements using reduced step sizes/sampling sizes were first carried out, and their coloured maps of IPF are shown in Figure 4-14.

![Figure 4-14 Coloured maps of IPF measured by different step sizes at various scales.](image)
4.2 EBSD observation

The corresponding step sizes are listed in Table 4-1

<table>
<thead>
<tr>
<th>Sampling area (μm²)</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step size (μm)</td>
<td>340x340</td>
<td>80x80</td>
<td>20x20</td>
<td>4x4</td>
</tr>
</tbody>
</table>

By comparing images in Figure 4-14, several conclusions could be drawn:

1. Typical tempered martensitic sub-structures were observed, with similar morphologies to those reported in the literature.\textsuperscript{140, 155, 192} Prior austenite grains can be bigger than 340μm (Figure 4-14a), thus the EBSD technique is not as efficient as optical microscopy for quantifying the size of prior austenite grains.

2. Following the first conclusion, in more magnified images (Figure 4-14bcd), the chance of capturing the interior area within grains was higher. In other words, the identified boundaries are more likely to be block, lath, or subgrain boundaries, rather than prior austenitic grain boundaries or packet boundaries.

3. It is clear from Figure 4-14d that the smallest microstructural feature in this material is around 0.1μm. In order to fully reveal these entities, the scanning step size should be smaller than this dimension. However, in some related publications on similar steels, the utilized step sizes ranged from 0.1μm to 0.5μm, or even over 1μm.\textsuperscript{193-195} It was highly possible that many microstructural features were overlooked by such large step sizes. Therefore, a maximum step size of 0.08μm was adopted in this study to guarantee an appropriate resolution.

4. The local microstructure was not totally homogeneous, as shown in Figure 4-14. In this respect, the sampling area should be large enough to suppress the microstructure unevenness. However, in reality a large sampling area usually costs much more resources. As an adequate compromise, it was finally decided to use an 8x8μm² sampling area, together with a 0.05μm step size for most of the scans. In addition, scans with a 20x20 μm² sampling area combined with a 0.08μm step size were also carried out for a broader view.

After the scanning parameters were all set, systematic EBSD measurements were performed. It was also worth mentioning here that in order to minimise the uncertainties associated with phase/orientation identification, a recommended three-stage clean-up process was executed, namely by 'Grain CI Standardization', 'Neighbour Orientation Correction' and 'Grain Dilation'. These clean-up functions have already been integrated in the OIM software.

The reason for this clean-up process was mainly due to the indexing difficulties at grain boundaries, where the superimposed patterns could lead to low quality indexing. Since both FB2 and 10Cr materials in the current study exhibited large numbers of precipitates (which were incoherent compared to the matrix), it was even more essential to implement the clean-
up process. Typically, after this process, about 0.1% of the scanning data would be optimized, which vice versa indicated that the original data was not over corrected.

To get rid of a few bad points, a parallel routine was to define the minimum CI. In the following analysis, the minimum CI was set to 0.1. Therefore, a few spots (especially on the boundaries) would be shown in total black, simply because they were disregarded.

The best way to express the raw scanned data is to plot different types of figures. As introduced before (i.e. in section 1.6.2), IQ map and coloured map of IPF are two commonly used images. The standard analysing scheme in this study included those two figures with/without boundary misorientation (representation by line segments), as well as the KAM (Kernel Average Misorientation) map. Two examples are given in Figure 4-15 and Figure 4-16, for FB2 and 10Cr in the as-received state.

As can be seen, coloured maps of IPF looked like the enhanced version of IQ maps, with different orientations designated with different colours. However, IQ maps could better represent boundaries and orientation gradient within the grain. But the disadvantage was that an IQ map could easily be affected by surface qualities, e.g. the one in Figure 4-16 was less sharp than the one in Figure 4-15.

For both FB2 and 10Cr materials in the as-received state, the coloured maps of IPF clearly indicated a block-lath morphology, where the strong directional feature could be viewed from the colour difference. From IQ maps, networks of grain boundaries could be observed, and judging from the construction of these networks it seemed that Figure 4-15 has captured an area within a single packet, whereas Figure 4-16 has captured a triple junction of prior austenitic grain boundaries.

In order to find out more information regarding boundary types, it was possible to directly impose the rotation angle values on IPF maps or IQ maps by categorizing angles into three ranges, namely, small angle (2°~5°), medium angle (5°~15°) and large angle (15°~180°). They were practically represented by coloured line segments in red, green and black/blue, respectively.

Alternatively, a KAM map could be plotted, which actually emphasized the misorientation gradient in the matrix on a kernel scale (difference between orientation value at targeted point and averaged value of neighbouring points). However, as demonstrated in Figure 4-15 and Figure 4-16, KAM maps are not really suitable for the materials under investigation, because there are just so many boundaries.

By such an analysis scheme, raw scanning records were graphically rationalized, which laid the foundation for the next discussion, i.e. distinguishing between different sub-structures.
Figure 4-15 Typical analysis scheme in this study: (from left to right, top to bottom) coloured map of IPF; IQ map; IPF map overlapped with different misoriented boundaries; IQ map overlapped with different misoriented boundaries; KAM map; and fraction of different boundaries for FB2 steel in the as-received state.
Figure 4-16 Typical analysis scheme in this study: (from left to right, top to bottom) coloured map of IPF; IQ map; IPF map overlapped with different misoriented boundaries; IQ map overlapped with different misoriented boundaries; KAM map; and fraction of different boundaries for 10Cr steel in the as-received state.
4.2 EBSD observation

4.2.2 Distinction between sub-structures

As introduced previously, one unique feature of the martensitic structure is the confined orientation dependence during formation (section 1.5.1 and 1.5.2). This characteristic can be approximated by calculated crystallographic orientation relations such as the K-S or N-W relationships. If such relationships also exist for the tested materials, then the process of identifying different sub-structures can be significantly facilitated.

One thing which should be noted here is that the materials investigated have gone through martensitic transformation as well as tempering. Technically, they can no longer be considered as pure martensites, but recovered sub-structures with a quasi-martensitic morphology. Accordingly, the ‘lath’ structure, which represents the smallest unit in lath martensites, will also decompose into a smaller sub-structure, i.e. subgrains. Misorientations between various sub-structures were carefully measured in both materials, in the as-received or deformed states. This was done by drawing a vector across several boundaries, where the orientation information of each passing points was extracted. After automatic calculation, the misorientation profile chart could be acquired.

In the following content, investigation on the misorientation profile is illustrated for the as-received materials (at a larger sampling area, i.e. 16x16 or 20x20μm² for larger sub-structures), and creep-fatigue deformed testpieces (at a smaller sampling area, i.e. 8x8 μm² for smaller sub-structures).

Figure 4-17a displayed a coloured map of IPF for FB2 in the as-received state, where the blocks/laths were clearly visible.

Figure 4-17b was overlapped with the rotation angle information, and denoted the location of the selected line vector.

Figure 4-17c was the misorientation profile chart along this vector, and the intercepted reference IPF was also superimposed for convenience. It was then possible to compare the misorientation values across various boundaries.

From the first impression, the values of misorientation could be categorized into two favoured ranges: 2°~10° and 50°~60°, corresponding to low-medium angle grain boundaries and high angle grain boundaries. There was also some noise (mostly below 1°), which was attributed to the orientation gradient and angular resolution of the microscope.

More specifically, most of the detected boundaries were high angle grain boundaries, which were more likely to be block boundaries (due to the two-tone colours, this sampling area should be within the same packet). A few smaller peaks (between 2° to 10°) could also be noticed in Figure 4-17c, which were supposed to be lath/subgrain boundaries. The observations were consistent with those reported on martensitic steels in the literature.¹⁴¹, ¹⁴³
Parallel analysis was also done for 10Cr, as displayed in Figure 4-18. Similar to FB2, the high angle grain boundaries were larger than 50°, whereas the others were smaller than 10°.

However, due to the relatively large sampling area, the detailed orientation information regarding the smaller sub-structures could not be fully revealed in Figure 4-17 and Figure 4-18. Thus two examples were selected from the slightly deformed state (EBSP measured near crack starter), one for each material.
4.2 EBSD observation

Figure 4-18 Misorientation profile for 10Cr in the as-received state.

Figure 4-19 Misorientation profiles parallel to the block/lath direction for FB2 in the slightly deformed state: (a) first vector; (b) second vector; (c) misorientation profile for the first vector; and (d) misorientation vector for the second vector.
In Figure 4-19, two vectors were chosen to be parallel to the block/lath boundary direction, targeting several equiaxed cells in the same block (based on the similar colour span). The red curve in the misorientation profile chart denoted the point-to-point misorientation angle, whereas the blue curve denoted the point-to-origin misorientation angle. As expected, the visible boundaries were all misoriented from 2° to 11°. But how to tell if they were lath boundaries or subgrain boundaries? Here comes the debatable definition of a subgrain: some researchers link subgrain directly to prior lath (and this leads to a classification of larger misorientation angles, e.g. >5°); some other researchers only regard the newly developed sub-structures (during recovery and creep, etc. which has inevitably small misorientation angle, e.g., <5°, or even <1°) or simply define those with low misorientation angles as subgrains.

By examining results in this study, it was found that those boundaries could indeed be divided into two sub-groups, i.e. 5°-15° and 2°-5°, corresponding to prior laths (inherited subgrains) and newly developed subgrains (thin walls of dislocations).

Figure 4-20 Misorientation profiles vertical to the block/lath direction for FB2 in the slightly deformed state: (a) first vector; (b) second vector; (c) misorientation profile for the first vector; and (d) misorientation vector for the second vector.
Additionally, in this case the point-to-origin misorientations were almost monotonically increased, which exhibited a gradient as high as 20°. This probably indicated that the dislocations were of the same sign along this direction inside a prior lath, and subgrain boundaries were overwhelmed by one kind of dislocation.

Previously, it was also not straightforward to distinguish between block boundaries and lath boundaries by solely looking at OM images or SEM images. However, with orientation information, it was then possible to determine the type of each boundary. This is illustrated in Figure 4-20. Vectors were drawn transverse to the lath/block direction, and the point-to-point as well as point-to-origin misorientations were outlined.

Three inferences could be made together with the morphological observation: (1) blocks (with high angle grain boundaries) could also be constructed from only one or two rows of laths; (2) there was no long-range cumulated misorientation across block boundaries; and (3) some of the block boundaries exhibited misorientations between 15° and 45°. Boundaries within this angle range were not stable and highly mobile. This potentially reflected the fact that during creep-fatigue deformation, various types of boundaries were able to travel, and the corresponding misorientation across boundaries could also evolve (to be discussed in more details in the next section).

Figure 4-21 Misorientation profiles vertical to the block/lath direction for 10Cr in the slightly deformed state: (a) first vector; (b) misorientation profile for the first vector; (c) second vector; and (d) misorientation vector for the second vector.
Finally, in order to verify the findings in FB2 samples, misorientation profiles for 10Cr in a slightly deformed state were also studied. This is illustrated in Figure 4-21, with two vectors parallel to the lath/block direction. The exactly same phenomena could be identified: subgrain boundaries were smaller than $5^\circ$; lath boundaries were mostly between $5^\circ$ and $10^\circ$; and block boundaries were high angle grain boundaries, with the tendency to grow during deformation.

4.2.3 Evolution of morphology and boundary misorientation

During creep-fatigue deformation at elevated temperatures, recovery normally takes place, and its degree is dependent on the testing conditions. Through recovery, microstructure usually develops into a more uniform, lower energy state, which is associated with the reduction of lattice defects. In other words, local inhomogeneity like dislocation and sub-boundary conditions will evolve, typically in the form of dislocation annihilation and sub-boundaries formation/growth (see also section 1.5.3).

Because the mechanical properties of a material are the external manifestation of the interior microstructure, the capability of a material to resist creep-fatigue deformation is directly connected to the magnitude of the recovery process. As introduced before, the entities to characterize the recovery process are dislocations and sub-boundaries. However, it is rather difficult to accurately determine dislocation densities in materials involved in this study, not only because of the martensitic structure, but also due to the fact that during reversed plastic deformation dislocation multiplication can occur. Therefore, in this study the focus was applied on the variation of sub-structures, which could be well supported by the EBSD technique. (Another possible characteristic entity is the number/size of precipitates. In some studies on 9Cr steels, it was found that more/larger precipitates could be revealed after creep-fatigue deformation,97, 189, 190 However, the dimension of precipitates is normally within the range of nanometres, which requires more sophisticated measurements to investigate, e.g. TEM).

It has already been demonstrated in previous sections that the martensitic sub-structures are directly linked to the defined orientation relationships, and thus by examining coloured maps of IPF it should be possible to qualitatively observe the evolution of microstructure. Indeed, along with the creep-fatigue deformation/recovery process, considerable changes of sub-structures were witnessed.

An example is given in Figure 4-22, where IQ maps and corresponding coloured maps of IPF are displayed. In order to provide information with regard to sub-boundaries, line segments were highlighted with different colours, denoting $2^\circ$–$5^\circ$ misorientation (red), $5^\circ$–$15^\circ$ misorientation (green) and $15^\circ$–$180^\circ$ misorientation (black/blue). Figure 4-22a, Figure 4-22b and Figure 4-22c were measured from a continuously cycled short-crack creep-fatigue crack growth test specimen, a dwelled long-crack creep-fatigue crack growth test specimen (see Appendix D), and a dwelled short-crack creep-fatigue crack growth test specimen, respectively (all of them are 10Cr testpieces).
4.2 EBSD observation

(a) From continuously cycled short-crack creep-fatigue crack growth test

(b) From dwelled long-crack creep-fatigue crack growth test (Appendix D)

(c) From dwelled short-crack creep-fatigue crack growth test

Figure 4-22 IQ maps and coloured maps of IPF from three different 10Cr testpieces. Boundaries were highlighted with coloured line segments: red for 2°–5°, green for 5°–15° and black/blue for 15°–180°.
Note that the sampling areas were all selected in the bulk material, i.e. remote from the intensified cracking region. Therefore they should represent the influence only from creep-fatigue deformation.

By comparing these images, it could be immediately noticed that the deformed morphology was strongly related to the testing condition, i.e. creep-fatigue deformation. While Figure 4-22a was not far from the as-received state, the sub-structure in Figure 4-22c has deviated significantly. More exactly, the low to medium angle (2°~15°) boundaries have strongly evolved during deformation, which presumably indicated that the lath and subgrain structures were coarsened by competitive growth. This led to a diminishing of the directional lath structure, where the sub-structures exhibited a more equiaxed network appearance. As far as the block structure was concerned, its directional morphology still remained, but it seemed that the number of block boundaries has decreased. However, this point requires further verification, since the original dimension of block structures are not homogeneous in the tested materials.

Figure 4-23 Change of misorientation across boundaries during deformation in 10Cr testpieces. Boundaries were highlighted with coloured line segments: red for 2°~5°, green for 5°~15° and black/blue for 15°~180°.
As can be seen from the coloured line segments in Figure 4-22, the change of sub-structure was likely to be associated with the adjustment of misorientation across sub-boundaries. This point is further elucidated in Figure 4-23.

In Figure 4-23a, two areas were denoted by the numbers 1 and 2, where the surrounding boundaries were pointed out by black arrows. Judging from its shape and the neighbouring sub-structures, area 1 was possibly a prior lath. During previous analysis as well as illustrated partly in Figure 4-22, lath boundaries should have a larger misorientation than subgrain boundaries, e.g. >5° (in most cases, this criterion works for the materials under investigation). However, the boundary enclosing area 1 was partly red and partly green, and the reason behind this could be intuitively viewed in the accompanying IQ map: this boundary could be the combined result from two prior parallel lath boundaries. Moreover, judging from the curvature of this boundary, the dimension change of this lath was assisted by the intrusion and extrusion of the sub-boundaries. (This was consistent with the reported recovery mechanism of the repeated bulging and migration of local parts of the lath boundaries).

Area 2 seemed to be a newly formed subgrain. Although its boundary could not be fully outlined in the coloured map of IPF, it was possible to identify it clearly in the IQ map. In other words, the misorientation along this subgrain boundary was mostly smaller than 2°. In fact, the modern EBSP acquisition device is totally capable of precisely resolving the misorientation down to 1°. The reason 2° was chosen as the minimum tolerance angle was on one side to be according to the generally accepted practice, and on the other side to suppress the noise from the large orientation gradient in the tested materials. (Theoretically, newly formed subgrains are composed of thin agglomerated dislocation walls. Assisted by plastic deformation, these subgrain boundaries tend to migrate quickly, because there are normally no segregated particles to pin them. By further absorbing free dislocations, misorientation at a subgrain boundary is likely to increase, i.e. >2°. Some of them can also accumulate to an angle higher than 5°, as displayed in Figure 4-22 and Figure 4-23).

Two areas were also marked in Figure 4-23b, with boundaries pointed out by the blue arrows. From the quadratic shape, it was hard to tell if area 1 and 2 were joined lath structures, or subgrains. But judging from the boundary misorientations, they were more likely to be coarsened laths, and the emphasis in this image was exactly on these values. As depicted in the coloured map of IPF, the boundaries encircling area 1 and 2 were partly green (5°~15°) and partly black (15°~180°). This potentially inferred that during creep-fatigue deformation, the migrating lath boundaries had the chance to accumulate enough misorientation to become high angle grain boundaries. Strictly speaking, at this time the ‘recovery’ procedure could no longer be regarded as recovery anymore, because the movement of sub-boundaries was associated with the formation/migration of high angle grain boundaries, i.e. within the scope of recrystallization.
4.2.4 Grain size variation along the crack propagation direction

As explained in the previous section, the morphology evolution and sub-structure development were closely dependent on creep-fatigue loading conditions. The differences between samples were already observed, and now it is interesting to see whether that applied also to the near crack regions. Because theoretically speaking, these regions sustained magnified creep-fatigue loading, which would correspond to more recovered sub-structures.

In addition, there should be a variation of the microstructure along the crack propagation direction. Material adjacent to the cracking plane was mainly subjected to two stages of loading: (1) when the crack front was still remote, with material (horizontal to the propagating crack) in the remaining ligament experiencing a slightly increased creep-fatigue loading relative to the bulk material, owing to the reduced cross-section; (2) when the crack front was immediately adjacent, with the near crack region suffering from a much heavier creep-fatigue loading relative to the bulk material, especially for the dwelled specimen where creep damage built up at the crack tip. After the major crack has penetrated through this region, the local material would only be influenced by aging, which in general could not induce any significant change in microstructure in a relatively short time. Therefore, materials at the near crack region should be able to represent the instantaneous microstructure, associated with the crack development; and a region with a larger crack depth should in principle have a more evolved microstructure than a region with a smaller crack depth (on the same sample), due to the longer period of creep-fatigue deformation.

With this assumption in mind (i.e. last paragraph), an evolution of sub-structure/morphology at the near crack region was expected to take place in various testpieces. With the help of the EBSD technique, this pattern could be observed in most of the measurements. Examples are given in Figure 4-24, where six samples from short-crack creep-fatigue crack growth tests are illustrated. It should be noted that for this series of measurements, all the sampling areas were chosen extremely close to the cracking plane, i.e. maximum 20μm away from the major cracks. In this way, they should be capable of representing sub-structures in the heavily deformed near crack regions.

For FB2 material, a continuously cycled sample (Figure 4-24a), a 30min tensile dwelled sample (Figure 4-24c) and a 60min tensile dwelled sample (Figure 4-24e) are shown; for 10Cr material, a continuously cycled sample (Figure 4-24b), a 30min tensile dwelled sample (Figure 4-24d) and a 30min compressive dwelled sample (Figure 4-24f) were also presented. Judging from the morphology (colour sections), it could be ascertained that:

1) All the sub-structures (blocks, laths, subgrains) could evolve under creep-fatigue deformation.

2) In each sample, material associated with a larger crack depth was more recovered.

3) Dwelled specimens apparently recovered further more compared to continuously cycled specimens.
4.2 EBSD observation

(a) Coloured maps of IPF measured at different crack depths near the cracking plane for FB2 continuously cycled specimen (FB2-RB01)

(b) Coloured maps of IPF measured at different crack depths near the cracking plane for 10Cr continuously cycled specimen (10Cr-RB01)

Figure 4-24 Comparison of coloured maps of IPF measured at different crack depths on different specimens.
Microstructural analysis

(c) Coloured maps of IPF measured at different crack depths near the cracking plane for FB2 30min tensile dwelled specimen (FB2-RB06)

(d) Coloured maps of IPF measured at different crack depths near the cracking plane for 10Cr 30min tensile dwelled specimen (10Cr-RB09)

Figure 4-24 Comparison of coloured maps of IPF measured at different crack depths on different specimens.
(e) Coloured maps of IPF measured at different crack depths near the cracking plane for FB2 60min tensile dwelled specimen (FB2-RB07)

(f) Coloured maps of IPF measured at different crack depths near the cracking plane for 10Cr 30min compressive dwelled specimen (10Cr-RB11)

Figure 4-24 Comparison of coloured maps of IPF measured at different crack depths on different specimens.
It was not convincing enough to just make qualitative comparison. To reduce the subjectivity, it was necessary to take a step forward by evaluating grain sizes in most of the measurements.

Actually, in such a hierarchical sub-structure in tempered/recovered martensite, the way to represent a ‘grain’ was not straightforward. Due to the complexity associated with different kinds of boundaries, it was difficult to apply the traditional concept of grains. As a compromise, the criterion of a 2° tolerance angle was used, as the lower limit for the misorientation across any boundary (in accordance with the previous analysis). In fact, the current scheme was not to distinguish between various kinds of boundaries, but to uniformly regard them as sub-boundaries. Thus, any area surrounded by sub-boundaries was referred to as a micro-grain (similar to the definition in Ref.\textsuperscript{144}). Note that micro-grains were not confined to subgrains, but could comprise also larger sub-structures.

The measurement of micro-grain size was done by a classical line-intercept method in scanned areas. Multiple lines were drawn across the sampling region, and the average intercept length could represent the mean diameter of the micro-grains, as long as enough lines were applied. In practice, 50 to 80 lines were used both vertically and horizontally. Finally, the characteristic micro-grain size for each specimen at various crack depths could be determined. Some of the results are listed in Table 4-2 for FB2 specimens and Table 4-3 for 10Cr specimens.

<table>
<thead>
<tr>
<th>FB2</th>
<th>Tolerance angle: 2°</th>
</tr>
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<tbody>
<tr>
<td>RB01 $t_h = 0$</td>
<td>Crack depth (mm) 0.2 0.5 2 3 3(M) 3(LM)</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm) 0.82 1 1.17 1.54 1.28 1.23</td>
</tr>
<tr>
<td>RB06 $t_h = 0.5h$</td>
<td>Crack depth (mm) 0.2 2.2 4.2 5.2(L) 5.2(M) 5.2(LM)</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm) 0.87 1.57 1.96 1.89 1.83 1.67</td>
</tr>
<tr>
<td>RB07 $t_h = 1h$</td>
<td>Crack depth (mm) 0.2 0.6 1 1.3 3.2(M)</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm) 0.82 1.13 1.02 1.17 2.67 1.32</td>
</tr>
<tr>
<td>RB07*</td>
<td>Crack depth (mm) 0.3 0.4 0.5 0.6</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm) 1.13 0.90 0.81 1.115</td>
</tr>
<tr>
<td>RB07**</td>
<td>Crack depth (mm) 0.4 0.7 1 1.3 2.3</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm) 1.2 1.19 0.85 1.37 1.76</td>
</tr>
</tbody>
</table>

* and **: two interrupted tests with the same testing condition as RB07.
(M): measured in the matrix, far away from the cracking plane.
(L): measured in a larger sampling area, i.e. 20x20 μm$^2$.

