Microstructural alterations in bearing steels under rolling contact fatigue

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A dissertation submitted for the degree of Doctor of Philosophy
at the University of Cambridge, December 2017
Preface

This dissertation is submitted for the degree of Doctor of Philosophy at the University of Cambridge. The research work presented herein was conducted under the supervision of Dr P. E. J. Rivera-Díaz-del-Castillo in the department of Materials Science and Metallurgy, University of Cambridge, between October 2013 and May 2017.

This work is original to the best of my knowledge, except where acknowledgement and references are made to previous work. Neither this, nor any substantially similar dissertation, has been or is being submitted for any other degree, diploma or qualification at any other institution. This dissertation contains less than 60000 words.

Part of this work has been presented in the following publications:


H. Fu, W. Song, E. I. Galindo-Nava, and P. E. J. Rivera-Díaz-del-Castillo. Strain-induced martensite decay in bearing steels under rolling contact fa-


Hanwei Fu

December 16, 2017
Acknowledgement

I would like to express my sincere gratitude to University of Cambridge and SKF UTC for offering me this opportunity to pursue my PhD degree. My special thanks go to SKF for the financial support for both my research and my life in Cambridge.

I am deeply grateful to my supervisor Dr P. E. J. Rivera-Díaz-del-Castillo, who has always encouraged me whenever I encountered difficulties in my research. He has also provided important guidance on both scientific research and my career, and I have learned so much from him on being a materials scientist. I appreciate the help, advice and encouragement from Dr J. Lai, my industrial supervisor, who has been so nice to me. I would like to thank Professor H. K. D. H. Bhadeshia for his valuable advice throughout my PhD study. I also thank Dr Steve Ooi for his help with my experimental plans. I am deeply thankful to Dr E. I. Galindo-Nava for the inspiring discussions and his help with my modelling work, and to Dr Wenwen Song from Aachen University for her assistance with APT.

I would like to thank JeeHyun Kang who patiently answered all my questions at the beginning of my PhD. I would also like to thank my colleagues in SKF UTC including Adam Gola, Andrej Turk, Beriham Basha, Miles Sto-
pher, Wen Cui, Gael Guetard, Jakub Rydel, Peter Walker, David Bombac, Isaac Toda, Chris Amey and Richard Earp for their help and friendship. I particularly thank Miles Stopher and Neelabhro Bhattacharya for their help with TEM operation, and Adam Gola for his help with FIB.

I am indebted to my family for their support, specially my wife Xinyu Jiang, who has accompanied me to get through all the hard times.
Abstract

Bearings made of high carbon martensitic steels undergo rolling contact fatigue (RCF) after exposure to cyclic loading, and different types of microstructural alterations occur at the subsurface, signalling the onset of component failure. These microstructural alterations exhibit different properties compared to the surrounding matrix and thus can be revealed by chemical etching, with distinct dark-etching or white-etching contrast displayed under visible light. Despite extensive characterisation on the fatigue-induced microstructural features, their formation mechanism has been puzzling researchers over several decades. This research aims at revealing the nature of the microstructural alterations by postulating novel theory, which leads to the establishment of models capable of making predictions in agreement with experimental observations.

A study on 52100 bearing steel was conducted to show the effects of heat treatment on the microstructure and mechanical properties of the material. A repetitive push test was also performed to obtain the plastic response of steel under cyclic loading, the results of which were used as an important input in the modelling work.

In order to address the essential role played by carbon in the formation
of the microstructural alterations, a dislocation-assisted carbon migration theory was postulated. The theory takes into consideration the unique stress history of rolling contact, and by combining the Cottrell atmosphere theory with the Orowan equation, an expression for the carbon flux generated by gliding dislocations is obtained. The postulated theory was then used to describe the thickening of pre-existing precipitates in dark etching regions (DERs), the growth of lenticular carbides in white etching bands (WEBs) and the formation of dislocation cells in white etching areas (WEAs). The predictions made by the suggested models were compared with experimental observations, with good agreement obtained. The effects of RCF testing conditions on the formation rates of the microstructural alterations were also discussed.