In Table 4-2, the focus was applied on the influence of hold time. FB2-RB01, RB06 and RB07 were specimens without hold time, with 30min tensile hold and 60min tensile hold, respectively (in accordance with Figure 4-24a,c,e). RB07* and RB07** were two interrupted tests under the same testing condition as RB07, but with different terminating crack depths (detailed testing conditions can be found in Table 2-5 in Chapter 2). Therefore, their results were also included for reference. In addition, the difference between the near crack region and the remote area was studied, by measuring the microstructure in the matrix far away from the cracking plane (denoted by the superscript M in Table 4-2). Last but not least, the superscript L represented a larger sampling area (i.e. 20x20 μm$^2$, instead of 8x8 μm$^2$).
4.2 EBSD observation

Clearly, it was found that the micro-grain size measured in the matrix was smaller than its counterpart measured in the near crack region, just as anticipated. This was certainly due to the fact that the matrix material underwent less recovery than the material close to the crack. For the measurements with a larger sampling area, a larger micro-grain size was initially expected, because the step size was bigger (0.08μm instead of 0.05μm). But in reality, the estimated micro-grain size in a larger sampling area was slightly smaller than that in a standard sampling area. This was perhaps partly owing to the inhomogeneity of the material itself, and partly caused by the high orientation gradient (because some more sub-boundaries were able to reach the critical value of 2° with a larger step size).

<table>
<thead>
<tr>
<th>10Cr</th>
<th>Tolerance angle: 2°</th>
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<tbody>
<tr>
<td></td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td>RB01</td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>t_h = 0</td>
</tr>
<tr>
<td>RB06</td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td>RB08</td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>t_h = 0</td>
</tr>
<tr>
<td>RB09</td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td>RB10</td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>t_h = 1h</td>
</tr>
<tr>
<td>RB11</td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td></td>
<td>t_h = 0.5h</td>
</tr>
<tr>
<td>CT04</td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td></td>
<td>Micro-grain size (μm)</td>
</tr>
<tr>
<td>t_s = 600s</td>
<td></td>
</tr>
</tbody>
</table>

In Table 4-3, the micro-grain size evolution in various testing conditions could be seen for the 10Cr samples. 10Cr-RB01, RB06 and RB08 were three samples from the continuously cycled tests; 10Cr-RB09, RB10 and RB11 were three samples from dwelled specimens.

Figure 4-25 Micro-grain size plotted as a function of crack depth for (a) FB2 specimens from Table 4-2 and (b) 10Cr specimens from Table 4-3.
It was then possible to plot the values of micro-grain size in Figure 4-25 according to Table 4-2 and Table 4-3. Due to the inherent scattering in the microstructural measurements, the patterns could not be fully revealed by directly comparing values in Figure 4-25. Therefore, a curve-fitting process was carried out to extract the main evolving path of micro-grain size associated with the crack development (in the short crack regime, i.e., until 2mm). In order to improve the accuracy in this regression process, a unified starting value for micro-grain size was required. By comparing micro-grain sizes in the as-received state, it was found that a starting value of 0.8μm was appropriate for both materials.

![Figure 4-26 Evolution of micro-grain diameter along the crack propagation path for FB2. (Fit from Figure 4-25a, truncated to 2mm crack depth).](image)

![Figure 4-27 Evolution of micro-grain diameter along the crack propagation path for 10Cr (the curve denoting 60min dwelled case was extrapolated to 2mm crack depth). (Fit from Figure 4-25b, truncated to 2mm crack depth).](image)
Finally, the evolution of micro-grain diameter along the crack propagation path is shown in Figure 4-26 for FB2 and in Figure 4-27 for 10Cr. In both figures, the effect of hold time was apparent: a 30min hold time would increase the micro-grain size considerably, while a 60min hold time further boosted this increment. For both materials, the evolving paths for continuously cycled tests were similar: the micro-grain size reached about 1.2μm at 2mm crack depth. However, for dwelled samples, things were different.

Compared to FB2, 10Cr clearly had a quicker expansion of micro-grain diameter near the beginning of the creep-fatigue tests (at the initial crack starter, i.e. 0.2mm crack depth). Afterwards, the micro-grain size developed even larger in 10Cr, ending up with much bigger values at the 2mm crack depth (e.g. about 2.3μm in 10Cr instead of 1.9μm in FB2 for the 60min dwelled case). This potentially implied that the microstructure in 10Cr steel was more vulnerable to the creep-fatigue deformation. Of course, it could be argued that 10Cr took more cycles to develop the same crack depth, i.e. suffered from more creep-fatigue deformation. However, by checking the cycle number information, this was not the case. For example, 10Cr-RB09 took 330 cycles to grow the crack till 1.5mm, but the micro-grain size at that depth was around 1.7μm, which was even larger than FB2-RB07 at 2mm crack depth (383 cycles). It could therefore be confirmed that as far as the microstructure was concerned, the tested FB2 steel (at 600°C) was more stable than 10Cr steel (at 625°C). (In reality, the FB2 steel is considered to be the stronger candidate for 625°C operating duty, whereas the 10Cr steel is normally adopted for 600°C service. It is acknowledged that a more appropriate comparison would have been between FB2 at 625°C and 10Cr at 600°C. The reason for the anomaly was that the 60min dwelled test for FB2 was carried out at 600°C, whereas its counterpart for 10Cr was conducted at 625°C. The focus for each steel was therefore developed in such a way to maximize the use of the available data to verify/establish creep-fatigue crack growth models).

Moreover, two additional points could be noticed in Figure 4-27. The first was that contrary to expectation, the continuously cycled sample at 625°C had a smaller micro-grain size than the one at 600°C. This was conflicting to the general perception that higher temperature should facilitate the recovery process, thus leading to a larger micro-grain size. However, after examination of the mechanical properties (e.g. life cycle to 2% load drop) of these two specimens, it was found that the one at 625°C actually had a larger number of cycles to 2% load drop point than the one at 600°C (see Figure 3-6). Therefore, the anomaly in sub-structure evolution was more likely to come from the variability of materials mechanical properties. In fact, this point exactly illustrated that mechanical property and microstructure were closely connected.

The second point was that the compressive dwelled sample seemed to have a smaller micro-grain size than the tensile dwelled sample, given the same crack depth. This might partly be due to the severer creep damage accumulation in the tensile dwelled specimen or the faster crack growth rate in the compressive dwelled specimen (less number of cycles until 2mm crack depth). Either way, the recovery of sub-structures was less prominent in the compressive dwelled sample.
In principle, the micro-grain size \( d_m \) should at least be a function of the crack depth \( a \), number of cycles \( N \), test temperature \( T \), hold time \( t_h \) and the total strain range \( \Delta \varepsilon_t \), i.e. in equation (4-1).

\[
d_m = f(a, N, T, t_h, \Delta \varepsilon_t)
\]  

(4-1)

The constitutional expression of \( d_m \) could only be obtained after a rather enormous effort on investigating the mechanical as well as microstructural properties. However, based on the current observations as shown previously, it was possible to semi-empirically model the evolution of \( d_m \) through a set of parameters. Since most of the tests were carried out with \( \Delta \varepsilon_t = 0.01 \) and the same strain rate, in addition to the fact that the influence of temperature on the micro-grain size was not substantial, the targeted number of variables could be reduced to two, i.e. the crack depth \( a \) and the hold time \( t_h \). (In this attempt, the influence of \( N \) is implicitly comprised in fitting parameters and variables). Finally, an expression with four fitting parameters was devised, in the form of:

\[
d_m = d_m_{\text{start}} (1 + b_1 a + b_2 a^c t_h^d)
\]  

(4-2)

where \( b_1, b_2, c_1 \) and \( c_2 \) were parameters related to material, temperature, strain range, cycle number and the position of hold period. Note that the units were \( d_m \) (μm), \( a \) (mm) and \( t_h \) (h). Best fit values (for tensile dwell) in accordance with the curves from Figure 4-26 and Figure 4-27 are listed in Table 4-4, and the effectiveness could be examined in Figure 4-28.

<table>
<thead>
<tr>
<th>Equation (4-2)</th>
<th>( b_1 )</th>
<th>( b_2 )</th>
<th>( c_1 )</th>
<th>( c_2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB2</td>
<td>0.29</td>
<td>0.34</td>
<td>1.0</td>
<td>2.1</td>
</tr>
<tr>
<td>10Cr</td>
<td>0.26</td>
<td>0.85</td>
<td>0.62</td>
<td>0.58</td>
</tr>
</tbody>
</table>

Table 4-4 Values of the parameters describing the evolution of micro-grain size in FB2 (600°C) and 10Cr (625°C).

Figure 4-28 Predicted and experimental values of micro-grain sizes for (a) FB2 at 600°C and (b) 10Cr 625°C (from Table 4-4).

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The open symbols (Figure 4-28) denoted the experimental values of $d_m$, whereas the solid curves represented the predicted values from equation (4-2) by using the best fit values from Table 4-4. A good correspondence could be observed, which presumably implied that this semi-empirical model could be extrapolated to a certain extent to predict the evolution of $d_m$ with different $t_h$ under similar testing conditions.

For example, if $t_h$ is increased from 1h to 5h, then the micro-grain size can be estimated as shown in Figure 4-29. If the boundary case can be verified, then all the intermediate predictions are likely to be reasonable. Certainly the validity of this type of extrapolation on $d_m$ should be limited to a characteristic threshold value, because in reality a saturation effect of the micro-grain size often exists for high chromium tempered martensitic steels. \(^{199}\)

### 4.3 Summary

**OM and SEM observation**

Optical microscopy and scanning electron microscopy were carried out on FB2 and 10Cr samples, mainly for the purpose of: observing the prior austenitic grain structure and the martensitic sub-structure; examining the crack propagation profile; detecting microcracks; and identifying the distribution of precipitates and/or inclusions. After a careful inspection, it can be concluded that:

1. Both materials (in the as-received state) exhibit a typical tempered martensitic morphology, i.e. a prior austenitic grain is partitioned into a number of packets, and each packet is subdivided into several parallel blocks, whereas each block is filled with organized laths (e.g. Figure 4-1 and Figure 4-2). Despite different prior austenitic grain sizes, the dimensions of martensitic features are similar in these two steels (FB2 steel has a larger size of prior austenitic grains, i.e. up to 500μm, compared with 200μm in 10Cr).
2. Upon crack initiation, multiple sub-cracks were generated at the same time, and one of them developed into the major crack (e.g. Figure 4-4a). The major cracking plane was roughly transverse to the loading direction, whereas many branch cracks were generated with an inclined angle to the major crack path.

3. The etched samples revealed that the (major) cracking path was almost fully transgranular under the employed creep-fatigue test conditions (e.g. Figure 4-6 and Figure 4-7). Moreover, the branch cracks also displayed a transgranular profile (e.g. Figure 4-4b).

4. Owing to the tendency to strain enhanced oxidation, all major cracks as well as branch cracks were covered with oxides (e.g. Figure 4-8 and Figure 4-9), which could be as thick as 30μm (up to 80μm in long-term dwelled samples). This inferred that the fresh raw material was actually very vulnerable to oxidation. When a crack tip was pulled open, the newly opened region had a high tendency to oxidize, which in turn weakened the fracture resistance dramatically (oxides are normally much more brittle than metals). This indicates that oxidation is likely to have played an important role in the crack initiation/propagation process.

5. Relatively large second-phase particles (diameter of around 1~10μm) can frequently be found in the tested FB2 steel (Figure 4-10 and Figure 4-11), which are most likely to be non-metallic inclusions. These particles are able to induce unexpected microcracks (even though creep deformation is minimal). This partly explains why the tested FB2 steel has a lower resistance against creep-fatigue deformation/crack propagation than expected.

6. For both FB2 and 10Cr specimens, fine precipitates (diameter smaller than 0.2μm) disperse on all sorts of boundaries (as well as in the matrices, Figure 4-12 and Figure 4-13).

**EBSD observation**

In the main stage of microstructural investigation, EBSD analyses were carried out to systematically investigate the evolution of microstructure (e.g. morphology, size and orientation of martensitic features) under creep-fatigue deformation. The sampling areas were mostly immediately adjacent to the crack propagation path to represent the true microstructural status at the crack tip. Before starting the formal EBSD measurements, the optimal scanning parameters and processing scheme (e.g. the clean-up process, the definition of CI and tolerance angle, and the selection of appropriate plot types) were determined. After that, it was possible to distinguish sub-structures, examine the evolution of morphology and boundary misorientation, and most importantly, analyse the grain size variation along the crack propagation direction. An overview of the findings is as follows:

1. The as-received materials have been tempered, thus the lath structure has (at least partly) decomposed into a smaller sub-structure, i.e. subgrains. (More specifically, the prior lath/block structure exhibited directional outlines, whereas the subgrains displayed
equiaxed shapes). In the examined as-received samples, the misorientation angles of subgrain boundaries, lath boundaries and block/packet boundaries are in principle smaller than 5° (small angle), between 5° and 15° (medium angle), and larger than 15° (large angle), respectively (e.g. Figure 4-17 to Figure 4-20). This finding is consistent with the common view on tempered lath martensitic steels.

2. Blocks may comprise only one or two columns of laths in the tested materials. While there is no long-range (accumulated) misorientation across several blocks, the orientation gradient in a single block can be as high as 20° (Figure 4-19 to Figure 4-21). This reflects the fact that high dislocation densities remain in the matrix.

3. During creep-fatigue deformation, both the morphology and boundary misorientation have evolved (e.g. Figure 4-22). In particular, subgrain coarsening took place, whereas the initial lath structure developed into a more equiaxed configuration by competitive growth, and the number of block boundaries also seemed to be decreased. The misorientation associated with sub-boundaries could either build up (in most cases) or diminish (Figure 4-23). Moreover, a certain amount of inhomogeneity could be observed during the evolution of sub-structures.

4. The degree of microstructural evolution was directly governed by the magnitude of creep-fatigue loading (e.g. dwelled specimens recovered further than continuously cycled specimens). Compared to the matrix, materials at the near crack region were further recovered, primarily due to the enhanced creep-fatigue loading. Along the crack propagation path, immediately adjacent materials also displayed sub-structures in a varying degree of recovery (e.g. Figure 4-24). Usually, larger crack depth was associated with heavier recovery in the sub-structure. The heavily recovered sub-structures can no longer be referred to as martensitic structures. In this sense, various kinds of boundaries were uniformly referred to as sub-boundaries, and the areas surrounded by sub-boundaries as micro-grains in this study.

5. By the line-intercept method, micro-grain diameter \( d_m \) was measured for various samples at different crack depths. It was verified quantitatively that the evolution of microstructure was indeed dependent on both the testing condition and the crack depth (Figure 4-25 to Figure 4-27). Compared with FB2 (at 600°C) samples, 10Cr samples (at 625°C) had a quicker expansion of \( d_m \) near the beginning of creep-fatigue tests, and afterwards \( d_m \) developed even faster. This infers that the microstructure of the tested FB2 steel (at 600°C) is more stable than the 10Cr steel (at 625°C) during creep-fatigue deformation.

6. Based on the investigation of microstructural evolution, it is possible to semi-empirically model the evolution of \( d_m \) through a set of parameters, i.e. \( d_m = d_{m,\text{start}} (1 + b_2 a + b_3 a^2 t_b) \) where \( b_1, b_2, b_3 \) and \( a \) are parameters related to material, temperature, strain range and the position of hold period (e.g. Figure 4-28). Best fit values for the current test set-up have been calculated.

At last, it should be emphasized again that all the above-mentioned estimations of micro-grain diameter were completed under one essential criterion: with a 2° tolerance angle. This value
was actually chosen only after careful consideration. As exhibited in the previous analysis, the newly developed subgrains could be comprised of boundaries with very small misorientation values. However, $1^\circ$ is normally the lower accuracy limit of the EBSD technique (of course during scanning, each adjacent segment can be recognized with any misorientation ranged from $0^\circ$ to $180^\circ$. But the instrument normally has an error band within $\pm0.5^\circ$).

In addition, there were two features in the materials under investigation which deviated from the ideal situation: (1) the orientation spread within prior lath/blocks could be as high as $4^\circ$. In other words, there was a big orientation gradient within micro-boundaries (could be caused by a high dislocation density); (2) segments with low angle misorientations could not always close up, but appeared as isolated boundaries. These segments might not be subgrain boundaries yet, but just clusters of dislocations. Therefore, if the tolerance angle was too small, the images would be overwhelmed by such segments, especially for the less recovered samples.

On the other hand, some researchers consider subgrains with low angle boundaries as some kind of immature recovered structure, and their threshold is set to $5^\circ$. This may be partly due to the fact that during their EBSD scanning, the step size was relatively big, e.g., $0.2\mu m$ to $0.5\mu m$, where the fine sub-structure could not be fully resolved. As highlighted before, for the investigated materials the misorientation of a subgrain boundary could well be smaller than $5^\circ$. But to be on the safe side, micro-grain sizes with the tolerance angle of $5^\circ$ were also measured. As expected, a larger tolerance angle would exclude the lower angle boundaries, thus leading to a larger value of line intercepts. However, the difference was relatively small in the current case. As an example, micro-grain sizes measured with $2^\circ$ and $5^\circ$ tolerance angles in $10Cr$ samples are plotted in a single graph (Figure 4-30). Except for a few outliers, all the values almost fell on the 1:1 line, which indicated that both criteria could give a similar evolution pattern. Although the absolute values were slightly different, the selection of $5^\circ$ as the tolerance angle could in principle also work for the materials under investigation (provided that the sampling step size is small enough).
Chapter 5

Modelling of short crack propagation

In order to rationalize crack growth behaviour under diverse testing conditions, various models have been applied to correlate different parameters with crack growth rates. In this chapter, illustrations of the effectiveness of those models are demonstrated. A brief introduction of these models can be found in Chapter 1, and the most important equations related to modelling are reemphasized during the following elaboration for convenience.

As mentioned before, although most of the short-crack creep-fatigue crack growth tests were terminated only after the relatively unstable load drop took place where the crack had propagated long enough, the criteria for the short crack regime was limited to a 2mm crack depth (the explanation with regard to the relationship between crack depth and the number of cycles can be found in Chapter 3). In other words, in this section of short crack growth modelling, the investigation was restricted to the regime where the crack developed from 0.2mm to 2mm.

In the following sections, the employed models for short-crack creep-fatigue crack development are: (1) the fracture mechanics based models (\(K\) type and \(J\) type); (2) the proposed model based on the SEDF; (3) the Tomkins model; and in particular (4) the Skelton model (as a separate section). Similarities and differences between the employed models are also compared and suggestions are given with regard to their practical application. It has been found that:

1. Compared with \(K\) type or \(J\) type models, the SEDF model appears to be similar but marginally more effective, mainly from two aspects: the systematic deviation between individual crack growth curves resulting from the applied total strain range can be reduced; and the enhancement on crack growth rates owing to the hold periods can partly be included. This indicates that at least with the employed test set-up, the SEDF model is advantageous for short crack growth characterisation.

2. One well known semi-empirical model, the Tomkins model, is able to describe the crack development only when a proper characteristic value \(\bar{T}\) is adopted. In this study, no universal \(\bar{T}\) value can be found to be compatible with all the test conditions (i.e. the optimal \(\bar{T}\) value in each test can only be obtained through regression, and this value varies a lot under different conditions). This is mainly because the Tomkins model was originally developed for pure fatigue tests, not creep-fatigue tests.
3. The other widely accepted semi-empirical model, the Skelton model, can almost predict short crack growth behaviour accurately for all the experiments. The influence of total strain range is accounted for in the $B$ term, whereas the influence of hold period is represented by the creep damage term $d_c$. It has also been found that the upper bound of $B$ in R5 is very conservative for the investigated materials.

4. Apart from the direct curve fitting, $d_c$ can also be estimated from the stress relaxation during hold periods by referring to the creep properties of the materials. By employing the Skelton model with $d_c$ deduced from the ductility exhaustion method, a realistic preliminary estimation of short crack growth behaviour can be obtained.

More importantly, with the support of microstructural analysis originated from Chapter 4, it was possible to link the acceleration of crack development (for those circumstances with a larger creep loading fraction—hold time effect) to the deterioration of microscopically strengthening sub-structures. In other words, the recovered/recrystallized microstructure led to a decrease in the resistance against creep-fatigue crack propagation. A microstructural condition parameter $\Phi$ has been proposed, together with a general degradation function to be combined with the original crack growth models. (A Hall-Petch type relationship was used as the bridge between macroscopic strength and microscopic grain size $d_m$.)

The effectiveness of the adjustment with $\Phi$ will be demonstrated with the SEDF model and the Skelton model (in section 5.3.4). The modified SEDF model (revised by $\Phi$) can make a more accurate prediction than the original SEDF model; and the modified Skelton model (revised by $\Phi$) also gives a closer estimation than the original Skelton model with $d_c$ calculated from the ductility exhaustion method.

By correlating $\Phi$ with friction stress $\sigma_F$ (rather than micro-grain size $d_m$) from the loop analysis (in section 5.3.5), it is possible to roughly estimate the influence of hold time on the short-crack creep-fatigue crack growth rates, without any pre-knowledge of the creep properties of the material, or the information regarding material’s microstructure. Potentially, this method serves as a reasonable compromise for materials with different microstructures.

Finally, a summary of employed short crack growth models with best fit parameters can be found in Appendix A.
5.1 Utilization of crack growth models

In this section, different models are demonstrated one after another with parallel comparison between the two tested materials (FB2 and 10Cr), and these models include various correlating parameters such as the equivalent stress intensity factor $\Delta K_{eq}$, the total cyclic integral $\Delta J$, the plastic cyclic integral $\Delta J_p$, and SEDF. The semi-empirical Tomkins model is also to be examined.

5.1.1 $\frac{da}{dN}$ ($\Delta K_{eq}$) correlation

The validity of the traditional stress intensity factor $K$ is normally restricted only to LEFM conditions, whereas the common testing condition for short-crack creep-fatigue crack growth experiments is mainly in the EPFM regime. One of the proposed modifications to tackle this inconsistency is to use the equivalent stress intensity factor $\Delta K_{eq}$, which also takes plastic loading into account (more details can be found in section 1.4.1 in Chapter 1). The expression of $\Delta K_{eq}$ utilized in this study is defined as:

$$\Delta K_{eq} = (E \Delta \epsilon_p + q_0 \Delta \sigma) Y \sqrt{\pi a} \tag{5-1}$$

For the FB2 testpieces, since the final crack front was always approximately straight, the geometry factor was calculated according to equation (1-23) in Chapter 1. For 10Cr, the crack fronts exhibited a changing part-elliptical shape, which was effectively between chordal crack and semi-circular crack. To stand on the conservative side, the geometry factor for a semi-circular crack was adopted for 10Cr specimens (equation (1-24)).

Since crack closure was not prominent at least before the 2% load drop cycle (or 2mm crack development) for the short-crack creep-fatigue crack growth experiments in this study (section 3.1.2 and 3.2.2), the crack opening fraction $q_o$ was presumed to be unity. Then the values of $\Delta K_{eq}$ were calculated for both materials by assuming the crack front was always open in the entire short crack regime. Afterwards, crack growth rates are plotted as a function of the equivalent stress intensity factor $\Delta K_{eq}$ in Figure 5-1 in the form of:

$$\frac{da}{dN} = A_k (\Delta K_{eq})^{\nu_k} \tag{5-2}$$

Several observations could be made from Figure 5-1a and Figure 5-1b:

1. Most of the curves had a transitional regime at the beginning (as cracking first developed from the starter notch), followed by a roughly linear stable crack growth stage. This resembled the typical $\frac{da}{dN} - \Delta K$ curve with Paris regime in the LEFM loading case. Since the crack depth information was deduced from DCPD values (where no obvious transitional regime was observed, see examples in Figure 3-10 and Figure 3-13), it could be speculated that this initial stage on $\frac{da}{dN} - \Delta K_{eq}$ curves may not be truly resulting from the crack development. They are more likely to be due to a rapid stress redistribution as a
consequence of initial high rates of dynamic recovery. In other words, the correlating parameters (e.g. $\Delta K_{eq}$) do not fully account for this change in deformation characterisation (e.g. in the initial 0.1~0.3mm crack advancement). In practice, the true crack initiation under creep-fatigue-oxidation conditions for the adopted testpieces were invariably complex (e.g. Figure 4-8 and Figure 4-9 in section 4.1.2). Current view holds that this transitional stage is too short to remarkably influence the crack growth modelling, but the effectiveness of crack growth models can be improved once the mechanisms behind this phenomenon are entirely understood.

2. There was a systematic deviation in crack growth rates resulting from the applied total strain range shown by the $da / dN - \Delta K_{eq}$ plots. Curves of lower $\Delta \varepsilon_t$ values were clearly separated from those with larger $\Delta \varepsilon_t$ values. This was presumably due to an underestimation of the plastic part contribution to the total $\Delta K_{eq}$ value (cracks developed much faster when the governing factor was the applied plastic strain range rather than elastic strain range).

3. As shown by the curves consisting of black points, crack growth rates from dwelled specimen were apparently higher than those from comparable tests without hold time. Numerically, tests with a 30min hold time propagated twice as fast as those without hold time. Certainly, this $da / dN - \Delta K_{eq}$ model was not able to account for this effect.

4. The red master curves were used to describe the relationship between crack growth rate and the equivalent stress intensity factor. Values of the fitting exponent were identical for both materials (i.e. $m_k = 1.5$), whereas the fitting constant in 10Cr was larger than that in FB2 ($A_K = 7.6 \times 10^{-6}$ for 10Cr and $A_K = 4.6 \times 10^{-6}$ for FB2, equation (5-2)).

5. The two red dashed lines denoting ±2 times of the master curve acted as the scatter bands. Although most of the points could be contained within the scatter bands, the deviation between curves was still large. This indicated that the equivalent stress intensity factor could hardly serve as a feasible driving parameter to correlate crack growth rate in the current testing conditions.
5.1 Utilization of crack growth models

In Figure 5-1, crack growth rates increase constantly from the beginning. This is consistent with a similar analysis from Skelton, where he plotted $da/dN - \Delta K_{eq}$ relationship for 316 steel and 1CrMoV steel under different testing conditions. He found that $m_k \approx 1.5$ for LCF tests at 550°C including those with 30min dwell. In another study from Holdsworth, $da/dN - \Delta K_{eq}$ relationships (with an alternative $\Delta K_{eq}$ definition) were obtained for 0.5CrMoV steel and 2.25CrMo steel at 538°C and/or 565°C. Crack growth rates also exhibited a transitional regime at the beginning (analogous to Figure 5-1), and curves from longer dwelled experiments were located well above those with shorter dwelled tests.

The best fit values of $A_k$ and $m_k$ in this study are listed in Table 5-1.

<table>
<thead>
<tr>
<th>$da/dN (\Delta K_{eq})$ correlation</th>
<th>$A_k$</th>
<th>$m_k$</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB2</td>
<td>4.6x10^{-6}</td>
<td>1.5</td>
</tr>
<tr>
<td>10Cr</td>
<td>7.6x10^{-6}</td>
<td>1.5</td>
</tr>
</tbody>
</table>

5.1.2 $da/dN (\Delta J_t$ and $\Delta J_p$) correlation

Another facture mechanics based correlating parameter is the cyclic $J$ integral. As already introduced in Chapter 1, the value of $\Delta J_t$ can be obtained by summing up the elastic portion $\Delta J_e$ and the plastic portion $\Delta J_p$. By combining equations (1-18) (1-19) and (1-20), the full form of $\Delta J_t$ could be obtained:

$$\Delta J_t = Y^2 \cdot 2\pi \cdot \frac{(\Delta \sigma)^2}{2E} \cdot a + Y^2 \left[ g(a/b,n) \cdot (n+1) \cdot \left( \frac{1-a/b}{b} \right)^n \right] \frac{\Delta \sigma \Delta \varepsilon_p}{1+n} \cdot a \quad (5-3)$$

where the $g$ function was further calculated according to the literature. Since $g(a/b,n)$ depends also on the strain hardening exponent, the $g(a/b)$ function should be different for the two tested materials (see Table 3-2 in section 3.3.1). The final expression could be written as

$$g(a/b,0.10) = \frac{10.96 + 0.314 \times \left[ 1 - 0.5(a/b) - 0.370(a/b)^2 - 0.044(a/b)^3 \right]^2}{1 + 12.6 \times (a/b)} \quad (5-4)$$

for FB2 and

$$g(a/b,0.06) = \frac{15.06 + 0.182 \times \left[ 1 - 0.5(a/b) - 0.370(a/b)^2 - 0.044(a/b)^3 \right]^2}{1 + 22.74 \times (a/b)} \quad (5-5)$$

for 10Cr.
Finally, the correlation between $\Delta J_i$ and crack growth rate could be illustrated in Figure 5-2a for FB2 and Figure 5-2b for 10Cr (in accordance with equation (5-6)), respectively.

$$\frac{da}{dN} = A_J (\Delta J_i)^m$$  \hspace{1cm} (5-6)

Alternatively, a simpler method to calculate $\Delta J_i$ comes from Dowling,\textsuperscript{24} which actually makes big simplifications on the original calculation scheme from Shih and Hutchinson (see section 1.4.1 for more details). Since Dowling’s solution is for round bar tensile specimens with a semi-circular surface defect, a modification term was employed to scale the geometry factors for FB2 specimens with straight front cracks (i.e. equation (1-22)). For 10Cr, the conservative estimation of $\Delta J_i$ was directly calculated through equation (1-21). The corresponding plots are shown in Figure 5-3.

Comparing Figure 5-3 to Figure 5-2, an advantage associated with the simple calculation method could be observed (as the curves were more consistent with the prediction lines), despite the simplifications/assumptions made by Dowling. Therefore, at least for the current
5.1 Utilization of crack growth models

test conditions, it could be more appropriate to use Dowling’s solution than the original Shih and Hutchinson’s solution.

Generally, in all four graphs most points lay within the scatter bands between the two red dashed lines representing ±2 times the mean values. However, similar to the(da / dN(ΔK<sub>eq</sub>)) correlation, there appeared to be a systematic influence of Δε<sub>c</sub> on each individual curve, which was potentially owing to the inapplicability of directly summing up the elastic and plastic fractions in ΔJ<sub>c</sub> calculation. Instead, a dominant damage concept might be more suitable (i.e. governed by the plastic part of ΔJ<sub>c</sub>). By employing this concept, the elastic contribution to ΔJ<sub>c</sub> could be omitted and the value of ΔJ<sub>p</sub> was provided by the plastic part of equation (5-3). Then the crack growth model could be written as equation (5-7):

\[
\frac{da}{dN} = A_J (ΔJ_p)^{m_J}
\]  

Both full and simple solutions were calculated, but only the simple solution according to Dowling is shown here due to the better consistency. The effectiveness to correlate crack growth rate by ΔJ<sub>p</sub> could then be viewed in Figure 5-4.

![Figure 5-4 Crack growth rates as a function of ΔJ<sub>p</sub> (simple solution according to Dowling) at 600/625°C for (a) FB2 and (b) 10Cr.](image)

By comparing with the previous correlations using ΔK<sub>eq</sub> or ΔJ<sub>c</sub>, an apparent progress has been achieved by using ΔJ<sub>p</sub>: the deviation between low Δε<sub>c</sub> and high Δε<sub>c</sub> could be significantly reduced. This in turn certified the fact that under the current creep-fatigue testing conditions, plasticity (assisted by oxidation) essentially controlled the damaging rate, especially when the proportion of plastic loading was much larger than that of the elastic loading. The best fit values of A<sub>J</sub> and m<sub>J</sub> are listed in Table 5-2.

<table>
<thead>
<tr>
<th>da/dN (ΔJ&lt;sub&gt;p&lt;/sub&gt;) correlation</th>
<th>A&lt;sub&gt;J&lt;/sub&gt;</th>
<th>m&lt;sub&gt;J&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB2</td>
<td>0.09</td>
<td>0.86</td>
</tr>
<tr>
<td>10Cr</td>
<td>0.14</td>
<td>0.86</td>
</tr>
</tbody>
</table>
Despite the better representation (than previous models), there were still two deficiencies in the $da/dN(\Delta J_p)$ correlation (e.g. Figure 5-4). The first problem was the slight inconsistency at lower strain ranges (although by leaving out $\Delta J_p$ the situation has been largely improved). The reason for this was probably the uncertainty associated with the determination of the cyclic strain hardening exponent $n$, when $\Delta \varepsilon_p$ was very small compared with $\Delta \varepsilon_c$ at a lower total strain range. In other words, since $n$ was derived by direct power-law fitting to the experimental stress-strain loops at midlife cycles, its capability of describing cyclic stress-strain behaviour was poorer at low stress/strain ranges, where the deviation from typical power-law hardening was larger.

The second problem was associated with the higher crack growth rates for dwelled specimens. Comparing black points with grey points in Figure 5-4, it is notable that the effect of hold time is still not fully accommodated. This is due to the reason that there is no term in the $\Delta J_p$ formula to take the damage from viscoplasticity into consideration.

### 5.1.3 da/dN (SEDF) correlation

In the previous sections, models based on fracture mechanics have been attempted to rationalize the short crack growth behaviour. A common problem was the insufficiency in comprising the enhanced crack growth rate owing to the accumulated inelastic strain during the hold time. Therefore, an alternative model based on strain energy density factor (SEDF) has been applied to try to overcome this problem (i.e. equation (5-8)). According to the calculation scheme introduced in section 1.4.3, the values of SEDF were computed for both continuously cycled tests and dwelled tests. Then the correlation between SEDF and crack growth rate could be obtained, which is graphically illustrated in Figure 5-5 for FB2 and Figure 5-6 for 10Cr.

$$\frac{da}{dN} = A_w \left( w_d \cdot a \right)^{m_w} = A_w \left( SEDF \right)^{m_w}$$

(5-8)

![Figure 5-5 Crack growth rates for FB2 at 600/625°C expressed as a function of SEDF.](image)
5.1 Utilization of crack growth models

As could be observed, the $da/dN$ (SEDF) correlation (i.e. equation (5-8)) appeared to be similar but marginally more effective to that of $da/dN - \Delta J_p$ (Figure 5-4). This was reflected by two visible improvements on the figures. The first was that the influence of total strain range on individual crack growth curve seemed to be reduced by using the SEDF model (i.e. curves were more parallel aligned). The second was that the curves with hold time were better correlated by the SEDF model, as the black curves were closer and more consistent with the red prediction line (especially represented by Figure 5-5, i.e. the case of FB2). (Strictly speaking, the advantage of this model should be verified with other materials and geometries).

Figure 5-6 Crack growth rates for 10Cr at 600/625°C expressed as a function of SEDF.

However, the scattering of crack growth curves for 10Cr was still unavoidable. As discussed before in section 3.2.1 and 3.2.2, this was most probably due to the inconsistency of crack front development in 10Cr testpieces, which could not be fully represented by a unified relationship. The best fit values of $A_w$ and $m_w$ are listed in Table 5-3.

Table 5-3 Fit values for the constants in $da/dN$ (SEDF) correlation at 600/625°C.

<table>
<thead>
<tr>
<th>da/dN (SEDF) correlation</th>
<th>$A_w$</th>
<th>$m_w$</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB2</td>
<td>0.7</td>
<td>0.86</td>
</tr>
<tr>
<td>10Cr</td>
<td>0.6</td>
<td>0.86</td>
</tr>
</tbody>
</table>

Nevertheless, SEDF could be potentially another parameter to correlate with the crack growth rate. In fact, this model could be viewed as a variation of the $da/dN(\Delta J_p)$ model, with adjustments in two aspects. First, the change of geometry factor and geometry function was included in the original derivation of $\Delta J_p$, but was not included in the SEDF model. However, the SEDF model could give a more consistent prediction, although theoretically the evolution of local crack front geometries associated with the loading intensity should be considered. This was probably the reason why Dowling also assumed a constant value of geometry factor to estimate $\Delta J_p$. By comparing the $da/dN(\Delta J_p)$ model according to Dowling’s solution and the SEDF model, the results were quite close to each other (Figure 5-4 compared to Figure 5-5...
and Figure 5-6). This resemblance was furthermore demonstrated by the fact that values of the regression exponent were identical in both models, i.e. \( m_I = 0.86 \) (equation (5-7)) and \( m_w = 0.86 \) (equation (5-8)).

The second adjustment was the use of cyclic plastic strain energy (the hysteresis energy) instead of monotonic plastic energy (i.e. the area under the ramping-up portion in a stress-strain plot) to characterize the crack growth driving force in a single cycle (see also Figure 1-13 versus Figure 1-15). This was advantageous in two ways: (1) for continuously cycled tests, an overestimation of the strain energy was avoided because upon unloading a small fraction of the energy (i.e. elastic energy) would be retrieved; (2) for tests with hold time, the additional strain energy associated with the expansion of the hysteresis loop could also be included. (In principle, this was an important advantage of this parameter).

It must also be pointed out that the effectiveness of this model has to be confirmed with the results for longer hold time tests (as well as for different geometries). For the materials under investigation, a test with 60min hold period generated higher crack growth rates than the one with 30min hold time. Therefore, it could be expected that further increase of hold time might finally violate the prediction model. The reason behind this was not only the accumulation of inelastic strain or the oxidation-assisted crack tip weakening during the hold period (the relaxation/oxidation behaviour will eventually approach saturation after a very long time), but more importantly, a degradation of microstructure and/or pure creep crack growth in the extreme case. A further modification of this model will be shown in a later section (i.e. section 5.3).

### 5.1.4 The Tomkins model

This widely used semi-empirical model (i.e. equation (5-9)) for fatigue crack propagation under elastic-plastic loading conditions was initially proposed by Tomkins, where he introduced a term called equivalent tensile stress (\( T \)) in order to characterize the critical deformation zone size. As explained in Chapter 1, the value of \( T \) should be in principle between the yield strength and the ultimate strength, which can vary depending on the testing condition. In practice, a reverse deduction of \( T \) is often carried out by the regression through known crack growth rates.

\[
\frac{da}{dN} = \frac{\pi^2 \Delta \sigma^2}{8 (2T)^2} \times a \left[ 1 + \frac{\pi^2 \left( \frac{\Delta \sigma}{2T} \right)^2}{8} \right]
\]  

(5-9)

Attempts to determine the appropriate \( T \) according to the test results have been made, and complications emerged as no unified \( T \) value could be acquired. In other words, although for each individual crack growth curve the best fit \( T \) could be obtained, in general these fit values
5.1 Utilization of crack growth models

deviated considerably from each other. This is shown in Figure 5-7a for FB2 and Figure 5-7b for 10Cr. (The detailed testing conditions can be found in Table 2-3 and Table 2-4).

For both materials, three representative testing conditions with different total strain ranges or dwell periods have been investigated. By plotting crack growth rate against crack depth (denoted by the black points), the influence of $\Delta \epsilon_t$ and hold time was obvious, which was consistent with the previous analysis. The red points came from the best fit Tomkins models, which basically depicted the evolution paths of crack growth rates accurately. However, as shown in the figures as well as in Table 5-4, the employed $\bar{T}$ values were so different.

![Figure 5-7 Examples of crack growth rates as a function of crack depth according to Tomkins model at 600/625°C for (a) FB2 and (b) 10Cr.](image)

The continuously cycled tests with $\Delta \epsilon_t = 0.01$ (FB2-RB01 and 10Cr-RB01) had much larger $\bar{T}$ values than those with a lower $\Delta \epsilon_t$ (FB2-RB05 and 10Cr-RB03). Although a certain amount of deviation was expected (because $\bar{T}$ could be stress/strain range dependent), the magnitude of the present deviation was too big to form any baseline of the characteristic $\bar{T}$ value for the material.

Table 5-4 Values of the (best-fit) equivalent tensile stress in Tomkins model for various samples.

<table>
<thead>
<tr>
<th></th>
<th>FB2</th>
<th>RB01 ($\Delta \epsilon_t = 0.01$)</th>
<th>RB05 ($\Delta \epsilon_t = 0.004$)</th>
<th>RB08 ($t_h = 30\text{ min}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\bar{T}$</td>
<td>520MPa</td>
<td>390MPa</td>
<td>405MPa</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>10Cr</th>
<th>RB01 ($\Delta \epsilon_t = 0.01$)</th>
<th>RB03 ($\Delta \epsilon_t = 0.005$)</th>
<th>RB05 ($t_h = 30\text{ min}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\bar{T}$</td>
<td>590MPa</td>
<td>500MPa</td>
<td>440MPa</td>
<td></td>
</tr>
</tbody>
</table>

Moreover, $\bar{T}$ values for the dwelled samples (FB2-RB08 and 10Cr-RB05) were also significantly smaller than those for the continuously cycled samples. If one regarded this equivalent tensile stress term as a sort of indication of the instantaneous resistance against external plastic deformation (similar to the idea that Tomkins applied), then it was reasonable that $\bar{T}$ values were smaller in dwelled specimens, because more microstructural degradation was associated with the additional hold period.

Likewise, as $\bar{T}$ values in 10Cr were apparently larger than those in FB2, it could be inferred that the internal resistance against shear in 10Cr was superior to that in FB2. In other words,
for the selected temperature conditions the yield strength or ultimate tensile strength of 10Cr would be larger than those of FB2.

Note that the longitudinal coordinate (y-axis) in Figure 5-7b had a smaller span than to that in Figure 5-7a, which inferred that at the same crack depth, the short crack growth rate in FB2 was much larger than that in 10Cr.

5.2 The Skelton model

5.2.1 Empirical fitting of $d_c$

This model has been specifically devised to represent short crack growth behaviour in strain/displacement control, which is also recommended in R5 for component assessment. As illustrated before, the influence of total strain range on creep-fatigue crack growth rates for both steels at 600/625°C was substantial. Examples are shown in Figure 5-8 and Figure 5-9 for a crack depth of 1 mm (the comparisons for specific crack sizes of 0.5 mm and 2.0 mm are similar, since $B$ is a function independent of crack depth), assuming $Q$ value equals unity (in accordance with equation (1-38) in Chapter 1, which is rewritten here, i.e. equation (5-10)).

$$\frac{da}{dN} = Bd^Q \quad (5-10)$$

Data points at different total strain ranges exhibit a log linear representation (red dashed line) parallel to the R5 upper bound line (i.e. the blue dashed line in Figure 5-8 and Figure 5-9), a commonly adopted conservative assessment line based on total surface strain range. (See section 1.4.2 for more details). This upper bound line is almost an order of magnitude higher than that representing the current experiments.
The triangular symbols denote the crack growth rates for tests with hold time, which clearly display larger values than those from continuously cycled tests (circular symbols). However, even for a cycle with 60min hold time, equation (1-39) (the upper bound) provided a very conservative estimate of short-crack creep-fatigue crack growth rates for the tested materials. The average values of $B$ were fitted to be $2.7 \times 10^3 \Delta\varepsilon_t^{2.85}$ for FB2 and $2.4 \times 10^3 \Delta\varepsilon_t^{2.85}$ for 10Cr at both testing temperatures, i.e. 600°C and 625°C.

Previous analysis has indicated that the interaction between creep and fatigue was mainly due to prior creep and strain enhanced oxidation damage effects on the fatigue crack growth component in performed experiments. According to Skelton, one modification of this empirical model is to incorporate this effect by adding a creep damage term $d_c$, such that:

$$\frac{1}{N'} = \frac{1}{N} + d_c \quad (5-11)$$

where $N$ denotes the number of cycles involved in a continuous-cycling test and $N'$ represents the reduced number of cycles in a similar creep-fatigue test with hold time. This was already partly introduced in section 1.4.2.

Based on equation (5-11), two types of derivative can be deduced, namely:

$$dN' = \frac{1}{(1 + Nd_c)^2} dN \quad (5-12)$$

$$dN = \frac{1}{(1 - N'd_c)^2} dN' \quad (5-13)$$

By integrating them into the original model (i.e. equation (5-10)), corresponding crack growth relationships can be written as:
Modelling of short crack propagation

\[
\frac{da}{dN} = \left( \frac{da}{dN} \right)_{\text{total}} = Ba^Q \left( 1 + Nd_c \right)^2
\]  
(5-14)

and

\[
\frac{da}{dN} = \left( \frac{da}{dN} \right)_{\text{total}} = Ba^Q \left( \frac{1}{1 - N'd_c} \right)^2
\]  
(5-15)

This indicates that when using equation (5-14), the number of cycles involved in a continuous-cycling test should be used; whereas when using equation (5-15), the reduced number of cycles (due to creep-fatigue interaction) should be used. The deviation of predicted crack growth rates from these two formulas increases when \( N' \) is further away from \( N \) (i.e. when creep-fatigue interaction becomes more significant).

One example is given in Figure 5-10 for FB2-RB06 (\( t_h = 0.5h \)) by employing equation (5-14), and another example is illustrated in Figure 5-11 for 10Cr-RB10 (\( t_h = 1h \)). In both cases, the exponent \( Q \) was assumed to be unity.

As shown in both figures, the original Skelton model (equation (5-9), assuming \( Q = 1 \)) well represented the crack propagation behaviour observed in the continuously cycled tests (denoted by open circles, where \( d_c = 0 \), \( B = 2.1 \times 10^3 \Delta \varepsilon_i^{2.85} \) for FB2 and \( B = 1.5 \times 10^3 \Delta \varepsilon_i^{2.85} \) for 10Cr). In order to represent the tests with hold time, it was necessary to derive an appropriate \( d_c \) value (by model fitting in this case). For the 30min tension-hold FB2 specimen, \( d_c = 7 \times 10^{-4} \) was found to be the best fit prediction, which yielded a perfect match to the experimental data (Figure 5-10, open triangles). On the other hand, for the 60min tension-hold 10Cr specimen, although \( d_c = 9 \times 10^{-4} \) was already the closest fit to the experimental data, some deviation still existed (Figure 5-11, open triangles).

To reduce any uncertainty associated with assuming \( Q = 1 \), a power law fit was also applied to the 10Cr case and the best fit \( Q \) value was found to be 0.8. Thus, a new value of \( d_c \) could be obtained by employing equation (1-44) and the result is shown in Figure 5-12 (with
5.2 The Skelton model

\( d_c = 1.1 \times 10^{-3} \). Although the effectiveness of prediction (i.e. dashed lines in Figure 5-12) is slightly higher (than that in Figure 5-11), it is still not able to fully describe the experimental crack growth rate.

![Figure 5-11 Example of the modified Skelton model with creep damage factor \( d_c \) for 10Cr-RB10 (at 625°C), assuming Q equals unity.](image)

![Figure 5-12 Example of modified Skelton model with creep damage factor \( d_c \) for 10Cr-RB10 (at 625°C), with \( Q=0.8 \).](image)

Indeed, it could be noticed from the above three figures that the typical evolution path of the prediction lines with \( d_c \neq 0 \) involved a steady incremental shape, which was especially suitable for describing continuously accelerating crack propagation (illustrated by the example of FB2, Figure 5-10). However, if there was an initial transitional regime (where crack initiated, e.g. Figure 5-11 and Figure 5-12), the applicability of the modified Skelton model was reduced. (The possible reason for this kind of transitional regime was explained in section 5.1.1).
5.2.2 Estimation of $d_c$ from ductility exhaustion method

Apart from the empirical way of determining $d_c$ by curve fitting, it is also possible to use the time fraction method or ductility exhaustion method to estimate $d_c$, provided that creep data for the material are available.\(^{59,200}\) (The results from this study inferred that the time fraction method would not be recommended in these circumstances). As introduced in Chapter 1, the expressions for the creep damage term $d_c$ can be written as

$$d_c = \int_0^h \frac{1}{t_g(\sigma, T)} \, dt$$

(5-16)

for the time fraction rule and

$$d_c = \int_0^h \frac{\dot{\varepsilon}_c}{\dot{\varepsilon}_{cf}} \, dt$$

(5-17)

for the ductility exhaustion rule.

One of the tested materials, FB2, was produced within the COST program. It was possible to estimate the creep properties of the tested FB2 steel by making reference to the reported findings on the same class of steel. Therefore, the following section is effectively a demonstration of how the time fraction method and the ductility exhaustion method could be applied to calculate $d_c$, which could then be incorporated into the modified Skelton equation (i.e. equation (5-14)) to account for the influence on crack growth rate from the accumulated creep damage.