It is suggested by this research that the observed microstructural alterations under RCF, i.e., DERs, WEBs, and WEAs all stem from the same mechanism, with the only difference being the migration distance of carbon. This research has produced powerful tools such as the microstructural alterations maps to appraise the extent of microstructural damage within a bearing component at any stage of its lifetime under any given RCF testing conditions, greatly reducing the necessity of carrying out a large number of time consuming full endurance RCF tests.
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### 7.4 Summary
Nomenclature

$\alpha$  Ferrite

$\alpha'$  Martensite

$\Delta_{HV}$  Hardness change in HV

$\Delta_\gamma$  Plastic shear strain amplitude in a stress cycle

$\Delta_\gamma_s$  Plastic shear strain amplitude at shakedown stage

$\Delta\tau_i$  Flow stress increment in dislocation cell interiors

$\Delta\tau_m$  Mean flow stress increment in dislocation cells

$\Delta\tau_w$  Flow stress increment in dislocation cell walls

$\Delta C_m$  Carbon content change in the matrix in at%

$\Delta L$  Dislocation glide distance per stress cycle

$\Delta x$  upper head displacement

$\dot{\gamma}$  Strain rate

$\dot{\rho}$  Rotational speed
\( \epsilon \)  \( \epsilon \) transition carbides

\( \eta \)  \( \eta \) transition carbides

\( \gamma \)  Shear strain

\( \lambda \)  Thickness of a fully developed white etching band

\( \rho_0 \)  Initial dislocation density in the material

\( \rho_i \)  Dislocation density in dislocation cell interiors

\( \rho_m \)  Mobile dislocation density

\( \rho_w \)  Dislocation density in dislocation cell walls

\( \sigma \)  Stress

\( \sigma_F \)  Fracture strength

\( \sigma_{VM} \)  von-Mises stress

\( \sigma_{Y0.2} \)  0.2\% offset yield strength

\( \sigma_Y \)  Yield strength

\( \sigma_{xx}, \sigma_{yy}, \sigma_{zz} \)  Normal stress components

\( \tau \)  Shear stress

\( \tau_a \)  Applied shear stress

\( \tau_{a}^{th} \)  Threshold applied shear stress for plastic deformation

\( \tau_{max} \)  Maximum shear stress
\( \tau_p \)  Peierls-Nabarro stress of body-centred cubic iron

\( \tau_{xy}, \tau_{xz}, \tau_{yz} \) Shear stress components

\( \tau_{yz}^{th} \) Threshold orthogonal shear stress for DER formation

\( \theta \) Cementite

\( \varepsilon \) Strain

\( \varepsilon' \) Strain calculated from upper head displacement

\( A \) Interaction energy between a carbon atom and a dislocation strain field

\( a \) Half long axis of rolling contact ellipse/radius of rolling contact circle/half width of rolling contact rectangle

\( a_\theta, b_\theta, c_\theta \) Lattice parameters of cementite

\( b \) Magnitude of Burgers vector

\( b_1, b_2, b_3 \) Coefficients for plastic strain amplitude fitting

\( b_e \) Half short axis of rolling contact ellipse

\( C_0 \) Initial carbon content in studied systems in at\% 

\( C_i \) Carbon content in dislocation cell interiors in at\%

\( C_{m0} \) Initial carbon content in the matrix in at\%

\( C_{V0} \) Initial carbon concentration in studied systems per unit volume
\(C_{Vb}\)  Carbon concentration in ferrite bands per unit volume

\(C_{VII}\)  Carbon concentration in carbon-enriched zones per unit volume

\(C_{VI}\)  Carbon concentration in carbon-depleted zones per unit volume

\(C_{Vi}\)  Carbon concentration in dislocation cell interiors per unit volume

\(C_{Vm}\)  Carbon concentration in the matrix per unit volume

\(C_{Vp}\)  Carbon concentration in pre-existing precipitates per unit volume

\(C_{Vw}\)  Carbon concentration in dislocation cell wall per unit volume

\(C_w\)  Carbon content in dislocation cell walls in at%

\(C_{V\theta}\)  Carbon concentration in cementite per unit volume

\(D\)  Diffusion coefficient of carbon in body-centred cubic iron

\(D_0\)  The maximum diffusion coefficient for carbon

\(d_r\)  Distance of dislocation cells from crack

\(E\)  The activation energy for carbon diffusion

\(e\)  The fundamental electric charge

\(F\)  Flight distance of an ion

\(f_i\)  Volume fraction of dislocation cell interiors

\(f_{p0}\)  Initial volume fraction of pre-existing precipitates
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$f_w$</td>
<td>Volume fraction of dislocation cell walls</td>
</tr>
<tr>
<td>$G$</td>
<td>Shear modulus</td>
</tr>
<tr>
<td>$h$</td>
<td>Thickness of dislocation cell walls</td>
</tr>
<tr>
<td>$h_f$</td>
<td>Final dislocation cell wall thickness</td>
</tr>
<tr>
<td>$J_d$</td>
<td>Dislocation-assisted carbon flux</td>
</tr>
<tr>
<td>$k$</td>
<td>Ellipticity</td>
</tr>
<tr>
<td>$k_B$</td>
<td>The Boltzmann constant</td>
</tr>
<tr>
<td>$l$</td>
<td>Crack length</td>
</tr>
<tr>
<td>$l_0$</td>
<td>Initial length of compression samples</td>
</tr>
<tr>
<td>$l_{LC}$</td>
<td>Thickness of lenticular carbides</td>
</tr>
<tr>
<td>$l_p$</td>
<td>Spacing of pre-existing precipitates</td>
</tr>
<tr>
<td>$m_{ion}$</td>
<td>Mass of an ion</td>
</tr>
<tr>
<td>$N$</td>
<td>Number of cycles</td>
</tr>
<tr>
<td>$N_c$</td>
<td>Number of cycles per revolution</td>
</tr>
<tr>
<td>$n_C$</td>
<td>Number of carbon atoms being captured by a mobile dislocation of a unit length in a stress cycle</td>
</tr>
<tr>
<td>$N_e$</td>
<td>Number of cycles where microstructural formation curves end</td>
</tr>
<tr>
<td>$n_{ion}$</td>
<td>Ionisation state of an ion</td>
</tr>
</tbody>
</table>
$P$  Applied compressive load

$p_0$  Maximum Hertzian contact pressure

$p_{\text{max}}$  Maximum contact pressure in compressive tests

$p_{\text{mean}}$  Mean contact pressure in compressive tests

$p_{\text{min}}$  Minimum contact pressure in compressive tests

$R$  The ideal gas constant

$r_{c, \text{max}}$  Maximum dislocation cell radius

$r_{c, \text{min}}$  Minimum dislocation cell radius

$r_c$  Radius of dislocation cells

$r_{p0}$  Initial half width of pre-existing precipitates

$r_p$  Half width of pre-existing precipitates

$T$  Temperature

$t$  Bearing operation time

$t_c$  Time interval between stress pulses

$t_{\text{ion}}$  Flight time of an ion

$U$  Electric potential

$v$  Velocity of dislocations

$V_{\text{II}}$  Volume of carbon-enriched zones
\(v_{\text{ion}}\) Velocity of an ion

\(V_i\) Volume of carbon-depleted zones

\(V_p\) Voltage at specimen tip

\(w\) White etching area thickness

\(w_{\text{max}}\) Maximum white etching area thickness

\(x, y, z\) Coordinates of rolling contact system

\(y_c\) Critical dislocation spacing for dislocation annihilation

1D One dimensional

2D Two dimensional

3D Three dimensional

APT Atom probe tomography

BCC Body-centred cubic

Cell\% Formation progress of dislocation cells

DER\% Formation progress of dark etching regions

DERs Dark etching regions

EDS Energy dispersive spectroscopy

FCC Face-centred cubic

FIB Focused ion beam
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
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<tbody>
<tr>
<td>HABs</td>
<td>High angle bands</td>
</tr>
<tr>
<td>LABs</td>
<td>Low angle bands</td>
</tr>
<tr>
<td>LCs</td>
<td>Lenticular carbides</td>
</tr>
<tr>
<td>OM</td>
<td>Optical microscopy</td>
</tr>
<tr>
<td>RCF</td>
<td>Rolling contact fatigue</td>
</tr>
<tr>
<td>ROI</td>
<td>Regions of interest</td>
</tr>
<tr>
<td>rpm</td>
<td>Revolution per minute</td>
</tr>
<tr>
<td>SDE</td>
<td>Strength differential effect</td>
</tr>
<tr>
<td>SE</td>
<td>Secondary electron</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>SPD</td>
<td>Severe plastic deformation</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>WEAs</td>
<td>White etching areas</td>
</tr>
<tr>
<td>WEB%</td>
<td>Formation progress of white etching bands</td>
</tr>
<tr>
<td>WEBs</td>
<td>White etching bands</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray diffraction</td>
</tr>
</tbody>
</table>
Chapter 1

Introduction

Bearings are amongst the most important engineering components employed in a variety of industries. The serving environment of bearings can be damaging for the material; such damage can be accelerated by high contact pressure and rotational speed [1], and sometimes elevated temperatures [2]. Under such circumstances, the components, although manufactured with high strength martensitic steels, are prone to failure due to rolling contact fatigue (RCF). Generally, the mechanisms for bearing failure are classified into two categories, surface-induced and subsurface-induced [3]. The former can be effectively eliminated with proper lubrication and mounting where bearings are operated under elastohydrodynamic (EHD) conditions, and are hence less likely to fail by surface-induced pitting [4]; the latter is almost unavoidable because of the unique Hertzian stress states of rolling contact under which conditions severe shear occurs at the subsurface [5]. Therefore, it is crucially important to obtain a better understanding of such subsurface-induced RCF, which has become the predominant failure mode for bearing operation nowa-
days [4].