For strain controlled creep-fatigue tests, the accumulated creep damage during dwell can usually be evaluated by the stress relaxation behaviour in each cycle. Since the total strain is kept constant, the change of instantaneous equivalent creep strain rate $\dot{\varepsilon}_c$ during the hold time is:\(^{201}\)

$$\dot{\varepsilon}_c = -\frac{1}{E} \frac{d\sigma}{dt}$$

(5-18)

![Figure 5-13 Stress relaxation behaviour at characteristic cycles for FB2-RB06 during the hold time (at 600°C): (a) the evolution of stress value and (b) the evolution of creep strain rate.](image)
To be consistent with the results in Figure 5-10, the test with 30min tensile hold (FB2-RB06) was also selected for this demonstration. The stress relaxation behaviour is illustrated in Figure 5-13. In Figure 5-13a, the evolution of stress values during the hold time at characteristic cycles (in accordance with the definition in section 3.1.2) was exhibited, after a smoothing process. (The corresponding characteristic hysteresis loops are displayed in Figure 3-3f). Due to the rapid load drop at the start of the dwell, it was found that a third order exponential fit could best capture the original trends while suppressing the scattering. Despite the decreasing initial stress values at the start of the dwell, the degree of stress relaxation was almost identical in the four characteristic cycles. This was also confirmed by the plot in Figure 5-13b, where the evolution of the instantaneous equivalent creep strain rate  \( \dot{\varepsilon}_c \) followed a similar path in four characteristic cycles.

The creep properties were sourced from the published work of Gianfrancesco et al., from where creep rupture data for FB2 at 600°C were available. After a careful reproduction, the time to rupture  \( t_R \) at various stress values is illustrated in Figure 5-14a and the relationship between creep strain rate and creep rupture elongation  \( \varepsilon_f \) is shown in Figure 5-14b (this elongation rate was calculated as the average rate until rupture, i.e.  \( \dot{\varepsilon}_c = \varepsilon_f / t_R \), in %/h, although strictly speaking, the relationship should be between creep-rupture ductility and the minimum creep rate).

According to Takahashi’s calculation scheme, the function of  \( t_R \) and  \( \varepsilon_f \) can be approximated by linear regressions through logarithmic values when the strain rate is smaller than a critical value, which is usually larger than 1%/h for 9%~10%Cr steels. (In reality, when plotted with creep strain rate, creep rupture elongation consists of three stages, i.e. upper shelf/lower shelf and the transitional regime between them. Schematic representation of upper/lower shelfs is also included in Figure 5-14b, but not to scale. Because in the current analysis the creep strain rate during relaxation was mainly situated in the transitional regime, a linear fit was presumably capable of representing the  \( \varepsilon_f (\dot{\varepsilon}_c) \) relationship). Therefore, for FB2 steel at 600°C the corresponding relationships (from Figure 5-14) were calculated to be

\[
\log(t_R) = 29.88 - 11.63\log(\sigma) \tag{5-19}
\]

![Figure 5-14 Creep properties for FB2 steel at 600°C: (a) time to rupture and (b) the relationship between creep strain rate and creep rupture elongation (from Ref.202).](image)
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(although this trend line from Ref.202 seems to be optimistically high for FB2 at 600°C) and

\[
\log(\varepsilon_f) = 1.66 + 0.18\log(\dot{\varepsilon}_c) \tag{5-20}
\]

As soon as \( t_k(\sigma,T) \) and \( \varepsilon_f(\dot{\varepsilon}_c) \) were known, the creep damage term \( d_c \) could be calculated according to equation (5-16) (time fraction rule) and equation (5-17) (ductility exhaustion rule). The basic assumption here was that the creep strain accumulation during dwell in a cracked specimen could be simulated by the (average) uniaxial creep rupture behaviour, therefore \( \varepsilon_f(\dot{\varepsilon}_c) \) could be rewritten to \( \varepsilon_f(\dot{\varepsilon}_c) \) in accordance with equation (5-18). The values of \( d_c \) for FB2-RB06 in the four characteristic cycles (Figure 5-13) are listed in Table 5-5. Table 5-5 Creep damage during dwell for FB2-RB06 (with 30min tensile hold at 600°C) at four characteristic cycles by the time fraction method and the ductility exhaustion method.

<table>
<thead>
<tr>
<th>Creep damage for FB2-RB06</th>
<th>Beginning of steady softening</th>
<th>Midlife cycle</th>
<th>End of steady softening</th>
<th>2% load drop</th>
</tr>
</thead>
<tbody>
<tr>
<td>( d_c ) (Time fraction method)</td>
<td>3.9x10^{-4}</td>
<td>2.0x10^{-4}</td>
<td>3.3x10^{-5}</td>
<td>2.1x10^{-5}</td>
</tr>
<tr>
<td>( d_c ) (Ductility exhaustion method)</td>
<td>1.6x10^{-3}</td>
<td>1.8x10^{-3}</td>
<td>1.6x10^{-3}</td>
<td>1.8x10^{-3}</td>
</tr>
</tbody>
</table>

For the time fraction method, \( d_c \) value was reduced significantly as the number of cycles increased. This was primarily due to the decreased absolute stress values at the beginning as well as during the dwell (Figure 5-13a). By comparing with the best fit value \( d_c = 7 \times 10^{-4} \) in Figure 5-10, the creep damage calculated according to the time fraction rule would lead to a substantial underestimation. Therefore for the current test set-up, it was likely that the time fraction method was less appropriate. (Time fraction is typically not used to determine \( d_c \) when creep damage is due to secondary loading).

For the ductility exhaustion method (Table 5-5), \( d_c \) value was almost constant in the four characteristic cycles. This was mainly owing to a consistent evolution path of the instantaneous creep strain rate during the hold time, as demonstrated in Figure 5-13b. More importantly, the calculated \( d_c \) values from the ductility exhaustion method in Table 5-5 were relatively close to the best fit value \( d_c = 7 \times 10^{-4} \) in Figure 5-10 (the representative value at the midlife cycle was \( d_c = 1.8 \times 10^{-3} \)). This potentially indicated that the creep damage term \( d_c \) in crack growth modelling (e.g. in equation (5-14)) could be estimated by this method (even though it was a relatively conservative prediction).

To further ascertain this point, the values of \( d_c \) for two other creep-fatigue crack growth tests (FB2-RB07 with 60min tensile hold and FB2-RB08 with 30min compressive hold) in the four characteristic cycles were also computed and recorded in Table 5-6 and Table 5-7. Similar to
the previous example (Table 5-5), $d_c$ values from the ductility exhaustion method were almost independent of the number of cycles (at least until the 2% load drop cycle). In particular, $d_c$ values for the 30min compressive dwelled experiment ($d_c = 1.8 \times 10^{-3}$, Table 5-7) were analogous to those for the 30min tensile dwelled experiment (Table 5-5); whereas $d_c$ values for the 60min tensile dwelled experiment ($d_c = 2.1 \times 10^{-3}$, Table 5-6) were slightly larger than those for the 30min tensile dwelled experiment (Table 5-5). Moreover, the creep damage up to 30min in the 60min tensile dwelled experiment resembled that in the other two 30min dwelled experiments, which presumably inferred that the stress relaxation behaviour was only material and temperature dependent.

Table 5-6 Creep damage during dwell for FB2-RB07 (with 60min tensile hold at 600°C) at four characteristic cycles by the time fraction method and the ductility exhaustion method.

<table>
<thead>
<tr>
<th>Creep damage for FB2-RB07</th>
<th>Creep damage for FB2-RB07</th>
<th>Creep damage for FB2-RB07</th>
<th>Creep damage for FB2-RB07</th>
<th>Creep damage for FB2-RB07</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Beginning of steady</td>
<td>Midlife cycle</td>
<td>End of steady</td>
<td>2% load drop</td>
</tr>
<tr>
<td></td>
<td>softening</td>
<td></td>
<td>softening</td>
<td></td>
</tr>
<tr>
<td>$d_c$ (Time fraction</td>
<td>3.9x10^{-4}</td>
<td>2.1x10^{-4}</td>
<td>3.9x10^{-5}</td>
<td>1.6x10^{-5}</td>
</tr>
<tr>
<td>method)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$d_c$ (Ductility exhaustion</td>
<td>2.0x10^{-3}</td>
<td>2.1x10^{-3}</td>
<td>2.0x10^{-3}</td>
<td>2.2x10^{-3}</td>
</tr>
<tr>
<td>method)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$d_c$ up to 30min</td>
<td>1.8x10^{-3}</td>
<td>1.9x10^{-3}</td>
<td>1.8x10^{-3}</td>
<td>1.9x10^{-3}</td>
</tr>
</tbody>
</table>

Table 5-7 Creep damage during dwell for FB2-RB08 (with 30min compressive hold at 600°C) at four characteristic cycles by the time fraction method and the ductility exhaustion method.

<table>
<thead>
<tr>
<th>Creep damage for FB2-RB08</th>
<th>Creep damage for FB2-RB08</th>
<th>Creep damage for FB2-RB08</th>
<th>Creep damage for FB2-RB08</th>
<th>Creep damage for FB2-RB08</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Beginning of steady</td>
<td>Midlife cycle</td>
<td>End of steady</td>
<td>2% load drop</td>
</tr>
<tr>
<td></td>
<td>softening</td>
<td></td>
<td>softening</td>
<td></td>
</tr>
<tr>
<td>$d_c$ (Time fraction</td>
<td>5.5x10^{-4}</td>
<td>2.9x10^{-4}</td>
<td>6.5x10^{-5}</td>
<td>3.1x10^{-5}</td>
</tr>
<tr>
<td>method)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$d_c$ (Ductility exhaustion</td>
<td>1.8x10^{-3}</td>
<td>1.8x10^{-3}</td>
<td>1.8x10^{-3}</td>
<td>1.7x10^{-3}</td>
</tr>
<tr>
<td>method)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

To visualize the magnitude of $d_c$ from two methods, values of $d_c$ at characteristic cycles for these three dwelled tests (data from Table 5-5, Table 5-6 and Table 5-7) are plotted in Figure 5-15. Time fraction predictions are represented by gradient fill, while ductility exhaustion predictions are represented by solid fill. From characteristic point 1 to point 4, instantaneous (nominal) stress values during the hold time became smaller, leading to a larger $t_R$ according to equation (5-19). Therefore for the creep damage $d_c$ in each cycle, the integration in the same time scale with a larger $t_R$ would induce a smaller value of $d_c$ according to equation (5-16). This is the main reason for the decreasing value of $d_c$ in Figure 5-15 with the time fraction method (and the larger $d_c$ in 30min compressive test is also simply due to a larger absolute stress value during dwell). Note that Figure 5-15 does not necessarily represent...
actual creep damage generated in respective cycles, but only serves as an indication of calculated $d_c$ values according to time fraction and ductility exhaustion methods.

![Figure 5-15 Values of $d_c$ at characteristic cycles for three dwelled tests at 600°C (data from Table 5-5 Table 5-6 and Table 5-7). Time fraction method is represented by gradient fill, while ductility exhaustion method is represented by solid fill.](image)

The values of $d_c$ at midlife cycles from the ductility exhaustion method were further integrated into the crack depth-crack growth rate relationship as expressed in equation (5-14). (Certainly it was also possible to use equation (5-15), provided that the reduced number of cycles $N'$ was employed accordingly. In case the original number of cycles $N$ from continuously cycled condition was utilized, equation (5-15) would lead to a larger estimation than equation (5-14)).

Finally, the experimental and predicted crack growth rates are plotted in Figure 5-16, for FB2-RB07 and FB2-RB08. The hollow circular symbols represented the continuous cycling case, based on which the dashed prediction curves were derived.

![Figure 5-16 Example of modified Skelton model with creep damage factor $d_c$ estimated by the ductility exhaustion method.](image)
It could be seen that although a certain amount of overestimation existed, the predicted crack growth rates seemed to act as reasonable upper bounds for the experimental values (denoted by red/blue hollow symbols). The upper bound in R5 was also plotted in this figure, which was still far away from the experimental results. Nevertheless, it could be concluded that by employing this modified Skelton model with \( d_c \) deduced from the ductility exhaustion method, a realistic preliminary estimation of the crack growth behaviour could be obtained. (As shown later in section 5.3.4, a more appropriate correlating parameter \( \Phi \) is introduced to account for creep-fatigue interaction for the adopted materials).

### 5.2.3 Prediction of crack propagation with longer hold periods

Since a much longer steady running period normally occurs during turbine operation, the validity of this model should be examined with respect to an extended hold time. From the above analysis it was found that the creep strain rate during the stress relaxation period for FB2 at 600°C was relatively insensitive to the initial stress value (see Figure 5-13b) as well as to the position of dwell. Hence, it might be legitimate to extrapolate the short time stress relaxation behaviour into a long time regime.

By assuming self-similar stress relaxation behaviour, the exemplary evolution of \( \dot{\varepsilon}_c \) during the hold time (assuming the nominal elastic modulus \( E = 140 \text{GPa} \)) could be described by a mathematical fitting of:

\[
\dot{\varepsilon}_c = \frac{29t^{-0.92}}{(52t^{0.079} + 9.5)^2}
\] (5-21)

The effectiveness of this fit could be seen in Figure 5-13b by the purple dashed line, which equitably depicted all the experimental curves. (Certainly long time tests can violate this trend line). By integrating equation (5-20) and equation (5-21) into equation (5-17), the expression for \( d_c \) could be rewritten as:

\[
d_c = \int_0^{t_h} \frac{0.346t^{-0.7544}}{(52t^{0.079} + 9.5)^{1.64}} dt
\] (5-22)

A direct integral for this equation was not available. Therefore numerical approximation (with a sufficiently small \( dt \) as the step size) was carried out, for the hold periods of 10h, 100h and 1000h. To minimize the error, the values of \( d_c \) were calculated by adding the extrapolated data to the already obtained experimental data (e.g. in the case of \( t_h = 100h \), \( d_c \) was the summation of \( 2.1 \times 10^{-3} \) for the 1st hour, and the extrapolated values from the 1st~100th hour). Ultimately, the values were tabulated in Table 5-8, as well as displayed in Figure 5-17.
Table 5-8 Estimated creep damage during dwell for FB2 at 600°C from extrapolated data according to the ductility exhaustion method.

<table>
<thead>
<tr>
<th>Creep damage for FB2 at 600°C</th>
<th>$t_h = 10h$</th>
<th>$t_h = 100h$</th>
<th>$t_h = 1000h$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d_c$ (Predicted from the Ductility exhaustion method)</td>
<td>$3.2 \times 10^{-3}$</td>
<td>$4.0 \times 10^{-3}$</td>
<td>$8.1 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

Figure 5-17 Prediction of crack growth rates for different hold periods from the modified Skelton model with creep damage factor $d_c$ estimated by the ductility exhaustion method.

The open circles ($t_h = 0h$) symbolized the known experimental results (the base line), whereas the four black curves were computed from the modified Skelton model (equation (5-14)) together with the ductility exhaustion method (equation (5-22)). It could be seen that an increase of the predicted crack growth rates occurred when the hold time was larger, as expected. The upper bound in R5 (i.e. equation (1-39)) was also displayed as the red dashed line for comparison. While this line is located well beyond the predicted curve with $t_h = 100h$, it cannot fully contain the circumstance with $t_h = 1000h$.

The prediction in Figure 5-17 was only an indicative analysis, where the three main uncertainties were: (1) the consistency between the referenced creep properties and the true creep properties for the tested material (the referenced values gave a rather conservative estimation, see Figure 5-16); (2) the validity of using short term relaxation behaviour to simulate long term relaxation behaviour (range of legitimacy in equation (5-21)); and (3) the form of ductility exhaustion model, which should have also included the upper/lower bounds of creep strain rate. Once these uncertainties were ruled out or minimized, the accuracy of this kind of prediction could be considerably improved.
5.3 Discussion

5.3.1 Comparison of short crack growth models

Over all, three types of short-crack creep-fatigue crack growth model have been studied for two advanced turbine steels at the operating temperatures. These included the fracture mechanics based models (correlated with $\Delta K_{eq}$, $\Delta J_t$ or $\Delta J_p$), semi-empirical models (the Tomkins model and the Skelton model), and the model based on SEDF. Generally speaking, all of those models could in principle more or less predict the short-crack creep-fatigue crack growth behaviour. But more specifically, the effectiveness could vary primarily due to the influence from either different total strain ranges or hold periods.

For the $\Delta K_{eq}$ model and the $\Delta J_t$ model, a big deviation between the curves with a lower $\Delta \varepsilon_t$ and a higher $\Delta \varepsilon_t$ could apparently be observed (Figure 5-1 and Figure 5-2). This was probably due to an underestimation of the plastic loading fraction (or an overestimation of the elastic loading fraction). Different from the classical pure fatigue case where only a small region ahead of the crack tip was under plastic loading, the whole specimen cross-section underwent plastic deformation for all the strain ranges tested in the current experimental campaign. In case of a high $\Delta \varepsilon_t$ value, the intensified local stress value at the crack tip had a large chance to easily exceed the critical shear stress. Rather than persistently building-up slip bands, the crack tip could be directly torn open under the assistance of oxidation (similar to the concept described in section 1.3.4). This effect might significantly reduce the weight of influence from the elastic loading, and an oxidation assisted plastic shear was in fact the dominant mechanism. Therefore, it seemed to be appropriate to take out the elastic loading portion in the short crack growth modelling. This is exactly what the $\Delta J_p$ model is based on, and the effectiveness of this parameter is clearly higher (Figure 5-4 versus Figure 5-3).

For the two semi-empirical models, one of the advantages compared with the fracture mechanics based models is the relatively simple expression (i.e. equation (5-9) and equation (5-10)). The analysis in section 5.1.4 has shown that originally as a fatigue crack growth model, the Tomkins model cannot be directly applied to creep-fatigue deformation condition. (The characteristic value in the Tomkins model is $T$, which is mainly dependent on the material’s tensile/ultimate strength and the applied stress range). With the best-fit $T$ values, this model could almost describe the short crack growth behaviour for every exemplary experiment (Figure 5-7). However, the major drawback was that no universal $T$ value could be found to be compatible with all the testing conditions. In other words, the actual value of $T$ seemed to be test dependent. It was certainly possible to obtain the optimal $T$ value through regression on the known crack growth curves, but it was difficult to find the right $T$ to predict the crack growth behaviour under other testing conditions. This model is therefore not promoted for the prediction of short crack development under creep-fatigue loading conditions. (One idea to mitigate this problem could be the application of upper/lower bounds, i.e. usage of the known $T$ values to estimate the limit situation in other test conditions).

On the other hand, the (modified) Skelton model (i.e. equation (5-14)) obviously had more physical significance as shown in section 5.2. The varying parameters in this model are only
the instantaneous crack depth and the strain range. It is even not necessary to consider the geometry factor associated with crack fronts. In spite of this, this model could fairly predict the short crack growth behaviour accurately, provided that the right characteristic value \( d_c \) (which is dependent on the material’s creep resistance in terms of ductility and time) was used.

Surely the value of \( d_c \) could be empirically fitted, but more importantly it could also be derived from the creep properties of the material (even though in this study only indicative creep properties were referred to). By comparing the ductility exhaustion method with the time fraction method, it was found that the former one could generate a more appropriately derived \( d_c \) value, which was much closer to the empirically fit \( d_c \) value (e.g. Figure 5-10 compared to Figure 5-16). (This observation supports what was already known for strain controlled loading).

It was then possible to estimate the creep damage during the hold periods according to the ductility exhaustion method, once the correct material’s creep properties were utilized. Therefore, crack growth rates under various conditions could then be predicted, and the effectiveness was schematically shown in Figure 5-17.

Alternatively, instead of focusing on each individual case, a boundary value has been proposed to give a conservative estimation of the overall crack growth behaviour under LCF conditions for various materials according to the Skelton model. This upper bound was found to be valid for all of the experiments in this study (the average short crack growth rate in performed experiments was roughly an order of magnitude smaller than that predicted by the upper bound line, Figure 5-8 and Figure 5-9), although according to the preliminary prediction a 1000h hold period might finally exceed this upper bound line (Figure 5-17).

However, one small complication in this model was that due to the formation of the mathematical expression, this model was not flexible enough to precisely describe a relatively complex crack propagation route (in the case of a transitional regime at the beginning of crack development, Figure 5-11 and Figure 5-12). As discussed before, this transitional regime could most probably be due to the resistivity change from stress/strain redistribution and the true fatigue crack initiation phase. However, this regime is short (e.g. in Figure 5-16, hollow symbols), and the current focus is crack development rather than crack initiation. Despite this complication, the semi-empirical model maintains a relatively simple formation and requires few input parameters, which can be extremely suitable for practical application in real components.

The model based on the strain energy density factor (SEDF) integrates features from the fracture mechanics based models and the semi-empirical models. The similarity between the SEDF model and the \( \Delta J_p \) model has been elucidated previously (section 5.1.3), with the conclusion that the SEDF model performed marginally better in short crack growth characterizations influenced by hold time. For the continuously cycled experiments, these two models generated very similar predictions. The resemblance between the SEDF model and the Skelton model is the consideration for tests with hold time: the additional contribution from \( d_c \) in the (modified) Skelton model can at least be partly accounted for by the effect of the increased \( w_d \) term in the SEDF model.
To sum up, while it is acknowledged that the effectiveness of the correlating parameters when applied to other geometries has still to be verified, the evidence indicates an advantage associated with use of the strain energy density factor (SEDF) and the modified Skelton model (with $d_c$ calculated from the ductility exhaustion method) relative to other correlating parameters/models. In spite of a relatively complex calculation process, the main obstacle for practical application of the SEDF model by industry is the requirement for a full record of cyclic stress/strain history. On the other hand, an exact determination of the crack growth rates from the modified Skelton model relies on the prior knowledge of creep properties of the material at the same temperatures, which is not a problem in practice.

### 5.3.2 Comparison of short crack growth rates for FB2 and 10Cr

FB2 and 10Cr specimens were tested under similar experimental conditions, which provided the premise for further comparison of short-crack creep-fatigue crack development. In sections 3.1.3 and 3.2.2, it was shown that FB2 testpieces generally reached 2% load drop point earlier than 10Cr specimens, and the corresponding crack growth rates at characteristic points were larger than those from 10Cr. It could almost be inferred that 10Cr has a higher resistance to crack development for the adopted experimental conditions in this study.

However, in section 5.1 where crack growth rates were plotted as a function of various correlating parameters (i.e. $\Delta K_{eq}$, $\Delta J_{r}$, $\Delta J_{p}$ and SEDF), there seemed to be no systematic advantage of 10Cr steel over FB2 steel. Figure 5-1 to Figure 5-4 displayed higher crack growth rates in 10Cr (as a function of $\Delta K_{eq}$, $\Delta J_{r}$, $\Delta J_{p}$), whereas Figure 5-5 and Figure 5-6 demonstrated higher crack growth rates in FB2 (as a function of SEDF).

In order to figure out the reason for this discrepancy, a short discussion is made in this part by a parallel comparison between two testpieces, i.e. FB2-RB06 (HT1138) and 10Cr-RB04 (HT1355). They were both tested with $\Delta e_r = 1\%$, $\dot{\varepsilon} = 0.1\%$, and 30min tension hold at 600°C. Six figures were plotted in Figure 5-18, with red (and blue) symbols denoting 10Cr and black symbols denoting FB2.

Figure 5-18a shows the number of cycles to 2mm crack depth. It is clear that in the first half part of cycles these two specimens had similar crack growth behaviour, and in the second half part of cycles the 10Cr testpiece took more cycles to reach the same crack depth of 2mm.

This is consistent with Figure 5-18b, where the crack growth rates at a smaller crack depth were comparable for both specimens. After crack depth reaching 0.7mm, the FB2 specimen propagated increasingly faster than the 10Cr specimen. (Crack depth is not fully proportional to the cycle number, because it takes more cycles for a crack to propagate a certain distance when the crack growth rate is lower).