In the past a few decades, numerous attempts [4] have been made to predict bearing life based on subsurface-induced RCF leading to empirical models (e.g. the Lundberg-Palmgren model [6] and the Ioannides-Harris model [7]) following Weibull strength theory. Modified computational models [8–10] have also been developed by taking into account microstructural factors. The life models are proposed mainly to deal with the nucleation and growth of subsurface cracks, either by assuming an unrealistic homogeneous material matrix or by simply combining initial microstructural parameters with damage accumulation. However, since 1946 [11], microscopic characterisation of fatigued bearing samples has revealed material instability when subjected to cyclic loading, manifested as microstructural alterations such as white etching areas (WEAs), dark etching regions (DERs) and white etching bands (WEBs) [2]. The development of these microstructural features covers all stages of bearing life and gradually alters the mechanical properties of the material. Under normal bearing operation conditions, WEAs emerge at early stages of bearing life, usually after $10^5$ cycles, adjacent to cracks initiated from inclusions and exhibiting higher hardness than the parent martensite; DERs arise and develop between $10^7$ and $10^8$ cycles; they are patch-like areas distributed at stress affected zones and are softer than the parent martensite; WEBs start to appear after as late as $10^9$ cycles with lenticular carbides (LCs) formed along deformation bands, the inhomogeneity introduced by which can weaken the microstructure [12]. As a matter of fact, the effects of these microstructural alterations have not been incorporated into any RCF life models because their formation mechanisms are still
unclear, let alone their quantitative description. This is the limitation of the present RCF life models and probably is the reason for the discrepancy between theoretical life predictions and experimental results. Therefore, more effort should be made into studying the occurrence of the microstructural alterations under RCF, and the complexity of these features calls for more physical insight into their formation process.

Extensive microscopic characterisation on the microstructural alterations has proved that the parent tempered martensite in bearings can decay and transforms into ferrite when subjected to cyclic rolling contact [13]. Given that martensite is formed in carbon steels as a result of a diffusionless process during quenching and that ferrite can accommodate much less solute carbon (∼0.02 wt%), it follows that the study on the martensite-ferrite transformation should be focused on the whereabouts of the excess carbon. There is no doubt that the microstructural alterations are stress-induced, but the migration of carbon also involves thermal diffusion. Despite a few attempts [14–16] to describe LC growth and DER formation by considering both stress and thermal effects, none of the models is capable of making accurate predictions under universal conditions. Actually, the formation rates for different types of microstructural alterations is found to be related to the distance of carbon migration, and the longer the migration distance is, the later the formation occurs. In WEAs, for instance, carbon redistribution occurs in dislocation cells which are tens of nanometre in size; in DERs, carbon travels between pre-existing precipitates with an average spacing of a few hundred nanometres; in WEBs, carbon migrates for micrometres. Figure 1.1 summarises the occurrence of different types of microstructural alterations at different RCF
stages by considering carbon migration distance. Therefore, this research is conducted on the establishment of a novel theory not only to explain the mechanism of the microstructural alterations under RCF, but also to produce accurate predictions on experimental observations.

Figure 1.1: Occurrence of different types of microstructural alterations at different RCF stages by considering carbon migration distance.

In Chapter 2, a literature review is carried out. Firstly, the properties of bearing steels and some basic RCF theories are introduced. Then the stress states under rolling contact with different contact geometries are calculated based on the Hertzian theory, which is crucial to the description of material plasticity in the following chapters. Finally, the reported work in the litera-
ture regarding microstructural alterations under RCF are critically analysed, summarising current understanding in this field and laying a foundation for the establishment of the new theory in this research. Experimental work on the material studied in this research, 52100 bearing steel, is presented in Chapter 3. Repetitive push tests are performed in order to obtain the plastic response of the material to applied load. A novel dislocation-assisted carbon migration theory, is postulated in Chapter 4 to account for the carbon redistribution under RCF; the theory is applied to precipitate thickening in DERs (Chapter 5), LC growth in WEBs (Chapter 6) and dislocation cell formation in WEAs (Chapter 7). The predictions made by the proposed models are compared to the experimental results gained from rolling contact fatigued samples from both this research and the literature, and good agreement has been achieved. The effects of testing conditions on the formation rate of these microstructural features are also discussed. In addition to traditional characterisation techniques (optical and electron microscopy), advanced atom probe tomography (APT) was also performed on a DER, considering the difficulty in detecting nano-sized precipitates in highly dislocated and magnetic martensite using transmission electron microscopy (TEM). Chapter 8 presents an overview on all the three types of microstructural alterations investigated in this research and postulates a universal theory for carbon redistribution promoted by contact fatigue. Remarks and suggestions for future work are presented in Chapter 9. The schematic in Figure 1.2 illustrates the work done in this research.