As mentioned in section 3.2.1, a distinctive difference between FB2 specimens and 10Cr specimens was the shape of crack fronts. For FB2 specimens, the developing crack fronts were almost always straight, whereas for 10Cr specimens, crack fronts developed from the
Modelling of short crack propagation

chordal crack starter through a varying transitional stage to a part-elliptical shape. To maximize the accuracy in accounting for this changing profile of crack fronts in 10Cr testpieces, a two-stage DCPD to crack depth calibration process was applied (section 3.2.1). Because this part-elliptical profile evolved halfway between straight and semi-circular profile (and the degree of curvature was testpiece-dependent), two geometry factors, i.e. $Y_{cc}$ (for edge chordal cracks) and $Y_{scc}$ (for edge semi-circular cracks) can be considered as two boundaries of the true geometry function.

![Figure 5-18](image)

Figure 5-18 Comparison of crack depths/growth rates for FB2-RB06 and 10Cr-RB04; (a) $a-N$; (b) $da/dN-a$; (c) $da/dN-\Delta K_{eq}$; (d) $da/dN-\Delta J$; full calculation; (e) $da/dN-\Delta J$; simple calculation; and (f) $da/dN$-SEDF
As a conservative estimation, in section 5.1, all the calculation involving geometry factors for 10Cr was carried out using \( Y_{sc} \) (which was invariably smaller than \( Y_{cc} \) at the same crack depth). In this sense, the crack growth correlating parameters calculated with \( Y_{sc} \) were supposed to be smaller than those calculated by adopting \( Y_{cc} \). This can be illustrated by Figure 5-18c, Figure 5-18d and Figure 5-18e. In these three figures, the red symbols represent values of 10Cr calculated with \( Y_{sc} \), which are the same as in Figure 5-1b, Figure 5-2b and Figure 5-3b (but the coordinates here are in linear-scale instead of log-scale). Apparently, the red lines are located above the black lines, which may be interpreted as that the 10Cr specimen has higher crack growth rates than the FB2 specimen at the same values of \( \Delta K_{eq} \) or \( \Delta J \).

Further examination has confirmed that this is mainly due to crack front profile, i.e. the geometry factor. If the geometry factor for chordal cracks (\( Y_{cc} \)) is applied rather than the one for semi-circular cracks (\( Y_{sc} \)), a horizontal shift of the curve will appear, as displayed in Figure 5-18c to Figure 5-18e (i.e. red curve position to blue curve position). In this case, the FB2 specimen seems to have higher crack growth rates than the 10Cr specimen. In reality, the true crack growth curve for the 10Cr specimen should situate between these two curves (evolving from the blue curve to the red curve).

In the SEDF model, the geometry factor is not explicitly expressed. As exhibited in Figure 5-18f, the FB2 specimen indeed shows a faster crack propagation than the 10Cr specimen. Referring back to the construction of SEDF (i.e. equation (1-48) and (1-50)), the cyclic stress-strain response as well as instantaneous crack depth play a decisive role.

Therefore, when comparing crack growth rates for these two materials, the precondition should also be mentioned. In principle, at the same cycle number, or crack depth, or SEDF value, FB2 specimens propagates faster than 10Cr specimens. At the same \( \Delta K_{eq} \) or \( \Delta J \), 10Cr specimens can have higher crack growth rates than FB2 specimens.

In fact, FB2 steel was supposed to be more resistant to creep-fatigue loading, with a designed operating temperature of 625°C, whereas the operating temperature for 10Cr steel is 600°C. (In general, the creep property (ductility, time to rupture, etc.) of FB2 is better than that of 10Cr; and absolute strength (e.g. yield strength, ultimate strength) of FB2 is similar to that of 10Cr, see Appendix B).

However, the experiments in this study seemed to suggest that there is no obvious advantage of FB2 steel (in some cases 10Cr steel even has a better performance). The reason for this anomaly may come from the following points:

(1) The cyclic strain hardening behaviour is different for these two materials. As illustrated in section 3.3.1, 10Cr has a much lower \( n \) than FB2. This potentially infers that 10Cr is relatively more resistant to plasticity-dominated fatigue, while FB2 is relatively more resistant to elasticity-dominated fatigue. When the total strain range is higher, i.e. \( \Delta \varepsilon_{t} = 1\% \) (as in all dwelled experiments), the testpieces underwent gross plastic deformation. It was likely that 10Cr had an overall better performance than FB2. In fact, at a lower total strain range, i.e. \( \Delta \varepsilon_{t} = 0.5\% \) where the testpieces experienced mainly elastic deformation, the difference in \( N_{2\%} \) was not as much as at \( \Delta \varepsilon_{t} = 1\% \) (by comparing Figure 3-5 and Figure 3-6).
(2) As demonstrated in section 4.1, oxidation may have played an important role in crack initiation and propagation under creep-fatigue loading conditions. While the highest creep resistance is achieved with 9% Chromium, the resistance to oxidation can be slightly improved by an additional Chromium content, i.e. 10% Chromium. In other words, 10Cr steel has a better performance against oxidation than FB2 steel.

(3) Microstructural analysis has shown that for the investigated FB2 steel, there are some large second-phase particles in the matrix (see section 4.1.3). Early micro-crack linkage near these particles could lead to faster crack propagation (e.g. Figure 4-11). (In fact, the reason for this quantity of second-phase particles can be as simple as that the raw material/block was cut from a part where inhomogeneous segregation took place in the manufacturing process). It is advised to use different batches of materials to verify the true difference of creep-fatigue resistance in these two steels if necessary.

5.3.3 Enhanced crack development

In practice, the total (creep-fatigue) crack growth behaviour may not simply be the sum of fatigue crack growth during ramp loading and creep crack growth rates during hold time. Therefore, an additional enhancement term should be applied to the basic fatigue crack growth part to account for the interaction, and a common fundamental crack growth model could be in the form of:

\[
\frac{da}{dN}_{\text{total}} = (\text{enhancement term}) \left( \frac{da}{dN} \right)_{\text{fatigue}} + (\frac{da}{dN})_{\text{creep}}
\]

As an example, one proposed model considers the enhancement to be achieved through the incorporation of a generic time dependent damage function,\(^{11}\) such that

\[
\frac{da}{dN}_{\text{total}} = \left( \beta_0 + (\beta_{\text{oxide}} + \beta_{\text{creep}})t_h \right) (\Delta K_{\text{eff}})^m_C + \int_0^{t_h} A_C (C^*)^{m_C} \, dt
\]

Where \(\beta_0\), \(\beta_{\text{oxide}}\), and \(\beta_{\text{creep}}\) are constants denoting the effect of time-independent damage, time dependent oxidation damage, and time dependent creep damage; \(A_C\) and \(m_C\) are constants in the creep crack growth rate equation.

In the analysis on short-crack creep-fatigue crack growth behaviour, the adoption of such a kind of function (i.e. equations (5-23),(5-24)) was also feasible, although the evidence from this study has shown that this might not be absolutely necessary in the case of the SEDF model (i.e. Figure 5-5 and Figure 5-6) or the modified Skelton model (i.e. Figure 5-10, Figure 5-11 and Figure 5-12) for these materials at these temperatures under the applied test conditions.

As far as the tests in this study are concerned, post-test microstructure inspection has revealed that even with a 60 minute hold time, the crack propagation route exhibited an almost fully
transgranular profile (e.g. Figure 4-7). (Technically, very small cavities could form on packet/lath boundaries in these materials. Growth down these creep-damaged boundaries would have the appearance of being transgranular). In addition, there seemed to be only negligible cavities or micro-cracks at triple junctions or prior-austenite grain boundaries in the bulk material for the fully reversed loading condition (excluding these micro-cracks induced by large second-phase particles, i.e. Figure 4-11). Moreover, the DCPD results showed that the voltage variation during hold periods was much smaller than that between respective cycles (Figure 3-19 and Figure 3-20). Therefore, it is justified to take the individual creep crack growth term out of equation (5-23), and the creep-fatigue interaction is essentially related to the enhanced crack growth rate during fatigue/cycling, i.e.

\[
\left( \frac{da}{dN} \right)_{\text{total}} = \left( \frac{da}{dN} \right)_{\text{fatigue}} \cdot \left( \text{enhancement term} \right)
\]  

(5-25)

For example, in the Skelton model, a similar expression is equation (5-14), with the damage term \( d_e \) to adjust for the creep contribution for short crack development.

As a matter of fact, instead of cavity formation or micro-cracking, another type of microstructural 'degradation' occurred in short-crack creep-fatigue crack growth tests on the two advanced martensitic steels, which decreased the material's resistance to crack propagation.

In Chapter 4 (sections 4.2.3 and 4.2.4), much has been discussed concerning the microstructural evolution during creep-fatigue deformation. It was found that initially tempered martensitic microstructures have developed into more equiaxed and coarsened sub-structures (owing to recovery/recrystallization), especially in the near crack regions. Therefore, it was speculated that the resistance of the material to further creep-fatigue deformation would actually decrease, along with sub-structure evolution. In other words, this microstructural degradation could enhance the crack growth behaviour, and the magnitude of this enhancement was associated with the degree of creep/recovery (which changed the crystallographic structures extensively). This was also a type of creep-fatigue interaction.

In all of the above mentioned models in sections 5.1 and 5.2, only typical mechanical parameters were considered, assuming the microstructure stayed the same. But with the information that the material did degrade during deformation (i.e. Figure 4-26 and Figure 4-27), it is more reasonable to also include a microstructural condition parameter to account for any loss in the resistance/deformation condition of the material.

### 5.3.4 Improvement by incorporating microstructural condition parameter

As introduced previously in section 1.5.2, three most important strengthening mechanisms for the targeted type of steel are sub-boundary strengthening, dislocation strengthening and precipitation strengthening. These three mechanisms don't just work independently, but interact and evolve with each other. This potentially means that even if the characteristics in
only one of the mechanisms are acquired, it can probably still be possible to estimate the status of the material to some extent.

It was shown that with the same total strain range, the major discrepancy of the employed crack growth models existed between continuously cycled and dwelled experiments (section 5.1 and 5.2). Accordingly, a significant difference in sub-structures between these two types of testpieces was also witnessed, as shown in Chapter 4 (e.g. in Figure 4-24). Hence, it was reasonable to come to the idea of integrating a microstructural condition parameter into the crack growth models. Consistent with equation (5-25), one way to represent this creep-fatigue interaction was to incorporate a general degradation function by combining it with the original crack growth model, i.e.

\[
(da/dN)_{\text{total}} = \left( \frac{da}{dN} \right) \cdot \left( \frac{1}{1 - \Phi^\beta} \right) ^\alpha
\]

(5-26)

where \( \Phi \) is a microstructural condition parameter; \( \alpha \) and \( \beta \) are damage coefficients.

For creep resistant martensitic steels, long term stability is believed to be directly linked to sub-boundaries (e.g. Refs. 134, 145, 148, 196). This means that the sub-structure dimension can potentially be an index of the microstructural condition. (For pure creep tests, subgrain development for these steels normally occurs in the tertiary regime, whereas in this study, creep-fatigue loading seemed to promote this development to a much earlier phase). In fact, that was exactly the reason why such an extensive test matrix of EBSD measurement on micro-grain size has been carried out in this study. With the knowledge of evolutionary micro-grain sizes at hand, it was then essential to tie this index to the damage function. As discussed before, the cracking mechanism in current short-crack creep-fatigue crack growth tests was closely associated with the plastic loading, i.e. the obstacle for the crack tip to extend was dependent on the material’s resistance against plastic deformation. Then the classical Hall-Petch type relationship seemed to be a convenient tool to describe this effect: 203, 204

\[
\sigma_y = \sigma_0 + \sigma_p + \sigma_s + \sigma_p + k_{HP} d_m^{-1/2}
\]

(5-27)

Where \( \sigma_y \) is the yield strength; \( \sigma_0 \) is the material’s inherent friction strength; \( \sigma_p, \sigma_s, \sigma_p \) denotes the contribution from precipitation hardening, solid solution hardening and dislocation hardening, respectively; \( k_{HP} \) is the Hall-Petch coefficient; and \( d_m \) is the average grain diameter.

For simplicity, the strengthening from precipitates, solid solution and dislocations under creep-fatigue conditions might be assumed to be correlated with the grain boundary strengthening, which led to a simpler equation:

\[
\sigma_y = \sigma_0 + k_{HP} d_m^{-1/2}
\]

(5-28)

with a modified Hall-Petch coefficient \( k_{HP} \) to include all the strengthening mechanisms. Certainly this was a massive postulation because in reality things were much more complex. But as a starting point, equation (5-28) was at least capable of capturing the material’s change
through micro-grain size. Therefore, the expression for the damage parameter $\Phi$ could be defined (which reflected decrease of the material’s resistance against plastic deformation), in the form of:

$$\Phi = \frac{\sigma_{y \_start} - \sigma_y}{\sigma_{y \_start}} \quad (5-29)$$

where $\sigma_{y \_start}$ and $\sigma_y$ are the material’s yield strength at the start of the test and at any time/cycle during the test. Because $\sigma_{y \_start} \geq \sigma_y$, the damage parameter should be always within the range of $1 > \Phi \geq 0$. By combining equation (5-28) with equation (5-29), equation (5-30) could be obtained:

$$\Phi = \frac{k_{HP}}{\sigma_{y \_start}} \left[ (d_{m \_start})^{0.5} - (d_m)^{0.5} \right] \quad (5-30)$$

Here $d_{m \_start}$ and $d_m$ are the initial and instantaneous micro-grain diameters. Note that in this equation the real variable is $d_m$, which is effectively an index depending on the stress/strain state, crack depth, temperature and hold time. The simplified formulation of $d_m$ has been discussed in Chapter 4 (section 4.2.4), in the form of

$$d_m = d_{m \_start} (1 + b_3a + b_2a^{\frac{2}{3}}t_6^{\frac{1}{3}}) \quad (5-31)$$

and the best fit parameters were obtained for the tested materials. By combining equation (5-31) with equation (5-30), the expression for $\Phi$ could be rewritten as:

$$\Phi = \Phi_0 \left[ 1 - \frac{1}{\sqrt{1 + b_3a + b_2a^{\frac{2}{3}}t_6^{\frac{1}{3}}}} \right] \quad (5-32)$$

where the constant $\Phi_0$ is:

$$\Phi_0 = \frac{k_{HP}}{\sigma_{y \_start} \cdot \sqrt{d_{m \_start}}} \quad (5-33)$$

It has been reported that the constant and coefficient in the original Hall-Petch equation (i.e. (5-27)) are dependent not only on material composition and temperature, but also on the variation of grain size itself. This problem could get even more complicated when taking other strengthening mechanisms into account (i.e. the modified Hall-Petch coefficient $k_{HP}$, equation (5-28)). Therefore, it was difficult to determine the precise values of $\sigma_0$ and $k_{HP}$ in each testing condition for the materials in this study. In view of those in the literature for low carbon steels, a reasonable range of the values for the Hall-Petch parameters in the current analysis is listed in Table 5-9 (by assuming the initial yield strength $\sigma_{y \_start} = 300MPa$ and the initial micro-grain size $d_{m \_start} = 0.8\mu m$).
Table 5-9 Possible range of the values for the Hall-Petch parameters in this analysis.

<table>
<thead>
<tr>
<th>$\sigma_0$ (MPa)</th>
<th>40</th>
<th>70</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k_{HP}'$ (MPa/μm$^{0.5}$)</td>
<td>233</td>
<td>206</td>
<td>179</td>
</tr>
<tr>
<td>$\Phi_0$ (-)</td>
<td>0.87</td>
<td>0.77</td>
<td>0.67</td>
</tr>
</tbody>
</table>

However, as far as the calculation of the damage parameter $\Phi$ was concerned, computing the absolute values of $k_{HP}'$ (i.e. $\Phi_0$) might not be entirely necessary, because the constant $\Phi_0$ would nevertheless be scaled up/down through the exponent $\beta$ in a later stage (equation (5-26)). Therefore, in the following discussion, the intermediate values ($\sigma_0 = 70$MPa, $k_{HP}' = 206$MPa/μm$^{0.5}$) were adopted as a first attempt.

In principle, this microstructural condition parameter $\Phi$ could be applied to most of the previously discussed short crack growth models (e.g. correlated with $\Delta K_{eq}$, $\Delta J$, or $\Delta J_P$, SEDF or the original Skelton model). As an example, its applicability was demonstrated first with respect to the SEDF model, and then the (original) Skelton model. Afterwards, a possible approach to link $\Phi$ to the change of friction stress $\sigma_F$ (see definition in section 3.3.3) is also presented in the next section.

**Modified SEDF model with $\phi$**

As demonstrated in section 5.1.3, the SEDF model could already almost describe short-crack creep-fatigue crack growth behaviour for the adopted materials. It is based on the area within the hysteresis loops, which in principle indicates the energy provided to the crack tip. However, it is not known that how this energy is distributed to various thermally activated process (as well as heat dissipation into the surroundings). In this sense, the microstructural condition parameter $\Phi$ gives an indication of sub-structures status in each cycle, which may improve the accuracy of crack growth modelling. (Strictly speaking, a faction of energy might have been double-counted in this process, but results in this part of study implies that even with this existing uncertainty, the advantage of this modified SEDF model with $\Phi$ is still big).

In terms of the SEDF model, equation (5-26) could then be rewritten as:

$$\left(\frac{da}{dN}\right)_{total} = A_w (\text{SEDF})^{m_w} \cdot \left(1 - \Phi^\beta\right)^\alpha$$

(5-34)

or by combining with equation (5-32):

$$\left(\frac{da}{dN}\right)_{total} = A_w (\text{SEDF})^{m_w} \cdot \left[1 - \Phi^\beta \left(1 - \frac{1}{1 + b_2 a + b_3 a^2 r_h^2}\right)\right]^{-\alpha}$$

(5-35)

The parameters $A_w$ and $m_w$ in this equation differed from the previously fit values (i.e. those in Table 5-3), because the earlier regression was done on all the test results (including those
5.3 Discussion

with hold time), which was effectively the average values for the tested materials. However, with the type of construction in equation (5-34), $A_w$ and $m_w$ should be associated with the reference case (the baseline), therefore smaller values (in particular, of $A_w$) were expected.

Three samples were chosen to represent the continuously cycled test condition (10Cr-RB06), the one with 0.5h hold time (10Cr-RB09) and the one with 1h hold time (10Cr-RB10), respectively. The modified Hall-Petch coefficient was firstly determined by using $\sigma_0 = 70\,\text{MPa}$, $d_{m,\text{start}} = 0.8\,\mu\text{m}$, thus $k_{IP} = 206\,\text{MPa} / \mu\text{m}^{-0.5}$ (Table 5-9) at the initial state. Hence, the constant $\Phi_0$ could be calculated to be $\Phi_0 = 0.77$ (dimensionless). (Later on, it was found that the small variation of $\Phi_0$, e.g. the span in Table 5-9 would not cause big deviations of the fit values, thus only the case of $\Phi_0 = 0.77$ is illustrated in the following analysis).

By referring back to the fit parameters of $d_m$ (which were used to describe the evolution of $d_m$ for the 10Cr steel in Chapter 4, Table 4-4), the remaining unknown terms in equation (5-35) were actually $A_w$, $m_w$, $\beta$ and $\alpha$. Values of these terms could then be calculated through best fit for all three tests (by limiting the smallest deviation in the continuously cycled case). The result is shown in Figure 5-19, with black symbols denoting experimental results and red curves designating the predicted relationships with the damage parameter.

The blue curve is the baseline for this series of tests, i.e. genuine crack growth rate if the microstructure stayed the same during deformation. As clearly shown in this figure, when plotted in linear scale instead of log scale, crack growth rates could not be unified by a single curve. At the same SEDF value, crack growth rate was higher for longer dwelled tests. However, by modifying the original SEDF model by adding a damage function, it was then possible to more accurately describe the respective crack growth rate according to different testing condition/creep-fatigue interaction. Meanwhile, the fictional baseline could be obtained, which designated the ideal case with no microstructure degradation.

In this example, the final expression (with calculated parameters and coefficients) was:
Note that $A_w$ in Figure 5-19 was smaller than that in Figure 5-6 (0.38 compared to 0.6). As illustrated in this example, the effectiveness of this short crack growth model (correlated with the SEDF) could indeed be improved by taking creep-fatigue interaction into consideration, i.e. through the microstructural condition parameter. In this case, SEDF indicates the work done by the creep-fatigue loading for crack development in each cycle, and the correction term with $\Phi$ signifies the instantaneous microstructural status in response to the external forces.

**Modified Skelton model with $\phi$**

Alternatively, this concept of combining the microstructural condition parameter $\Phi$ with the short crack growth model could be applied to the (original) Skelton model (i.e. equation (5-10)), such that the final formula was in the form of:

$$
(da/dN)_{total} = Ba^Q \cdot \left( \frac{1}{1 - \Phi^\beta} \right)^\alpha
$$

or in terms of the crack depth:

$$
(da/dN)_{total} = Bd^Q \cdot \left[ 1 - \Phi^\beta \left( \frac{1}{1 + b_1c_0 + b_2c^\alpha c_h^\beta} \right)^\alpha \right]
$$

Once again, this refined model was demonstrated by the results from three tests with the 10Cr steel, namely, 10Cr-RB06 (no dwell), 10Cr-RB09 (0.5h dwell) and 10Cr-RB10 (1h dwell), in the same way as the previous example of the SEDF model. This time the unknown terms were $B$, $Q$, $\beta$ and $\alpha$. By taking the appropriate known parameters into equation (5-38), the effectiveness of this modified model could be seen in Figure 5-20.
5.3 Discussion

**Figure 5-20** Effectiveness of the modified Skelton model on crack growth rate for 10Cr at 625°C.

The corresponding final expression for this example could also be obtained:

\[
(da/dN)_{total} = 0.002a^{0.5} \cdot \left( \frac{1}{1 - \Phi^{0.53}} \right)^{2.0} \tag{5-39}
\]

In this case the dependency of \( B \) on the controlling strain was not shown, because all the three tests were carried out with the same strain range (i.e. \( \Delta \varepsilon_c = 0.01 \)). However, it is possible to refer back to the previous analysis with the Skelton model, where the relationship between \( \Delta \varepsilon_c \) and \( B \) was already known (e.g. Figure 5-8 and Figure 5-9). In Figure 5-20, the black hollow symbols represent the experimental values, whereas the red curves denote those predicted from equation (5-39). Generally, a close consistency could be found, which was as good as the previous analysis where the pure empirical fit of \( d_c \) was performed (Figure 5-11 and Figure 5-12). The difference lay in the absolute values of \( B \) and \( Q \), and in the current model they were smaller. This was certainly true, because they (i.e. \( B = 0.002 \) and \( Q = 0.5 \)) could in fact estimate the fictional pure crack growth rate when no microstructural change took place (blue dashed baseline in Figure 5-20). (Note that this baseline was lower than that in Figure 5-19).

Moreover, it was possible to compare equation (5-39) (or to be more specific, equation (5-37)) with the previous discussed modified Skelton model with \( d_c \) (i.e. equation (5-15)). The concept of approaching the enhancement on crack growth rate (due to the hold period) was similar: both by introducing an adjustment function to the original crack growth model. Despite the different values of \( B \) and \( Q \) (i.e. equation (5-15) took the low cycle fatigue case as the baseline, whereas equation (5-37) took a pure fatigue case as the baseline), the construction of the adjustment function was similar. More specifically, the exponent had the same value (i.e. \( \alpha = 2 \)); the creep damage \( d_c \) in equation (5-15) was magnified by the number of cycles \( N' \) (i.e. \( N' \cdot d_c \)), whereas the damage parameter \( \Phi \) in equation (5-37) was amplified by a factor of \( \beta \). Since \( \Phi \) was derived from the evolution of micro-grain size, which was inherently
dependent on the elapsed cycles during test, it was reasonable to assume that $\Phi$ was indirectly associated with the number of cycles $N'$. Therefore, $N' \cdot d_c$ and $\Phi^\beta$ were effectively analogous functions to account for the creep-fatigue interaction, but from two different aspects: $d_c$ came from the mechanical response (i.e. creep damage), whereas $\Phi$ came from the microstructural response (i.e. deformation condition). In this study, $d_c$ was estimated from the relaxation behaviour by referring to the material’s creep properties, and a certain amount of overestimation could be observed (i.e. Figure 5-16 for FB2). On the other hand, with the accurately determined values for the damage parameter $\Phi$, those predicted crack growth rates seemed to be more consistent with the experimental values (i.e. Figure 5-19 and Figure 5-20).

This potentially suggested an advantage of a correction on the crack growth models from the microstructural investigation instead of from the mechanical investigation, because the main creep-fatigue interaction in these martensitic steels is a deformation interaction. Consequently, $d_c$ should be replaced by $\Phi$. (In principle, when both creep damage and microstructural variation during creep-fatigue deformation are significant for such martensitic steels, correction terms comprising $d_c$ as well as $\Phi$ should be employed to the basic crack growth models). In future study, the applicability of this $\Phi$ type model should also be verified on other types of materials showing a significant dynamic recovery process during creep-fatigue loading.

### 5.3.5 Improvement by incorporating friction stress $\sigma_F$

In practice, it was easier to obtain the stress/strain response rather than the evolution of microstructural features. Therefore, it would be beneficial if the change of microstructure could be captured by some sort of mechanical response. According to the previous assumption, the damage parameter was based on a Hall-Petch type relationship, which was originally used to describe the yield strength (could be viewed as the upper limit of elastic deformation). During the cycling loading in line with the current test set-up, the elastic regime corresponded to two times the friction stress (i.e. $2\sigma_F$, see section 3.3.3). This cyclic yield strength should in principle be proportional to the uniaxial yield strength, thus a simple postulation could be made:

$$\sigma_y = k \sigma_F$$  \hspace{1cm} (5-40)

where $k$ is the coefficient.

In accordance with equation (5-29), the damage parameter $\Phi$ could be written as:

$$\Phi = \frac{\sigma_y \text{ start} - k \sigma_F}{\sigma_y \text{ start}}$$  \hspace{1cm} (5-41)
5.3 Discussion

In order to estimate the value of \(k\) in equation (5-41), the expression of \(\Phi\) was referred to equation (5-30), such that:

\[
\Phi = \frac{k'_{lp}}{\sigma_{y, start}} \left[ (d_{m, start})^{0.5} - (d_m)^{0.5} \right] = \frac{\sigma_{y, start} - k\sigma_F}{\sigma_{y, start}}
\]  
(5-42)

After rearrangement, \(k\) could be approached by:

\[
k = \frac{\sigma_{y, start} - k'_{lp}}{\sigma_F} \left[ (d_{m, start})^{0.5} - (d_m)^{0.5} \right]
\]  
(5-43)

By bringing in the known experimental values of \(\sigma_F\) (from section 3.3.3) and micro-grain size (from section 4.2.4), the determined values of \(k\) ranged from 1.9 to 2.2 for the examined 10Cr specimens. This was consistent with the previous expectation of 2 (i.e. the range of the elastic regime in a cyclic loaded case, i.e. \(k = 2\)). Finally, the total crack growth rate could be approximated by the following equation:

\[
\left( \frac{da}{dN} \right)_{total} = \left( \frac{da}{dN} \right) \left[ 1 - \left( 1 - \frac{2\sigma_F}{\sigma_{y, start}} \right)^\beta \right]^{-\alpha}
\]  
(5-44)

The effectiveness of this correction function is illustrated in Figure 5-21, with the results from three experiments on 10Cr by using the Skelton model (i.e. with the same derived values for \(\alpha\) and \(\beta\) from equation (5-39)). A larger deviation between the experimental and predicted crack growth rates emerged, which could most probably be from the simplified assumption of \(k\). (The value of \(k\) was set to 2 in equation (5-44), whereas according to the results from equation (5-43), \(k\) ranged from 1.9 to 2.2 from test to test).
Nevertheless, by using this correction function (equation (5-44)), it was possible to roughly estimate the influence of hold time on the short-crack creep-fatigue crack growth rates, without any pre-knowledge of the creep properties of the material, or the information regarding material’s microstructure. All that was required was the stress-strain history in a typical creep-fatigue loading condition. (In order to increase the validity of this model, accurate determination of the exponents (i.e. $\alpha$ and $\beta$) should be employed beforehand).
Chapter 6

Conclusion and outlook

The primary objective of this PhD project is to identify and establish an effective relationship to describe sub-critical crack growth behaviour for two advanced tempered martensitic creep-resistant steels (within the short crack regime where a large proportion of component lifetime is spent). The underlying questions to be answered are:

1) How do stress and strain respond to the applied fatigue and creep loading?

2) How do cracks propagate under different testing conditions?

3) How does microstructure evolve during creep-fatigue deformation?

4) Which model can best describe short crack propagation?

5) Is there a way to increase the effectiveness of short crack growth prediction?

In order to answer these questions, strain controlled short-crack creep-fatigue crack growth experiments have been carried out with single edge-notched round-bar specimens, where the crack development was continuously monitored by using DCPD instrumentation (introduced in Chapter 2). Subsequently, some of the creep-fatigue cracked specimens underwent microstructural investigation by optical and electron microscopy. In particular, an extensive EBSD analysis was carried out to study and characterize the evolution of microstructural entities associated with dynamic recovery due to creep-fatigue loading (e.g. the dimensions of micro-grains).

Based on the experimental results, a comprehensive investigation has been completed on the mechanical response (section 3.1), crack development (section 3.2) as well as the evolution of microstructure (sections 4.1 and 4.2) for the materials of interest subjected to different testing conditions. Finally, various models have been applied to correlate diverse parameters with crack growth rates (section 5.1 and 5.2). The effectiveness of these models has been compared, and possible adjustments for further improvement have been discussed (section 5.3).

Although most of the findings and interpretations have already been stated in previous chapters, the most important conclusions are again summarized in the following paragraphs. Afterwards, the applicability of proposed short crack growth models is reviewed. Finally, suggestions are provided for the unsolved issues in this study.
6.1 Conclusions

In accordance with the major objectives of this PhD project, the most important conclusions from the strain controlled short-crack creep-fatigue crack growth tests are:

i. The applied strain amplitude and hold period (position and duration) are the most influential factors on the number of cycles to 2% load drop as well as on crack development. An increase of temperature from 600°C to 625°C does not significantly accelerate crack propagation.

ii. Instead of direct creep crack growth during the hold time, the enhancement of apparent crack growth rates is due to accelerated fatigue crack propagation as a result of prior creep and strain enhanced oxidation at the crack tip.

iii. In the as-received state, both FB2 and 10Cr exhibit typical martensitic morphologies with finely dispersed precipitates. During deformation, microstructural evolution takes place, which in turn reduces the resistance of the materials to crack development.

iv. The degree of microstructural evolution is governed not only by the applied creep-fatigue loading (i.e. test condition dependent), but also by the measurement locations along the crack propagation path (i.e. crack depth/accumulated inelastic strain dependent). Characteristic values of micro-grain size $d_m$ have been measured for various samples by the EBSD method.

v. When correlating with short crack growth rates, the SEDF model appears to be marginally more effective than the other fracture mechanics based models (for the materials and test conditions adopted in this study), by better incorporating the influence of the applied total strain range and hold periods.

vi. The semi-empirical Skelton model can also predict short crack growth behaviour accurately. Apart from direct curve fitting, it is possible to estimate $d_c$ from stress relaxation behaviour during hold times by referring to the material’s creep properties. With $d_c$ deduced by the ductility exhaustion method, a reasonable preliminary estimation of short-crack creep-fatigue crack growth behaviour can be obtained.

vii. A microstructural condition parameter $\Phi$ has been devised as a more appropriate alternative to account for any loss in the creep-fatigue resistance of advanced martensitic steels. By using a Hall-Petch type relationship, $\Phi$ can be directly related to the micro-grain size $d_m$. Therefore, it is possible to combine a microstructural degradation function with the previously suggested crack growth models to account for changes in deformation condition.

viii. The effectiveness of the modified crack growth models incorporating $\Phi$ has been demonstrated for the SEDF model and the Skelton model. The predicted crack growth rates are more consistent with the experimental values, suggesting an advantage of using both mechanical and microstructural parameters to characterize short-crack creep-fatigue crack growth behaviour for 9-10%Cr martensitic steels.
6.2 Application of short crack growth models

The results in this study promote the use of the SEDF model and the Skelton model relative to other candidate short-crack creep-fatigue crack growth models for advanced martensitic steels. A comparison between them was given in section 5.3.1. Several kinds of modification have also been discussed, which can indeed improve the effectiveness of the prediction on crack growth behaviour.

In principle, these enhanced (short) crack growth models can also be applied in practice (e.g. as part of a high temperature assessment procedure for steam turbines, preferably for martensitic steels). A successful prediction of crack propagation is generally dependent on three factors: a good knowledge of material properties at the operating temperatures; the temperature variation during start-up and shut-down at the target location on components; and the accurate analysis of stress-strain fields. Once all the information is at hand, the models examined in this study can be applied. A recommendation for short-crack creep-fatigue crack growth models to be applied is listed in Table 6-1, illustrating different choices depending on the quality of the inputs (i.e. the required information).

Table 6-1 Recommended short-crack creep-fatigue crack growth models under different conditions for advanced martensitic steels.

<table>
<thead>
<tr>
<th>Required information in addition to crack depth</th>
<th>The SEDF model</th>
<th>The Skelton model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Indicative cyclic stress-strain properties</td>
<td>-</td>
<td>( \frac{da}{dN} )_{Skelton} = Ba^Q</td>
</tr>
<tr>
<td>Complete stress/strain history</td>
<td>( \frac{da}{dN} )_{SEDF} = A_w \left( SEF \right)^{m_w}</td>
<td></td>
</tr>
<tr>
<td>Complete stress/strain history +</td>
<td>( \frac{da}{dN} )_{Skelton} = Ba^Q</td>
<td></td>
</tr>
<tr>
<td>Indicative cyclic yield strength</td>
<td>( \frac{da}{dN} )_{Skelton} \left( 1 - \left( \frac{\sigma_y}{\sigma_y^{\text{start}}} \right)^{\beta} \right)^{-\alpha}</td>
<td></td>
</tr>
<tr>
<td>Complete stress/strain history +</td>
<td>-</td>
<td>( \frac{da}{dN} )_{Skelton} \times \left( 1 + N d_c \right)^2</td>
</tr>
<tr>
<td>Creep properties</td>
<td>( \frac{da}{dN} )_{Skelton} \times \left( 1 - \left( \frac{\sigma_y}{\sigma_y^{\text{start}}} \right)^{\beta} \right)^{-\alpha}</td>
<td></td>
</tr>
<tr>
<td>Complete stress/strain history +</td>
<td>( \frac{da}{dN} )_{Skelton} \times \left( 1 - \left( \frac{\sigma_y}{\sigma_y^{\text{start}}} \right)^{\beta} \right)^{-\alpha}</td>
<td></td>
</tr>
<tr>
<td>Indicative instantaneous micro-grain size</td>
<td>( \frac{da}{dN} )_{Skelton} \times \left( 1 - \left( \frac{\sigma_y}{\sigma_y^{\text{start}}} \right)^{\beta} \right)^{-\alpha}</td>
<td></td>
</tr>
<tr>
<td>( \frac{da}{dN} )_{Skelton} \times \left( 1 - \left( \frac{\sigma_y}{\sigma_y^{\text{start}}} \right)^{\beta} \right)^{-\alpha}</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Generally speaking, when more information is available, a more complex expression of crack growth models can be adopted, which leads to a higher accuracy of estimation.

**Level 1:** The basic Skelton model is typically favoured by industrial users, due to its relatively simple formulation and low requirement of input information (i.e. strain range and instantaneous crack depth). Investigation in this study supports this view, and the results have shown that a further refinement is indeed necessary when creep loading becomes more significant (with a longer hold time) for advanced martensitic steels (as shown in section 5.2.1).

**Level 2:** When the complete stress/strain history is accessible, it is perhaps more appropriate to use the SEDF model to better account for the influence from hold periods (as shown in section 5.1.3). If a similar scheme for calculating SEDF as used in this study is to be employed, a closed loop of stress-strain response is a prerequisite.

**Level 3:** Moreover, if the indicative cyclic yield strength is known (e.g. can be estimated from friction stress in hysteresis loop analysis as shown in section 3.3.3, or from additional hardness test results which can be translated by correlation into strength properties), a refinement can be made by relating the decrease of yield strength to the evolving microstructural condition (i.e. lose of resistance against further deformation/cracking).

**Level 4:** For materials with known creep properties at the operating temperature (e.g. creep ductility, creep rupture time), it is recommended to use the Skelton model modified by a creep damage term $d_c$. This term can be estimated by using the ductility exhaustion method based on material’s creep properties (as shown in section 5.2.2).

**Level 5:** Ideally, for advanced martensitic 9-10%Cr steels, when the microstructural condition is known (e.g. instantaneous micro-grain size), the modified SEDF model or the modified Skelton model with a microstructural condition parameter (e.g. as a function of micro-grain size) has the highest effectiveness for predicting short-crack creep-fatigue crack development (as illustrated in section 5.3.4). In fact, if the dependency of crack depth, hold period, strain range, etc. on the evolution of micro-grain size has been previously obtained (as in this study), then this type of modification requires no further input data at all. Certainly, this approach has the highest requirement for input information, which is in reality difficult to acquire. One possibility might be performing systematic mechanical and microstructural analysis on full-size specimens with close-to-practice testing conditions to establish a database (e.g. on the limiting cases). By comparing real-time microstructure (e.g. from an ex-service replacement part) with the database, a logical estimation of microstructural status can be attained.

It should also be noted that the models including $\Phi$ could be especially useful for materials exhibiting a strong deformation interaction (e.g. martensites). Only when $\Phi$ is not accessible, other models including $d_c$ can be considered as an approximate option. On the other hand, for other materials where no significant deformation interaction takes place (e.g. low alloy steels), $\Phi$ may not be the correct term to account for the creep-fatigue interaction, and a more appropriate correlating parameter is $d_c$. 
6.3 Future work

Despite the promising advantages of these crack growth models, the range of applicability needs to be further validated. For example, the maximum applied hold time for this kind of experiment was limited to one hour in this study. Although evidence has shown that stress relaxation tends to saturate after only a short time, tests with longer hold periods are still worth investigating, e.g. like those involved in real turbine operation. This is because in addition to the deformation response, another type of creep damage is the physical damage (e.g. generation of voids and microcracks). It is absolutely necessary to explore the limitation of the proposed models on the tolerance to creep loading.

It would also be interesting to see the influence of hold periods on crack development at different strain ranges. In practice, the total strain range applied on a laboratory testpiece corresponds to the heating-up or cooling-down rate in turbine operation. This study has adopted the common limiting case of $\Delta \varepsilon = 1\%$ as the starting point for the assessment of creep-fatigue interaction. In future, it is suggested to investigate creep-fatigue crack growth behaviour at diverse strain ranges with respect to practical application.

Moreover, the influence of other testing variables (e.g. strain rate, strain ratio, specimen geometry, notch root radius) on crack development has not been systematically studied in the current analysis. The sensitivity of the calculated/fitted parameters (in various crack growth models) to material variability also needs to be verified (e.g. for other batches of material).

Apart from the above mentioned uncertainties, there were still some unsolved issues revealed during this PhD project. Future work on these remaining topics needs to be carried out to get a full understanding of creep-fatigue crack growth behaviour.

**Transition regime:** As illustrated in Chapter 5, most samples exhibited a transitional $da/dN$ regime at the beginning of each experiment (as cracking first developed from the starter notch), while others displayed a roughly linear stable $da/dN$ right from the start. The transitional regimes were likely to be due to the rapid stress redistribution at the start of test as a consequence of the initially strong phase of dynamic recovery. The current view is that this transitional regime is too short to notably influence crack growth modelling, but the overall effectiveness of crack growth modelling could be improved once the mechanism behind this regime is understood. In future work, interrupted tests can be carried out to examine actual crack initiation/development during this transitional regime.

**Effect of oxidation:** Microstructural analysis has revealed that for all the testpieces in this study, the major/branch crack paths are filled with oxides. Moreover, the oxide layer was already thick compared to relatively shallow crack tips after only a few initial creep-fatigue cycles. It was therefore concluded that oxidation is likely to have played an important role in crack initiation and propagation at high temperatures. However, the magnitude of its influence on crack growth rates has not been rationalized quantitatively. The accuracy of crack development prediction will be increased if the effect of oxidation (time and temperature dependent) can be expressed explicitly. The exact investigation of oxidation during deformation may require a rather complex experimental technique. For example, one
possibility is to employ testing in vacuum or in a chamber with different oxygen partial pressures, preferably with an in situ crack monitoring system.

**Role of second-phase particles:** There were actually two issues concerning second-phase particles in this study. The first issue was related to the large particles (diameter of 5~10µm) in FB2 short crack growth samples, which appeared to induce unexpected microcracks even when no creep loading was present. And the second issue was related to the medium-size particles (diameter of around 1µm) in 10Cr long crack growth samples (shown in Appendix D), which could cause micro-cracking due to creep loading. Explanation and postulation have been made on the identity/origin of these second-phase particles, but no conclusion can be drawn unless examination/confirmation is carried out for the tested materials. One method is to use energy-dispersive X-ray spectroscopy to analyse the chemical composition of these particles.

**Applicability of crack growth models on different materials:** In principle, $d_c$ and $\Phi$ represent different entities: $d_c$ is related to physical creep damage, while $\Phi$ is related to deformation condition. This study has shown that for 9-10%Cr martensitic steels, $\Phi$ is better than $d_c$ for incorporating the effect of creep deformation (when no actual creep crack growth has occurred during hold times). However, its applicability to other materials needs to be verified (e.g. other materials with a strong deformation interaction). It is also necessary to examine the extreme cases where both physical damage and deformational damage are significant.

**Long crack propagation:** This thesis is concerned with short crack growth behaviour. It is also of interest to know how cracks develop when they propagate into the long crack regime. A complementary long-crack creep-fatigue test campaign was carried out for the 10Cr steel (as well as for P91), the details of which can be found in Appendix D. It seemed that $\Delta K_{eq}$ or $\Delta J$, could serve as a common correlating parameter for both short crack and long crack creep-fatigue crack development. However, due to the high creep resistance of the 10Cr steel, the performed long crack propagation tests were only able to reflect an apparent influence of load range, but not of hold time. Future work needs to be implemented to extend the test matrix, as well as to validate the recommended long-crack growth models. Only by that time, similarities and differences between short and long crack growth behaviour under creep-fatigue loading can be obtained more objectively.
Appendix A: Summary of employed short crack growth models

Various models have been employed to correlate with short crack growth rates in Chapter 5. Their formulation and best fit mean values of parameters/ constants are summarized in the following table (i.e. Table A-1).

**Table A-1 The employed short-crack creep-fatigue crack growth models with the computed/best-fit mean values for different materials and testing conditions.**

<table>
<thead>
<tr>
<th>Short-crack creep-fatigue crack growth models</th>
<th>FB2</th>
<th>10Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\frac{da}{dN} = A_K (\Delta K_{eq})^{m_K}$</td>
<td>$A_K = 4.6 \times 10^{-6}$</td>
<td>$A_K = 7.6 \times 10^{-6}$</td>
</tr>
<tr>
<td>$m_K = 1.5$</td>
<td>$m_K = 1.5$</td>
<td></td>
</tr>
<tr>
<td>$\frac{da}{dN} = A_J (\Delta J_r)^{m_J}$</td>
<td>Full calculation (from Shih &amp; Hutchinson)</td>
<td>$A_J = 0.44$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$A_J = 3.96$</td>
</tr>
<tr>
<td></td>
<td>$m_J = 1.31$</td>
<td>$m_J = 1.55$</td>
</tr>
<tr>
<td></td>
<td>Simple calculation (from Dowling)</td>
<td>$A_J = 0.16$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$A_J = 0.64$</td>
</tr>
<tr>
<td></td>
<td>$m_J = 1.1$</td>
<td>$m_J = 1.28$</td>
</tr>
<tr>
<td>$\frac{da}{dN} = A_J (\Delta J_p)^{m_J}$</td>
<td>Full calculation (from Shih &amp; Hutchinson)</td>
<td>$A_J = 0.34$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$A_J = 4.22$</td>
</tr>
<tr>
<td></td>
<td>$m_J = 0.99$</td>
<td>$m_J = 1.4$</td>
</tr>
<tr>
<td></td>
<td>Simple calculation (from Dowling)</td>
<td>$A_J = 0.09$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$A_J = 0.14$</td>
</tr>
<tr>
<td></td>
<td>$m_J = 0.86$</td>
<td>$m_J = 0.86$</td>
</tr>
<tr>
<td>$\frac{da}{dN} = A_w (SEDF)^{m_w}$</td>
<td>$A_w = 0.7$</td>
<td>$A_w = 0.6$</td>
</tr>
<tr>
<td>$m_w = 0.86$</td>
<td>$m_w = 0.86$</td>
<td></td>
</tr>
<tr>
<td>$\frac{da}{dN} = \frac{\pi^2}{8} \frac{\Delta \varepsilon \Delta \sigma^2}{(2T)^2} \times a \left[ 1 + \frac{\pi^2}{8} \left( \frac{\Delta \sigma}{2T} \right)^2 \right]$</td>
<td>$t_h = 0$</td>
<td>$(\Delta \varepsilon = 0.01)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 520$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$(\Delta \varepsilon = 0.01)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 590$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$(\Delta \varepsilon = 0.04)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 390$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$(\Delta \varepsilon = 0.01)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 500$</td>
</tr>
<tr>
<td></td>
<td>$t_h = 0.5h$</td>
<td>$(\Delta \varepsilon = 0.01)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 405$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$(\Delta \varepsilon = 0.01)$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\bar{T} = 440$</td>
</tr>
<tr>
<td>$\frac{da}{dN} = Ba^Q$ with $Q = 1$</td>
<td>$B = 2.7 \times 10^3 \Delta \varepsilon_i^{2.85}$</td>
<td>$B = 2.4 \times 10^3 \Delta \varepsilon_i^{2.85}$</td>
</tr>
<tr>
<td>$(\frac{da}{dN})_{total} = Ba^Q (1 + N \delta_c)^{2}$ with $Q = 1$</td>
<td>$B = 2.1 \times 10^3 \Delta \varepsilon_i^{2.85}$</td>
<td>$B = 1.5 \times 10^3 \Delta \varepsilon_i^{2.85}$</td>
</tr>
<tr>
<td>$d_c$ from direct curve-fitting</td>
<td>$(t_h = 0.5h)$</td>
<td>$(t_h = 1h)$</td>
</tr>
<tr>
<td></td>
<td>$d_c = 7 \times 10^{-4}$</td>
<td>$d_c = 9 \times 10^{-4}$</td>
</tr>
</tbody>
</table>
### Appendix A: Summary of employed short crack growth models

<table>
<thead>
<tr>
<th>$d_c$ from ductility exhaustion method</th>
<th>$t_h = 0.5h$</th>
<th>$d_c = 1.8 \times 10^{-3}$</th>
<th>$t_h = 1h$</th>
<th>$d_c = 2.1 \times 10^{-3}$</th>
<th>$t_h = 10h$</th>
<th>$d_c = 3.2 \times 10^{-3}$</th>
<th>$t_h = 100h$</th>
<th>$d_c = 4.0 \times 10^{-3}$</th>
</tr>
</thead>
</table>

\[
(\frac{da}{dN})_{total} = A_w \left( SEDF \right)^{m_w} \cdot \left( \frac{1}{1 - \Phi^a} \right)^\alpha
\]

- $A_w = 0.38$
- $m_w = 0.86$
- $\alpha = 2$
- $\beta = 0.8$

\[
(\frac{da}{dN})_{total} = B a^Q \cdot \left( \frac{1}{1 - \Phi^b} \right)^\beta
\]

- $(\Delta \varepsilon_t = 0.01)$
- $B = 0.002$
- $Q = 0.5$
- $\alpha = 2$
- $\beta = 0.53$

\[
\Phi \text{ from micro-grain size: } \Phi = \Phi_0 \left[ 1 - \frac{1}{\sqrt{1 + b_1 a + b_2 a^a t_h^2}} \right]
\]

- $b_1 = 0.29$
- $b_1 = 0.26$
- $b_2 = 0.34$
- $b_1 = 0.85$
- $c_1 = 1.0$
- $a_1 = 0.62$
- $c_2 = 2.1$
- $c_2 = 0.58$

\[
\Phi \text{ from friction stress: } \Phi = 1 - \frac{k \sigma_F}{\sigma_{y, \text{start}}}
\]

- $k \approx 2$
Appendix B: Compliance check for short crack growth specimens

Although for both FB2 and 10Cr, SENT testpieces (i.e. round-bar shape with a chordal crack starter) were taken from the same batches of material and instrumented in an identical way, the effect of material variability might still influence the mechanical properties revealed by different specimens. This could result from microstructural inhomogeneity within the source blocks of material from the large production forgings. In addition, geometry errors through the specimen manufacturing process, or assembly differences during testing (e.g. extensometer placement) could also contribute to the uncertainty. To ensure a valid test result, the compliance of testpieces was firstly examined, by comparing the apparent Young’s modulus/compliance and (nominal) yield strength before the formal creep-fatigue tests.