In summary, the study conducted in this research enhances the understanding of microstructural alterations under RCF and, for the first time,
makes accurate predictions on the evolution of the microstructural features as a function of temperature, rotational speed, contact pressure and number of cycles. The proposed theory and models lead to useful tools for bearing application, potentially having a significant impact on industry.
Chapter 2

Literature review

2.1 Introduction

A literature review is presented in this chapter. In order to comprehensively understand the fatigue process of bearings, the background of bearing steels is introduced in terms of composition and microstructure. Furthermore, analysis of the stress state under Hertzian contact is presented, and then the major microstructural alterations as a result of RCF, which are the focus of this research, are described in detail.

2.2 Bearing steels

In order to meet the challenges from the harsh environment of bearing operation, including a large number of repetitive stress cycles, very high rotational speed, and elevated temperature, bearing steels are expected to possess both high strength and outstanding fatigue resistance. To achieve the desired
mechanical properties, the microstructures of bearing steels are tailored via both composition and heat treatment. The carbon content in bearing steels usually ranges within 0.8 – 1.1 wt% with total substitutional solution less than 3 wt% [17]. Amongst all the steel grades for bearing applications, the most popular is 52100, also referred to as 100Cr6. The typical composition of 52100 type steel can be found in Table 2.1.

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Mn</th>
<th>P</th>
<th>Si</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.98-1.1</td>
<td>1.3-1.6</td>
<td>0.25-0.45</td>
<td>≤ 0.025</td>
<td>0.15-0.3</td>
<td>≤ 0.025</td>
</tr>
</tbody>
</table>

The as-received steel, for example 52100, is always in a spheroidised condition, with ~ 13 vol% spheroidised cementite particles distributed in a ferrite matrix, as such microstructure possesses excellent machinability [19]. After machining into bearing components, the steel is hardened in the martensitic or bainitic condition via heat treatment. Although bainitic microstructures with good ductility exhibit better resistance to crack propagation and hydrogen embrittlement [20], the hardness of bainite is too low to sustain the high contact pressure of bearings. Therefore, the majority of bearings in service display a martensitic microstructure. The standard martensitic heat treatment is described as follows. The steel is first austenitized to ~860 °C where 3 - 4 vol% of cementite is maintained whilst the rest is dissolved into the solid solution, followed by oil quenching to 60 – 70 °C to form martensite. Due to the high carbon content, the quenched martensite contains ~10 vol% retained austenite. During quenching, the temperature decreases so rapidly that carbon in solid solution does not have enough time to diffuse out of the resultant
body-centred tetragonal (BCT) Fe lattice which can accommodate much less carbon than the original face-centred cubic (FCC) Fe lattice of austenite [17]. This results in a carbon supersaturated martensitic matrix with a high level of internal stress. The carbon remaining in solid solution distorts the Fe lattice and the FCC - BCT transformation involves a volume change. Therefore, a large amount of dislocations is generated to accommodate the plastic deformation that accompanies such displacive transformation. Although the quenched martensite can reach a hardness of 900 HV, the microstructure is too brittle and contains too much residual stress. Therefore, the quenched martensite must be tempered to release the residual stress and to transform some retained austenite before application. Tempering of the bearing steel is always conducted at relatively low temperatures (160 – 220 °C) for 30 – 240 min. However, tempering softens the steel, because during tempering carbon can leave solid solution to form nanosized precipitates. These precipitates are identified to be transition carbides (ε and η) and cementite (θ) [21], the type, morphology and amount of which depends on the tempering temperature and time [22]. Figure 2.1 shows a typical microstructure of tempered martensite, consisting of globular primary cementite particles and nano-sized tempered carbides distributed in the plate-like martensite matrix.

Under cyclic rolling contact, the components of the microstructure function together to withstand the load. In this context, it is important to understand the complex stress state under rolling contact.
Figure 2.1: TEM image of martensitic microstructure of 52100 bearing steel. The steel was tempered at 160 °C for 2 h. Image adapted from [23].