The apparent Young’s modulus/compliance was individually checked at room temperature (before heating up the specimen) as well as at the testing temperatures (right before the creep-fatigue loading). Although $E$ was determined from the net section stress, the presence of the 0.2mm deep crack starter made the testpiece slightly deviate from the purely tensile loaded condition. However the influence was considered to be negligible (the net section was 99.33% as large as the gross section). Examples from 10Cr testpieces are illustrated in Figure B-1.

At room temperature, the average elastic modulus was around 210GPa, whereas at the testing temperatures the average value was about 145GPa. One would expect a larger elastic modulus at 600°C than at 625°C. However, judging from the respective positions of the square and circular symbols in Figure B-1, this pattern was almost fully concealed due to the scattering. Nevertheless, the deviation was smaller than the ±10% scatter range.

For convenience, values of (nominal) yield strength were obtained by inspecting the first cycle of each creep-fatigue test. Because there was no prior deformation history, this first cycle could potentially be regarded as a monotonically loaded case, and the (nominal) yield strength was
simply determined by identifying the first point deviated from linearity (the proportionality limit). This scheme would lead to slightly smaller stress values than the traditional 0.2% proof strength. The results are plotted in Figure B-2 and the average values are listed in Table B-1. Because the testpieces were manufactured with 0.2mm deep crack starters, the yield strength values here were technically nominal values calculated from the net section area of the edge-notched round bar specimens.

Table B-1 Average yield strength values for FB2 and 10Cr at 600°C and 625°C (effectively nominal values of the proportionality limit).

<table>
<thead>
<tr>
<th></th>
<th>FB2</th>
<th>10Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp (°C)</td>
<td>600</td>
<td>625</td>
</tr>
<tr>
<td>Yield strength (MPa)</td>
<td>304.4</td>
<td>303.6</td>
</tr>
</tbody>
</table>

It was found that there was indeed some deviation in yield strength values, even for the same material at the same temperature under the same strain rate. However, the variability was within the ±10% scatter band (red dashed lines in Figure B-2). For both materials, the average yield strength was slightly above 300MPa at 600°C, with 10Cr actually having slightly larger values. Moreover, the yield strength of 10Cr dropped more quickly as temperature rose to 625°C.

![Figure B-2](image)

**Figure B-2** Comparison of (nominal) yield strength values for FB2 and 10Cr specimens, with ±10% scatter range marked by arrows.

Apart from the purpose of examining material variability and experimental set-up, a more important reason for this part of the study was to obtain average values which could serve as reference values to assist crack growth modelling in section 5.1.4 and 5.3.4.
Appendix C: Factors on DCPD variation during dwell periods

Generally, the recorded DCPD signals could be decomposed into the true values and the background noise. The reason behind the noise signal could be mostly attributed to either temperature fluctuation or thermoelectric EMFs. Although throughout all the tests, temperature variation was controlled to within ±1°C, it was possible that the local variation was larger (e.g. due to heavy localized deformation at the crack tip). The electrical resistivity of metals is normally very sensitive to temperature, therefore the voltage value could also change in accordance with a changing temperature. At the same time, the thermoelectric EMF could not be fully avoided and induced some errors in the data acquisition process. These two obstacles prevented the direct assessment of the raw data, thus smoothing techniques were applied.

After noise reduction, the comparatively true variation of the signal could be acquired. Based on these results, it was possible to analyse the influence of different factors (other than pure creep crack growth) on the change of voltage values during the dwell periods. This included oxidation effects, microstructural evolution and the formation of branch cracks from the main crack.

Under tensile peak force, the crack was initially fully open at the start of dwell. Oxygen reacted with freshly torn metal surfaces and formed chromium and iron rich oxide layers, which interconnected upper and lower crack surfaces. These includes (Fe,Cr)$_2$O$_3$ and (Fe,Cr)$_3$O$_4$. However, Fe$_3$O$_4$ can be semi-conductive when the temperature is above 300K (10$^{-3}$ as conductive as pure iron). Thus, additional surfaces could be produced for the current to pass by and resulted in a small decrease in voltage values. As the time lapsed, the oxide layer grew thicker and generated more linkages between crack surfaces, which brought about a gradual decrease in PD value. Note that the final oxidized layer in dwelled specimens was found to be 20~50μm thick (after 2% load drop point), whereas the absolute crack advancement in a single cycle was normally as large as several microns. (Of course, when comparing experiments under vacuum and in air as introduced in section 1.3.4, it appears that oxidation effect will generally lead to faster crack propagation).

Another significant factor was the microstructural evolution. From the analysis shown in section 4.2.3 and 4.2.4 in Chapter 4, the tested martensitic steels had experienced significant changes in microstructure, especially those tested with hold periods. More specifically, recovery may take place during the dwell, which is likely to be associated with the decrease of dislocation densities as well as sub-grain numbers (see section 1.5.3). This change had an impact on the electrical resistivity of the material, because local inhomogeneity could largely influence the ability of free electrons to travel through the lattice. To put it simply, a more recovered microstructure would have a lower electrical resistivity, thus a decrease of the voltage value. This was specific to circumstances where dynamic recovery dominated. (Certainly at the start of hold period, the resistivity of the specimen was increased due to plastic deformation/dislocation multiplication).
Finally, a common feature for the dwelled specimens was the occurrence of branches of the major propagating crack (e.g. Figure 4-9). They were normally inclined from the loading direction, and stopped growing at around hundreds of microns. For a compressive dwelled specimen, branch cracks could already appear right from the beginning (technically, it was not known at that time which of the crack branches would grow into the major crack), assisted by oxidation effects. In case of an already existing major crack, branch cracks near the crack tip might still have the driving force to grow, provided that a rough surface-pair and the compressive force could cause locally uneven stress distribution. Although the major crack stayed the same, the changed current flow at the crack tip could lead to different electrical potential, which could explain the positive value of mean voltage change during the hold period in compressive dwelled tests. On the other hand, those already existing branch cracks could also close up under compressive force, leading to a decrease in voltage values.

Table C-1 Influence of different factors on voltage variation during the hold period (with ‘+’ showing a positive change and ‘−’ showing a negative change).

<table>
<thead>
<tr>
<th>Influence of different factors on DCPD values during the hold period</th>
</tr>
</thead>
<tbody>
<tr>
<td>True signal</td>
</tr>
<tr>
<td>Creep crack growth</td>
</tr>
<tr>
<td>+</td>
</tr>
<tr>
<td>Noise signal</td>
</tr>
<tr>
<td>Temperature fluctuation</td>
</tr>
</tbody>
</table>

Table C-1 has listed the discussed parameters for voltage variation during hold periods, and it could be concluded that the voltage change during the dwell was the consequence of a complex interaction between multiple contributory factors. It was difficult to separate the respective contributions of the different factors. Indeed, 30min hold time was relatively short for significant true creep crack growth to appear (unless the driving force was very big, e.g. a specimen with an already large crack development, thus a small ligament in a tensile dwell). What was observed from the DCPD readings during dwell could only partially be related to crack depth information.
Appendix D: Long-crack creep-fatigue crack growth tests

In order to fully understand the differences and similarities between short-crack and long-crack creep-fatigue crack growth behaviour, additional investigation concerning long crack development had been carried out, serving as a good supplement and acting as the starting point for future study. (The original intention was to participate in the ASTM inter-laboratory comparison/round-robin organized to provide the basis for precision and bias statements for the new E2760 standard concerned with creep-fatigue crack growth testing).²

A different type of specimen geometry was adopted, in order to compensate for the different loading conditions, (Figure 1-7b rather than Figure 1-7a, as used in short crack growth tests). Two series of isothermal creep-fatigue tests on compact tension specimens were conducted in accordance with the requirements of the ASTM standards and guidelines for 10Cr and P91 (a 9%Cr creep resistant steel). CT testpieces were cracked under load control with a positive ratio of \( R = 0.1 \). Hold time \( t_h \) was either zero or \( t_h = 600s \) at the tensile peak load. Afterwards, the testpieces were broken open for the final crack depth calibration, by linear interpolation and by Johnson’s formula.

It was found that the employed maximum load had the dominant effect on creep deformation as well as crack growth rate. Under the same testing condition, P91 testpieces exhibited a larger \( \Delta V_c \) (LLD during the hold period) than that in the 10Cr testpieces (because the strength of P91 is significantly lower than that of 10Cr). Accordingly, for the same crack size 10Cr testpieces displayed a much slower crack growth rate than the P91 testpieces. This confirmed that the tested P91 material was more susceptible to creep-fatigue crack propagation than the 10Cr steel. In the meantime, some microstructural analyses had been performed to identify the crack propagation path and the occurrence of physical damage due to creep-fatigue loading. Despite the transgranular profile of branch cracks, an increasing quantity of micro-cracks were observed along with crack propagation.

Finally, to rationalize crack growth behaviour under diverse creep-fatigue testing conditions, various models were used to correlate different parameters with long crack growth rates. Three fracture mechanics based models (\( K \) type, \( J \) type and \( (C)_{avg} \) type) were employed. For P91 testpieces, the results are generally in good agreement with the ASTM round-robin pilot study.²¹² For 10Cr testpieces, comparisons were made with the results from short crack growth tests.

These points will be explained in more details in the following sections. First, the experimental conditions and the test matrix for the long crack growth specimens are given. Afterwards, the cyclic load-displacement response as well as the crack development characteristics exhibited by the tested specimens are shown. Then, the applicability and effectiveness of three crack growth models are discussed. Finally, a short section concerning the microstructural investigation is presented.
D.1 Test conditions for CT specimens

One of the materials for long crack growth tests is P91 (modified 9Cr-1Mo steel), which was developed in the early 1980s by Oak Ridge National Laboratory for application in main steam pipes, boilers, headers, etc. in supercritical fossil power plants as a replacement for low alloy austenitic and ferritic stainless steels.\textsuperscript{119, 213} The test material in this study was provided by EPRI (the Electric Power Research Institute) for the round-robin in support of ASTM E2760. Testpieces were obtained from a reheat treated ex-service pipe.\textsuperscript{214} The composition of P91 is listed in Table D-1.\textsuperscript{119} (The composition of 10Cr was already given in Table 2-1 in Chapter 2).

Table D-1 Chemical composition of the tested P91 material (wt.%).

<table>
<thead>
<tr>
<th>Steels</th>
<th>Chemical composition, wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>P91</td>
<td>0.11</td>
</tr>
</tbody>
</table>

Tests were conducted in accordance with the requirements of the ASTM standards and guidelines to investigate long-crack creep-fatigue crack growth behaviour.\textsuperscript{2, 212, 214} CT type specimens were manufactured, with width $W=50$mm, thickness $B=12.5$mm and height=62.5mm. The testpieces were then side-grooved to a depth of 1.25mm (i.e. with $B_N=10$mm) to prevent crack front tunnelling at high temperatures.

![Figure D-1 Details of compact tension specimen geometry in accordance with ASTM standard.](image)

The initial crack starter was machined by EDM with a final thickness of 0.15mm, and the crack length to width ratio, $a/W=0.4$ (i.e. initial crack size of 20mm). A sketch of the geometry and dimensions is shown in Figure D-1, with dimensions given in blue and DCPD wire locations indicated in red.
Appendix D: Long-crack creep-fatigue crack growth tests

Load line displacement was measured by using the same side-contacting extensometer as in the short-crack creep-fatigue crack growth tests, whereby the extensometer legs were inserted into specially machined grooves, to cross the crack plane at the load line. The development of cracks was continuously observed by a DCPD monitoring device, with two voltage output leads connected diagonally across the crack plane, with 5mm vertical distance (DCPD method).

Three type-K thermocouples were spot welded onto the two sides and the back of the testpiece to ensure a homogeneous temperature distribution was provided by the resistance heating furnace. An illustration of this experimental set-up is shown in Figure D-2 (the clevice and extensometer are not shown for clarity).

Load controlled tests were performed with the load/stress ratio $R_\sigma=0.1$ and a triangular waveform where the loading and unloading times were 2s each. A hold period of 600s was implemented at the maximum load (7.5kN, 9kN or 12kN), with the temperature of 600°C or 625°C. Testing parameters are listed in Table D-2.

For tests with hold time, a short period of continuous cycling with the same load range was employed to produce true natural crack starters and to eliminate any machined notch effect (analogous to pre-cracking). Therefore, the true starting crack depth was not always 20mm. All tests were stopped far away from total fracture, with a maximum of 10mm net crack extension. After the experiment, each testpiece was broken open by room temperature fatigue loading, where the initial and final crack sizes could be measured optically. This was done by drawing five parallel equally spaced measurement lines through the net thickness, and
Appendix D: Long-crack creep-fatigue crack growth tests

adopting average crack size values from them (Figure D-3). Finally, by using a linear conversion formula, crack depth information could be obtained.

Table D-2 Creep-fatigue testing conditions for CT specimens.

<table>
<thead>
<tr>
<th>Testpiece No.</th>
<th>Internal No.</th>
<th>TEMP (°C)</th>
<th>Hold time (s)</th>
<th>Max load (kN)</th>
<th>Min load (kN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P91-CT01</td>
<td>HT1613</td>
<td>625</td>
<td>600</td>
<td>9.0</td>
<td>0.90</td>
</tr>
<tr>
<td>P91-CT02</td>
<td>HT1614</td>
<td>625</td>
<td>600</td>
<td>7.5</td>
<td>0.75</td>
</tr>
<tr>
<td>10Cr-CT01</td>
<td>HT1473</td>
<td>600</td>
<td>0</td>
<td>9.0</td>
<td>0.90</td>
</tr>
<tr>
<td>10Cr-CT02</td>
<td>HT1474</td>
<td>600</td>
<td>0</td>
<td>7.5</td>
<td>0.75</td>
</tr>
<tr>
<td>10Cr-CT03</td>
<td>HT1731</td>
<td>625</td>
<td>600</td>
<td>7.5</td>
<td>0.75</td>
</tr>
<tr>
<td>10Cr-CT04</td>
<td>HT1732</td>
<td>625</td>
<td>600</td>
<td>9.0</td>
<td>0.90</td>
</tr>
<tr>
<td>10Cr-CT05</td>
<td>HT1733</td>
<td>625</td>
<td>600</td>
<td>12.0</td>
<td>1.20</td>
</tr>
</tbody>
</table>

Figure D-3 Method of crack depth measurement.

Figure D-4 Cutting scheme for CT specimens.
Appendix D: Long-crack creep-fatigue crack growth tests

For CT specimens, only a few were sectioned and examined under the microscope. The cutting scheme is illustrated in Figure D-4. The thickness of the first extracted thin slice was 3mm, in order to ensure a sufficient remaining testpiece thickness for the final crack measurement, as well as enough material for the microstructural analysis. Immediately after this, the second cropping was made to obtain a 21x10mm² sample. This area was intended to contain the initial notch and the whole cracking region (material influenced by creep-fatigue deformation), while not being too big to increase the cost of sample preparation.

Preliminary results for the CT test campaign are listed in Table D-3. Detailed analyses will be demonstrated in the following sections.

Table D-3 Creep-fatigue test results for CT specimens.

<table>
<thead>
<tr>
<th>Testpiece No.</th>
<th>Internal No.</th>
<th>Initial $\Delta K$ (MPa√m)</th>
<th>Final $\Delta K$ (MPa√m)</th>
<th>Initial crack length (mm)</th>
<th>Final crack length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P91-CT01</td>
<td>HT1613</td>
<td>24.25</td>
<td>44.24</td>
<td>20.77</td>
<td>30.48</td>
</tr>
<tr>
<td>P91-CT02</td>
<td>HT1614</td>
<td>19.10</td>
<td>41.95</td>
<td>20.17</td>
<td>31.37</td>
</tr>
<tr>
<td>10Cr-CT01</td>
<td>HT1473</td>
<td>23.02</td>
<td>38.17</td>
<td>20.00</td>
<td>28.49</td>
</tr>
<tr>
<td>10Cr-CT02</td>
<td>HT1474</td>
<td>19.17</td>
<td>28.43</td>
<td>20.00</td>
<td>26.80</td>
</tr>
<tr>
<td>10Cr-CT03¹</td>
<td>HT1731¹</td>
<td>19.99</td>
<td>22.63</td>
<td>20.27</td>
<td>22.86</td>
</tr>
<tr>
<td>10Cr-CT04</td>
<td>HT1732</td>
<td>23.98</td>
<td>32.64</td>
<td>20.49</td>
<td>26.13</td>
</tr>
<tr>
<td>10Cr-CT05²</td>
<td>HT1733²</td>
<td>31.60</td>
<td>35.65</td>
<td>20.41</td>
<td>22.59</td>
</tr>
</tbody>
</table>

(1) This testpiece was terminated due to an excessively long duration.
(2) This testpiece was terminated due to machine interruption.

D.2 Experimental results

Cyclic load-displacement behaviour

The experiments to determine long crack growth were all performed in load control, while the degree of deformation was measured by the load line displacement $V$ across the crack opening. Therefore, instead of the stress-strain behaviour, cyclic load-displacement behaviour was studied.

As already explained in Chapter 1, the relatively small applied load range (compared with a short crack growth test) would generate mainly elastic deformation, whereas the dwell period at maximum load could slightly increase the inelastic deformation (i.e. Figure 1-4). This is illustrated in Figure D-5, where the evolution of hysteresis loops for 10Cr-CT01 and P91-CT01 is shown.

In Figure D-5a, the load-$V$ curves in the ramping-up and ramping-down periods were almost linear within a single continuously cycled loop, which demonstrated that the deformation during ramping was predominantly elastic. With increasing number of cycles, Load line displacement
Appendix D: Long-crack creep-fatigue crack growth tests

at the maximum load increased significantly, while the shift at the minimum load was comparatively small.

In Figure D-5b, a clear plateau could be observed at the maximum load, together with a wider loop width which represented the inelastic deformation during the hold period. This illustrated the accumulation of creep deformation, which led to a ratchetting effect when $V$ grew to large values.

![Figure D-5 V(P) hysteresis loop evolution for: (a) a continuously cycled 10Cr specimen and (b) a 600s dwelled P91 specimen.](image)

As one could anticipate from Figure D-5b, large inelastic deformation occurred during long-duration tests especially for P91 material, which made the $V$ too big to be accommodated by the knife edges of the extensometer (the distance between extensometer legs reached its limit). Therefore, the extensometer had to be pulled out at a certain stage and then the test was continued without continuous information of $V$ (although load and DCPD values were still recorded).

One difficulty associated with the raw data extraction was to determine the $V$ values accurately, especially in the case of dwelled specimens. In principle, the logic to calculate $\Delta V_c$ was to compare the $V$ change between the end and start of the hold period (the last and first sampling point of the dwell). However, due to the different sampling frequencies during test recording ($f = 50\text{Hz}$ at the ramping stages; and $f = 0.2\text{Hz}$ during the dwell in order to keep a relatively small output file), the traditional scheme might not be appropriate. This was because the change of $V$ was very quick during the start of the dwell, i.e. the first sampling point of the hold period might already be away from the end of the last sampling point of the ramping-up period. Therefore, a new method was devised to overcome this problem, which is shown in Figure D-6.

The ramping-up stage was modelled through a polynomial fit, while the ramping-down period was made by a linear fit. The $V$ difference during the dwell was calculated via fictional interceptions at the peak load. Actually, by applying this scheme another problem was also solved, which was the scattering of load values at designated peak positions. Finally, it was
possible to accurately determine $V'$ values, which were crucial for the subsequent crack growth modelling.

![Figure D-6 Method to determine LLD values.](image)

**Crack development**

Along with the load-displacement variation in each cycle, the voltage value obtained from the DCPD monitoring system also changed, both in the ramping parts and dwell parts. One example of the voltage evolution during a 600s tensile hold test is shown in Figure D-7.

![Figure D-7 Voltage and load evolution during a single cycle for a long crack growth test with 600s hold in tension (for a P91 testpiece).](image)

This figure is plotted in double y-axis style, with black points denoting voltage values and blue points denoting load values. The horizontal axis designated the lapsed time in this cycle. Because the hold period was much longer than the ramping period, a break in the x-axis has been introduced to set this figure in a proper scale. There seemed to be a good
correspondence between (smoothed) load and (smoothed) voltage values, and two main features could be noticed: (1) the voltage value at the end of this cycle was apparently larger than that at the beginning of this cycle, which could be interpreted as the occurrence of a certain crack advancement; (2) the voltage value during the hold period increased slightly, which presumably also indicated a minute crack development. (In reality the scattering of voltage values during the hold time was much higher than that exhibited in Figure D-7. This limitation restrained the DCPD technique from being directly applied in the determination of starting and ending voltage values for a hold period. Thus only the maximum PD values in each cycle were used for the calibration process).

For CT specimens with the standard geometry and DCPD set-up, the recorded voltage values can easily be transferred to crack depth value via two calibration methods: by linear interpolation and/or by Johnson’s formula.

The first method is recommended by the ASTM standard, under the condition of a known initial and final crack size. Since all the crack fronts were almost straight in the investigated CT specimens, the instantaneous crack extensions at any intermediate point was scaled linearly through equation (D-1).

\[
a = a_0 + (a_f - a_0) \cdot \frac{V_f - V_02}{V_f - V_02}
\]

where \(a_0\) and \(a_f\) are the initial and final crack depths; \(V_02\) and \(V_f\) are the initial and final voltage values.

Johnson’s formula was also used as a reference method to validate the accuracy of linear interpretation and rule out errors associate with final crack depth measurement. The expression of Johnson’s formula is

![Comparison between Linear interpolation method and Johnson’s formula (in DCPD to crack length conversion).](image)
Appendix D: Long-crack creep-fatigue crack growth tests

\[
\frac{a}{W} = \frac{2}{\pi} \cos^{-1} \left[ \frac{\cosh \left( \pi \frac{Y_0}{2W} \right)}{\cosh \left( \frac{V}{V_0} \cos^{-1} \left( \frac{\cosh \pi \frac{Y_0}{2W}}{\cos \pi a/W} \right) \right)} \right] \tag{D-2}
\]

where \(Y_0\) is the half distance between the output voltage leads.\(^2\)

For all long-crack creep-fatigue crack growth experiments, the correspondence between those two methods was satisfying. One example is shown in Figure D-8, in which the two curves were almost identical. Hence, voltage values were confidently transferred to crack depth values, for subsequent analysis.