2.3 Stress state under Hertzian contact

The stress state under rolling contact varies with the geometry of the contact bodies. Depending upon the rolling contact geometry, as shown in Figure 2.2, the contact surface can be an ellipse (ball bearing and ball-on-rod), a circle (flat washer) or a rectangle (roller bearing), and hence the resultant stress states within the contact bodies are different, which can be analysed by the classical Hertzian elastic contact theory [5].

Although the shape of the contact surface is determined by the contact geometry alone, the size of the contact surface is altered by the applied load
Figure 2.2: Contact surfaces for different rolling contact methods.

$P$. Under elliptical contact, for example, the contact surface is an ellipse with a long axis of $2a$ and a short axis of $2b$, and the ellipticity $k$, which is defined to be the ratio between the semi-axes, $k = \frac{b}{a}$, is a fixed value for a given contact geometry. In the context of Hertzian contact, the contact pressure $p_0$ is defined to be the highest stress on the contact surface. Circular contact is a special case of elliptical contact when $k = 1$, and $a$ then becomes the radius of the contact circle. Under line contact, the contact surface is a rectangle with a width of $2a$. It should be noted that the size of contact surface is dependent upon $P$ and material properties. Due to the symmetry of these contact surfaces, it is obvious that the stress levels maximize at the centres of the contact surfaces ($= p_0$), and gradually diminishes with
increasing distance to the centres. Figure 2.3 shows the stress distributions at the contact surfaces.

In terms of the stress states within the contact bodies, the stress components are calculated based on the Hertzian elastic contact theory [24]. The theory assumes complete elastic contact without sliding or traction. To address the most typical case for RCF testing, the calculation in this section is mainly carried out on elliptical contact. Before the calculation, it is necessary to define the coordinate axes first. For elliptical contact, as shown in Figure 2.4 (a), $x$ direction is defined to be along the long axis of the contact ellipse, $y$ axis to be along the short axis of the contact ellipse, and $z$ axis to be along the depth within the contact body, perpendicular to the contact surface. The three coordinate axes intersect at the centre of the contact ellipse. Figure 2.4 (b) illustrates the stress components in three dimensions.

During the analysis, the stress components are often calculated as ratios of $p_0$, while the coordinates are calculated as a ratio of $a$. Under different applied load $P$, these dimensionless ratios keep constant. The equations used for the calculation of the stress components at any given point in the contact body can be found in Appendix A.

Figure 2.5 (a) shows the distribution of the stress components along $z$ axis, right below the contact centre. It should be noted that the normal stress components ($\sigma_{xx}$, $\sigma_{yy}$, $\sigma_{zz}$) are all compressive, and their magnitudes gradually decrease with depth. The von Mises stress ($\sigma_{VM}$) and the maximum shear stress ($\tau_{\text{max}}$) are also plotted in Figure 2.5 (a). Equations (2.1) and (2.2) show how these two stresses are calculated. It is very important to note they both peak at the subsurface, indicating where the severest material
Figure 2.3: Contact surfaces for different rolling contact methods. (a) Elliptical contact for $k = 0.56$. (b) Circular contact. (c) Line contact.
Figure 2.4: (a) Defined coordinate axes under elliptical contact. (b) Stress components in three dimensions.

decay may occur.

\[
\sigma_{VM} = \frac{1}{4} \sqrt{\left(\sigma_{xx} - \sigma_{yy}\right)^2 + \left(\sigma_{xx} - \sigma_{zz}\right)^2 + \left(\sigma_{yy} - \sigma_{zz}\right)^2 + 6\left(\tau_{xy}^2 + \tau_{yz}^2 + \tau_{xz}^2\right)} 
\]

(2.1)

\[
\tau_{\text{max}} = \max\left(\frac{1}{2} |\sigma_{xx} - \sigma_{yy}|, \frac{1}{2} |\sigma_{xx} - \sigma_{zz}|, \frac{1}{2} |\sigma_{yy} - \sigma_{zz}|\right)
\]

(2.2)

It should also be noted that the shear stress components are zero along z axis, but non-zero elsewhere. Figure 2.5 (b) illustrates the distribution of stress components along y axis at the depth of \(z = 0.3a\). It can be seen along y axis, \(\tau_{yz}\) peaks at \(y = \pm 0.5a\) with the same magnitude but opposite directions.

When a ball rolls over a bearing inner ring or a rod, the \(yz\) plane is stressed most. The 2D stress state in \(yz\) plane is shown in Figure 2.6. The observed microstructural alterations under RCF are usually studied in this plane, and
therefore the calculated results are very important for the determination of stress components related to certain types of microstructural features.