D.3 Crack Growth Modelling

In this section, three fracture mechanics based models are used to predict the crack development in long-crack creep-fatigue crack growth tests. They are a \(K\) type model; a \(J\) type model and a \(\mathcal{C}_{tavg}\) type model. For the P91 testpieces, their results were in principle in good agreement with the ASTM creep-fatigue crack growth round-robin pilot study. Finally, a short discussion on the three models and the possible resemblance between short and long crack growth behaviour is also given.

\textit{da/dN-\(\Delta K\) model}

For CT specimens with geometry/dimensions in accordance with the ASTM standard E2760, the expression of stress intensity factor \(K\) is given as:\(^{2,212}\)

\[
K = \frac{P}{(BB_N)^{1/2} W^{1/2}} F\left(\frac{a}{W}\right) \tag{D-3}
\]

where the crack depth dependent term \(F\left(\frac{a}{W}\right)\) can be approximated by

\[
F = \begin{bmatrix} 2 + \frac{a}{W} \\ \left(1 - \frac{a}{W}\right)^{3/2} \end{bmatrix} \left( 0.886 + 4.64\left(\frac{a}{W}\right) - 13.32\left(\frac{a}{W}\right)^2 + 14.72\left(\frac{a}{W}\right)^3 - 5.6\left(\frac{a}{W}\right)^4 \right) \tag{D-4}
\]

Therefore, the relationship between \(\Delta K\) values and crack growth rates can be determined, in the form of a Paris type equation:

\[
\frac{da}{dN} = A_K \left(\Delta K\right)^{mc} \tag{D-5}
\]
Appendix D: Long-crack creep-fatigue crack growth tests

Figure D-9 Creep-fatigue crack growth rates expressed as a function of $\Delta K$ for (a) P91 specimens at 625°C and (b) 10Cr specimens (hatched area and reference line from the pilot study). (The testing parameters can be found in Table D-2).

For the load controlled P91 experiments, this was depicted in Figure D-9a. The blue dashed reference line is plotted by using the known Paris coefficient and exponent as $A_k = 3.5 \times 10^{-6}$ and $m_k = 1.568$, from the regression data of continuously cycled tests (from the pilot study, i.e. Ref.212); whereas the hatched data band also came from the pilot tests with the same testing conditions as in the current study (i.e. 600s hold period at 625°C). Reasonable agreement between the current test results and those determined in the pilot study was found.

A similar calculation scheme was employed on 10Cr specimens. As shown in Figure D-9b, crack growth rates for two continuously cycled testpieces (10Cr-CT01 and 10Cr-CT02) as well as two dwelled testpieces (10Cr-CT04 and 10Cr-CT05) were correlated with $\Delta K$. The reference dashed line was plotted by using a fitting constant and the exponent derived from the continuously cycled tests in P91 (i.e. $A_k = 3.5 \times 10^{-6}$ and $m_k = 1.568$). As can be seen, this line could almost also describe the crack growth behaviour of 10Cr, regardless of the hold period. This on one side illustrated the similarity in long crack growth behaviour for these two materials in tests without hold time. On the other side, it demonstrated a strong resistance of the 10Cr steel against creep crack propagation during hold time relative to the P91 material (i.e. compared with Figure D-9a).

Since the equivalent stress intensity factor $\Delta K_{eq}$ (i.e. equation (5-1)) reduces down to the classical stress intensity factor $\Delta K$ when the plastic portion is absent, potentially $\Delta K_{eq}$ should be able to unify the expressions to designate crack growth behaviour in both the short and long crack regimes. By comparing the fitting parameters in Figure D-9b (10Cr, long crack growth) and those in Table 5-1 (10Cr, short crack growth), the values of exponent $m_k$ are indeed similar.

As an example, two 10Cr short crack growth tests were selected, i.e. 10Cr-RB03 (at 600°C) and 10Cr-RB08 (at 625°C). These two experiments were performed with a relatively lower total strain range (i.e. $\Delta \varepsilon_t = 0.5\%$), and therefore their creep-fatigue deformation conditions should be comparable with those in long crack growth tests. The relationship between $\Delta K_{eq}$ and $da/dN$ is plotted in Figure D-10. It is clear that despite different specimen geometries and crack front shapes, there is a rather consistent correlation between the (equivalent) stress...
Appendix D: Long-crack creep-fatigue crack growth tests

intensity factor and crack growth rate. It can therefore be inferred that (at least) for long crack growth tests without significant creep damage and short crack growth tests without large plastic deformation (and/or substantial creep damage), $\Delta K_{eq}$ can be adopted as a unified crack tip parameter.

\[
\frac{da}{dN} \cdot (C_t)_{avg} \text{ model}
\]

In the meantime, while the stress intensity factor is often not able to take account of the influence of hold periods on crack growth rate for P91 specimens (reflected by the large deviation between the dashed line and the hatched area in Figure D-9a), another crack tip parameter $(C_t)_{avg}$ is believed to be potentially the best correlating parameter to describe creep crack growth behaviour during dwell. As introduced in section 1.4.1, for CT specimens $(C_t)_{avg}$ can be written as:

\[
(C_t)_{avg} = \frac{\Delta P \Delta V_c}{(BB_N)^{0.5} W_h} \cdot \frac{F'}{F}
\]  \hspace{1cm} (D-6)

where the geometrically associated factor $F'/F$ is given by:

\[
\frac{F'}{F} = \left[ \frac{1}{2 + a/W} \right] + \left[ \frac{3}{2(1-a/W)} \right]
\]  \hspace{1cm} (D-7)

\[
\left[ \frac{4.64 - 26.64(a/W) + 44.16(a/W)^2 - 22.4(a/W)^3}{0.886 + 4.64(a/W) - 13.32(a/W)^2 + 14.72(a/W)^3 - 5.6(a/W)^4} \right]
\]
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As suggested by the standard (i.e. ASTM E2760)\(^2\), either a linear damage summation model or a dominant damage model can be used to assess crack growth behaviour under creep-fatigue interaction conditions. Since the crack growth rate per cycle could simply be transferred into a time scale by dividing the hold time in each cycle, crack propagation during creep can be written as

\[
\left(\frac{da}{dt}\right)_{avg} = \frac{1}{t_h} \left(\frac{da}{dN}\right)_{creep} = \frac{1}{t_h} \left[ \left(\frac{da}{dN}\right)_{total} - \left(\frac{da}{dN}\right)_{fatigue} \right]
\]

(D-8)

according to the damage summation model. Actually, as displayed in Figure D-9a, crack growth rates for tests with 600s dwell were an order of magnitude larger than those in continuously cycled tests (e.g. the hatched scatter band compared to the dashed blue line). Therefore, the dominance of time-dependent creep crack growth was expected, whereas the contribution from fatigue crack growth was rather small. Therefore, an application of the dominant damage model was reasonable, where the average crack growth rate per unit time could be related to the \((C_i)_{avg}\) parameter:

\[
\left(\frac{da}{dt}\right)_{avg} = A_c \cdot (C_i)_{avg}^{mc}
\]

(D-9)

The results for P91 specimens were presented in Figure D-11. Compared with the results from the pilot tests (i.e. the blue hatched area from Ref.\(^{212}\)), current analysis showed a higher value of \((C_i)_{avg}\), which effectively shifted the curves to the right side. This was possibly due to the fact that values of load line displacement \(\Delta V_c\) during dwell was strictly calculated by the difference between the end of ramp and start of dwell, without using any smoothing function (see Figure D-6 for more details).

In view of the evidence in Figure D-9b, it was speculated that creep crack growth was not the dominant damage mechanism for the tested 10Cr specimens under the adopted testing conditions (because otherwise the crack growth rates of the dwelled samples would be much lower).
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larger than those of continuously cycled samples). Therefore, although the values of \((C_t)_{avg}\) for 10Cr were also calculated (which were in fact close to those in P91 samples), the plot of \((C_t)_{avg}\) associated with the crack growth rates was not shown here, because the dominant damage model could not be legitimately applied in this case.

**da/dN-\(\Delta J\_t\) model**

\(J\) integral was also used to correlate crack growth rates, where the calculation scheme was based on ASTM E1820.\(^{216}\) As has already been introduced in Chapter 1, the value of \(\Delta J_t\) comprises an elastic part and a plastic part. For CT specimens at the beginning of an experiment, a general expression is:

\[
\Delta J_{t(i+1)} = \Delta J_{t(i)} + \Delta J_{p(i+1)}
\]

\[
= \frac{\Delta K_{\text{eff}}^2(1-\nu^2)}{E} + \left[\frac{2.0 + 0.522(W-a_0)/W}{B_N(W-a_0)}\right] \cdot A_{pl(i+1)}
\]

(D-10)

Here \(i\) represents the number of each cycle, and \(\nu\) is the Poisson’s ratio. \(A_{pl}\) stands for the surrounded area in the load-displacement curve in a single loop, and \(B_N\) is the net section thickness. Subsequently, the plastic part of \(J\) integral can be calculated in an incremental way, such that:

\[
\Delta J_{p(i+1)} = \left[\Delta J_{p(i)} + \left[\frac{2 + 0.522(W-a_0)/W}{W-a_0}\right] \cdot \frac{A_{pl(i+1)} - A_{pl(i)}}{B_N}\right] \times \left[1 - \frac{a_{i+1} - a_i}{W-a_i}\right] \left[1 + \frac{0.76(W-a_i)}{W}\right]
\]

(D-11)

where

\[
A_{pl(i+1)} - A_{pl(i)} = \frac{[\Delta P_{(i+1)} + \Delta P_{(i)}] \times [\Delta V_{pl(i+1)} - \Delta V_{pl(i)}]}{2}
\]

(D-12)

with \(\Delta V_{pl} =\)plastic part of the load line displacement

\[
\Delta V_{pl(i)} = \Delta V_{\text{total}(i)} - \Delta P_{(i)} C_{LL(i)}
\]

(D-13)

and the compliance function is

\[
C_{LL} = \frac{1}{E(BB_N)^{1/2}} \left[\frac{W+a}{W-a}\right]^2 \times \left[2.1630 + 12.219\left(\frac{a}{W}\right) - 20.065\left(\frac{a}{W}\right)^2 - 0.9925\left(\frac{a}{W}\right)^3 + 20.609\left(\frac{a}{W}\right)^4 - 9.9314\left(\frac{a}{W}\right)^5\right]
\]

(D-14)
Finally, creep-fatigue crack growth rates expressed as a function of $\Delta J_t$ for 10Cr are represented in Figure D-12 (the continuously cycled tests were calculated according to the elastic part of equation (D-10)).

![Figure D-12 Creep-fatigue crack growth rates expressed as a function of $\Delta J_t$ for 10Cr specimens. (The testing parameter can be found in Table D-2). The comparison between short and long-crack creep-fatigue crack growth rates by using $\Delta J_t$ as the unified correlating parameter is also presented.)](image)

In this figure, the potential correspondence by using the cyclic $\Delta J_t$ integral is also examined. With the trend lines available from the short-crack creep-fatigue crack growth tests (e.g. Figure 5-3), it was possible to compare them with the results from the long-crack creep-fatigue crack growth tests. This is shown in Figure D-12, where the curves denoting long crack growth tests and short crack growth tests (from Figure 5-3b) are plotted together. A good consistency could be observed, as the hollow red symbols overlap well with other curves. It should be noted that these two short crack growth specimens came from low strain range experiments (i.e. $\Delta \varepsilon_t = 0.5\%$ at 600°C and 625°C), where the cyclic stress-strain response was nearest to that in long crack growth tests. Certainly for short-crack creep-fatigue crack growth tests with a large strain range (i.e. $\Delta \varepsilon_t = 1\%$) or even with hold period, there would be a bigger difference between these two kinds of experiments.

**Discussion**

The theories and models for long-crack fatigue crack growth and creep crack growth have already been well established in the last century. The task involved in the current study relating to this topic was only to validate the recommended models in the ASTM standard with the round-robin test material (P91) and the own material (10Cr). As expected, the suggested models correlating with $\Delta K$ or $(C_t)_{avg}$ could reasonably predict the long-crack creep-fatigue crack growth behaviour for continuously cycled or dwell specimens (Figure D-9a and Figure D-11 for P91, whereas further investigation was required for 10Cr).

By comparing test results for P91 and 10Cr, a considerable creep-fatigue crack development advantage of the 10Cr steel with respect to the P91 steel was observed. No matter which
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correlating parameter was applied, cracks developed more slowly in the 10Cr steel. In fact, the testing durations of 10Cr samples were also much longer than those of P91 samples (for the same crack size).

As demonstrated by the P91 samples, in load controlled tests, when the hold time was long enough (e.g. 600s), the contribution of creep loading to crack development was much more significant than that of fatigue loading. Therefore, the fatigue crack growth term could even be neglected by using the dominant damage concept rather than the linear damage summation concept. In this sense, creep-fatigue interaction was not really considered.

On the other hand, for the 10Cr samples, there seemed to be a negligible contribution of the creep loading on the total crack growth rates under the current test set-up. This potentially inferred the applicability of $\Delta K$ or $\Delta J_t$ as the correlating parameters. More importantly, this promoted the idea of that these fracture mechanics based parameters could be employed as a bridge between long-crack creep-fatigue crack growth behaviour and short-crack creep-fatigue crack growth behaviour when creep damage was not significant (despite the fact that the governing damage mechanisms might be different).

With the evidence in Figure D-10 and Figure D-12 it is reasonable to conclude that there are indeed some similarities between short-crack and long-crack creep-fatigue crack growth behaviour, which can generally be demonstrated by the correlating parameters $\Delta K_{eq}$ or $\Delta J_t$. This in turn indicates that $\Delta K_{eq}$ and $\Delta J_t$ are effective crack tip parameters to describe elastic-plastic deformation (when the creep contribution is not significant). Another advantage of them is that $\Delta K_{eq}/\Delta J_t$ reduces down to $\Delta K/\Delta J_t$ when the plastic loading is absent (which can be more adaptive to the long crack growth circumstances), whereas the parameters for correlating short crack growth rates (e.g. $\Delta J_p$ or the SEDF) reduce to zero.

D.4 Investigation of microstructure

To examine how different loading conditions and geometries can influence microstructural substructures, post-test examination was also carried out for long-crack creep-fatigue crack growth testpieces (for comparison with Chapter 4). It was interesting to see the crack propagation paths in CT specimens and compare them with those in round-bar specimens in short crack growth tests.

**Crack propagation path**

As introduced in section D.1, this kind of creep-fatigue tests was carried out with relatively small peak loads to keep the testpiece in a predominantly elastically deformed condition. Due to the loading symmetry as well as the side grooves, the crack path was in principle kept within the pre-defined plane (in reality the crack profile was more complicated). One example is shown in Figure D-13, by an SE image and a BSE image.
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Figure D-13 SE image (top) and BSE image (bottom) of a long crack propagation path in a CT specimen for 10Cr.
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While the BSE signal is effectively obtaining sub-surface information, the brightness of the image is normally more uniform than an SE image. SE images are largely affected by surface roughness, where over-exposure or shadow can appear. Moreover, branch cracks can more easily be discerned in a BSE image, owing to the low scattering ability of oxides. Thus by examining both signals, a comprehensive view of the sample could be attained for this example.

Along the crack propagation direction, the profile of the major crack changed considerably. Unlike the previous example of short crack growth tests, the gap between cracked surfaces in this CT specimen was actually reduced at a larger crack depth. The reason behind this was apparently the accumulated inelastic deformation resulting from the positive load ratio. Near the initial machined notch, the cracking plane was almost straight, which indicated a typical elasticity-dominated fatigue fracture. With further crack development, this path became wavier and deviated from the pre-defined centre plane.

At the same time, an increasing number of branch cracks appeared (an enlarged view of the final crack tip is exhibited in Figure D-14). Similar to the previous short crack example, the branch cracks exhibited a ‘Y’ shape, which branched into finer cracks (which were all covered with oxide layers). Moreover, some of the branch cracks were able to connect to each other, forming a tangled loop structure. It was then even harder to discriminate the major crack.

![Figure D-14 Enlarged view of the final crack tip of Figure D-13, showing branch cracks filled with oxides. (Red rectangular area is for EBSD mapping).](image)

In order to find out whether these branch cracks were transgranular or intergranular, EBSD measurements were performed as illustrated in Figure D-15. This sampling area is identical to that in Figure D-14 highlighted by the rectangular zone with an arrow.
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Figure D-15 Illustration of transgranular branch cracks by (a) coloured map of IPF and (b) grayscale graph showing at least two prior austenitic grains.

As clearly indicated by the different colour tones in the coloured map of IPF (Figure D-15a), at least two main prior austenitic grains could be identified. They are marked in a grayscale graph (Figure D-15b) with arrows denoting parts separated by those branch cracks. Apparently, the cracking path was not following prior austenitic grain boundaries, exhibiting a transgranular cracking profile.

Formation of creep damage

When the deformation condition became critical (with hold time involved), creep damage could then appear during creep-fatigue loading. For the investigated materials, the physical creep damage was mostly found to be in the form of microcracks along prior austenitic grain boundaries. An example is shown in Figure D-16 for 10Cr, illustrating two damaged zones both at triple junctions (one in the middle, and the other at the left-bottom, indicated by the red arrows). The loading direction was marked by the blue arrows.

Judging from the appearance, it seemed that decohesion originated from the stress concentrated areas owing to large precipitates (diameter at around 1μm) and grain boundaries. (Or they could also be creep voids, except that when voids have grown to this size and coalesced, more voids should have appeared, which was not the case here). Theoretically, the dimensions of M_{23}C_{6} and MX precipitates are normally smaller than 0.1μm (in the as-received state) for this class of steel; and the maximum size of the Laves phase up to 1μm can only be observed after long term aging/creep. However, it can be found from the literature that some primary precipitates (Nb carbonitrides) as large as 1μm can also be present due to excessive grain growth during austenitisation. This might be the explanation for the relatively large dimension of precipitates here, but this speculation needs further analysis to verify).
Owing to the local irregularity, the boundaries immediately adjacent to the second-phase particles experienced high stresses and acted like crack tips. The developed microcracks effectively coalesced across several particles, and the joint microcrack had a larger dimension which might continue to develop along grain boundaries under further creep loading (marked as area 1 in Figure D-16).

On the other hand, although most of the noticeable points of microstructure have already been discussed in the main part of this thesis (Chapter 4), there were still some interesting features exhibited by the long-crack creep-fatigue crack growth test samples. Probably the most prominent feature for this type of sample was a growing fraction of creep damage along the cracking direction (which was much more obvious than that in short-crack creep-fatigue crack growth test samples), and this fact is illustrated by the case of 10Cr-CT04.

As displayed in Figure D-17a, this sample had a similar microstructure to the short-crack creep-fatigue crack growth test samples, thus repetitive statements are not made again here. However, by taking a closer look at the microstructure along the crack path, an increasing number of microcracks could be found.

In the remote area before the pre-crack (Figure D-17a), no observable microcracks could be found. This was easily understandable, since this part of the specimen was only subjected to small elastic loading and aging; in the areas along the crack (close to the cracking plane, Figure D-17b), a certain amount of cracked boundaries from precipitates emerged; in the areas adjacent to the crack tip (Figure D-17c), more microcracks had been developed, which were all at prior austenitic grain boundaries.

The occurrence of these microcracks was assumed to be associated with the loading state and testing duration (the loading direction is marked by the blue arrows in Figure D-17).
Figure D-17 Microstructures at different locations in 10Cr-CT04: (a) before the pre-crack (b) along the crack propagation path and (c) close to the crack tip.
Although the absolute load (as well as $K$ or $J$) was not as big as in the short-crack creep-fatigue crack growth tests, the load ratio was always positive in long-crack creep-fatigue crack growth tests (i.e. $R = 0.1$), which implied that the accumulated inelastic deformation in near-crack regions could not be fully reversed. Therefore, the concentrated boundaries constantly suffered from the stretching force and microcracks tended to form. The deeper the major crack, the more intensified the stress field would be. In addition, for this type of experiment the testing duration was relatively long (up to 3 months), which permitted enough time for creep damage to form. In this case (10Cr-CT04), the hold time was 600s, which was considerably longer than the ramping time of 4s. All those factors made the test similar to a real creep crack growth test and no wonder that such microcracks could come out, along with the propagation of the major crack. Hence, it was appropriate to conclude that load-controlled long-crack creep-fatigue crack growth tests were more prone to physical creep damage formation than strain-controlled short-crack creep-fatigue crack growth tests.

An examination of micro-grain diameter on this sample was also done by EBSD (in the same way as described previously in Chapter 4). The results are listed in Table D-4. With an increased crack depth, the corresponding magnitude of microstructural evolution was also growing. Together with Figure D-17, it can be concluded that for such testing conditions, microstructural damage occurs in two ways: sub-structure coarsening and micro-crack formation.

Table D-4 Measured micro-grain diameters at different crack depths for a 10Cr specimen (10Cr-CT04).

<table>
<thead>
<tr>
<th>10Cr</th>
<th>Tolerance angle: 2°</th>
</tr>
</thead>
<tbody>
<tr>
<td>CT04</td>
<td>Crack depth (mm)</td>
</tr>
<tr>
<td>$t_h = 600s$</td>
<td>Micro-grain size (μm)</td>
</tr>
</tbody>
</table>

D.5 Summary

Studies on the load controlled long-crack creep-fatigue crack growth experiments serve as a complementary part of this PhD project. A standard compact tension geometry was adopted for this type of load controlled test, which was performed in accordance with the requirements of the ASTM standard E2760 (where the methodology has been well established). After creep-fatigue deformation, some of the long crack (CT) testpieces were also carefully prepared for microstructural analysis. Finally, three fracture mechanics based models ($K$ type, $(C_t)_{avg}$ type and $J$ type) have been applied to describe long-crack creep-fatigue crack growth behaviour for P91 and 10Cr testpieces, including the comparison with short crack growth tests. To summarize, the most important conclusions in this appendix are:

1. Cracks propagate much faster in the P91 specimens than in the 10Cr specimens under the same testing conditions. In other words, the 10Cr steel displays a substantial creep-fatigue crack development advantage relative to the P91 steel at 600/625°C.
2. For P91 samples, crack growth modelling correlated with $\Delta K$ is in good agreement with that determined in the ASTM standard E2760 pilot study (Figure D-9a). In particular, the contribution of creep loading (tensile dwell) to crack development is much more significant than that of fatigue loading. Higher values of $(C_t)_{avg}$ are obtained in this analysis relative to the pilot study (Figure D-11).

3. For 10Cr samples, the influence of the 600s creep loading on the total crack growth rates was almost negligible under the adopted testing conditions (Figure D-9b). This also infers that the 10Cr steel is much more resistant to creep-fatigue crack propagation than the P91 steel. In this case, the correlating parameter $\Delta K$ is sufficient to describe the crack development for 10Cr specimens, regardless of the hold periods.

4. A good consistency was observed when comparing long-crack growth behaviour with short-crack growth behaviour by correlating crack growth rates with $\Delta K_{eq}$ or $\Delta J$, (Figure D-10 and Figure D-12). This indicates that although $\Delta K_{eq}$ (or $\Delta J_{eq}$) is not the best correlating parameters to precisely describe short-crack or long-crack growth behaviour, it can serve as a reasonable unified parameter for both short crack development and long crack development (under similar creep-fatigue deformation conditions).

5. The number of branch cracks is to an extent associated with the magnitude of inelastic deformation. For long crack growth tests, branch cracks only appear after the major crack has propagated to a certain depth (Figure D-13), i.e. when the inelasticity at the crack tip becomes significant. (For short crack growth tests, branch cracks can be generated already at the crack starter (section 4.1.2)).

6. There appeared to be some microcracks along the crack propagation path (Figure D-16 and Figure D-17) in long crack growth tests with the 10Cr steel. The primary micro-cracking mechanism seems to be decohesion of the prior austenitic grain boundaries, assisted by the accumulated inelastic deformation adjacent to second-phase particles (diameter of around 1μm). (On the contrary, creep cracking is limited to a very small extent even for the specimens with a long dwell period in short crack growth tests).
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