2.4 Subsurface microstructural alterations under RCF

The microstructural alterations occur at the subsurface under RCF are usually studied on the circumferential section or axial section of a bearing inner ring. Figure 2.7 illustrates these two sections as well as the location of transformation zone where the microstructural alterations are observed. According to the definition of the coordinate system in section 2.3, the circumferential section corresponds to the $yz$ plane and the axial section corresponds to the $xz$ plane. The stress states in the planes determine the morphology of the observed microstructural features.
Figure 2.6: 2D stress distribution in $yz$ plane.
2.4.1 White etching areas (WEAs)

The formation of white etching areas (WEAs) has been widely reported in both martensitic and banitic bearing steels subjected to RCF. The white contrast of WEAs to the surrounding matrix can be revealed by nital solution under optical microscopes, which means the resultant microstructure is more resistant to the etchant. WEAs are frequently found adjacent to cracks initiated from non-metallic inclusions such as Al$_2$O$_3$ and MnS, although studies [25–28] have shown that primary carbides can also produce WEAs. Nevertheless, WEAs always appear in pairs, decorating cracks generated on both sides of an inclusion or carbide, but in most cases, a WEA only decorates one side of a crack. Figure 2.8 (a) shows a pair of WEAs observed from the circumferential section of a fatigued sample, exhibiting a special wing-type shape, and hence WEAs are also referred to as “butter-
flies” [29]. Another interesting feature of butteries is their formation angle, approximately $45^\circ$ to the rolling direction. This special orientation is suggested to be related to the maximum shear stress under Hertzian contact [30], despite some studies [31–33] indicating that butteries may be formed with smaller angles under high contact pressures. The occurrence of WEAs is generally believed to be a symptom of crack growth [34,35], which is to say, that cracks are formed prior to WEAs or that WEAs cannot be formed in the absence of cracks. This argument has been experimentally validated by Solano-Alvarez et al. [34, 36], who performed RCF tests on bearing steels with pre-introduced cracks and found full WEA decoration on these cracks. But those experimental results cannot eliminate the possibility that WEA formation and crack growth may actually promote each other, given the fact that WEAs are sometimes observed ahead of crack tips [37].

During the past decades, a great amount of characterisation work, mainly scanning and transmission electron microscopy, has been carried out to reveal the microstructure of WEAs. Generally speaking, a WEA consists of nano-sized ferrite grains/dislocation cells (Figures 2.9 (a) and (b)) [29,30], formed by the dissolution of carbides. It should be noted that some studies show complete carbide dissolution within WEAs [30], while some others indicate there to be finely distributed carbide fragments (Figure 2.9 (c)) [37, 38]. It was also found that the dislocation cells within WEAs vary from 10 nm to 100 nm in size. This variation may be related to the distance to the major crack as Grabulov et al. [30] argued that the cells are ultra fine near the crack and are much larger at the WEA-matrix boundary. Moreover, micro-cracks and micro-cavities were also detected in the WEA microstructure [29,33,39].
Figure 2.8: A pair of WEAs initiated from a Al$_2$O$_3$ inclusion. (a) OM image of the WEAs. (b) SEM image of one butterfly wing showing two types of boundaries: with a crack (boundary a) and without a crack (boundary b). Images adapted from [29].

In addition to two dimensional (2D) investigations, some studies on the three dimensional (3D) view of WEAs have also been carried out. Becker et al. [37] sliced a pair of WEAs by repeated mechanical material removal and optical microscopic observation, and for the first time revealed the size and shape of WEAs within the axial section. Later, Grabulov et al. [30] and Evans et al. [40] employed focused ion beam (FIB) to generate 3D tomographical images at selected WEA sections for reconstruction. This “slice and view” FIB method allows for a large number of image slices with high resolution, and hence reveals more microstructural information of WEAs. An example of a 3D WEA is illustrated in Figure 2.10. The result shows the distribution
Figure 2.9: Microstructure of WEAs. (a) EBSD map showing a cellular structure in a butterfly wing. The black particles represent carbides and the red spots (marked by the arrow) represent high misorientation zones. (b) Bright field TEM image of a WEA. The selected area diffraction pattern for area “A” is shown with the diffraction by cementite indexed. The typical diffraction pattern from the cellular structure is also presented as “B” showing a $(110)_\alpha$ ring. (c) Dark field TEM image from the cementite diffraction as marked in pattern “A” highlighting the carbide particles in the WEA. Image (a) adapted from [29]; images (b) and (c) adapted from [38].

of voids and cavities in WEAs, suggesting the growth and coalescence of the defects during WEA formation. The shape of the main crack is also revealed.

Figure 2.10: 3D reconstruction of a WEA. (a) SEM image of the investigated WEA. (b) 3D image of the WEA. (c) Shape of the main crack. Images adapted from [40].
Furthermore, the distribution of elements within WEAs was investigated at the atomic scale [38, 41]. With the atom probe tomography (APT) technique, as shown in Figure 2.11, carbon atoms were found segregated to dislocation cell walls, leaving carbon depleted interiors. It was argued that the enrichment of carbon at cell walls can stabilise the cellular structure. Also, some carbon clusters were detected and were described as partially dissolved carbides.

![APT image of carbon distribution in WEAs. Images adapted from [41].](image)

The origin of WEAs has been discussed for decades. Earlier theories stating that WEAs are heat induced were refuted by the clear evidence of plastic deformation in the microstructure. Grabulov et al. [30] postulated a low temperature recrystallization process assisted by plastic strain, but lacking direct experimental evidence. It is more likely that WEAs are directly caused by
plastic strain, because the major features of WEAs, cellular structure, carbide dissolution and carbon segregation to dislocation cell walls, strongly resemble the microstructures of pearlitic steels undergoing severe plastic deformation (SPD) such as cold drawing [42,43], high pressure torsion [44] and mechanical milling [45, 46]. The overwhelming plastic strain near cracks, as argued by many authors [29,34], stems from the repetitive beating and rubbing of crack surfaces due to cyclic rolling contact. Whilst the impurities (inclusions or primary carbides) where cracks are initiated, acting as stress concentrators can also add to localised plastic deformation. As for precipitate dissolution, there is no doubt that it is caused by the strong interaction between gliding dislocations and pre-existing carbides, and the latter, with such small size (∼ 6 nm in thickness), can be sheared and cut through if the local stress is sufficiently high [47]. Another striking phenomenon of WEAs formed in martensite is their distinct hardness increase. Nano-indentation tests [34,36] show the resultant hardness can be as high as 1200 HV (martensitic matrix ∼840 HV). This hardness increase is believed to owe to carbide dissolution, which injects excess carbon into the matrix and hardens it. This point of view was confirmed by the finding of softer WEAs formed in a carbide free bainite steel [48], where no carbide dissolution is involved under RCF. The finding also suggests that the white etching phenomenon of WEAs stems from dislocation rearrangement and carbon redistribution rather than the removal of carbides.
2.4.2 Dark etching regions (DERs)

Dark etching regions (DERs) exhibit a form of martensite decay at the subsurface of RCF samples. As the name indicates, the resultant microstructure displays dark contrast to the surrounding matrix under visible light after etching with nital, which indicates the formation of a new phase. DERs start to appear at the late stages of RCF, usually after $10^7$ cycles under normal RCF testing conditions [13]. First observed by Jones in 1946 [11], DERs were described as “mechanical troostite”, which were later confirmed to be ferrite. The dark etching effect is a sign of more interfaces in the microstructure and probably poor resistance to etch attack. Moreover, micro-indentation tests [49–51] show the dark etching phase is softer than the parent martensite, which can be expected considering much lower carbon solubility in ferrite ($\sim0.02$ wt%). Then the question arises as to the whereabouts of the excess carbon during the martensite-ferrite transformation, since apparently carbon is the key element that controls the formation of DERs.

Figure 2.12 shows the development of DERs with respect to the number of cycles ($N$). These DERs were observed from the circumferential sections of fatigued bearing inner ring samples. It can be clearly seen the DER phase consists of dark patches distributed in the matrix, and the microstructural change becomes severer at higher $N$. Interestingly, the distribution of these dark patches is inhomogeneous along depth. According to Figure 2.12 (a), starting from $\sim 100$ $\mu$m below the contact surface, the density of the dark patches of a DER increases with depth, and then peaks at a depth of $\sim200$ $\mu$m, followed by a gradual decrease when going deeper, and finally becomes...
nil at \(~500\ \mu m\). Although some studies \([49, 50]\) show the position where the highest density of the dark patches occurs goes deeper and the DERs become wider with increasing \(N\), this unique distribution within DERs is definitely related to the stress state under Hertzian contact, which also suggests that DERs are stress induced phase transformations, and the finding of threshold stresses \([49]\) under which DERs are eliminated confirmed the essential role played by stress. The occurrence of DERs softens the material, resulting in the loss of capability to withstand loads, and is thus detrimental to bearing life. Besides, the resultant softer phase is favourable to the formation of 30° and 80° white etching bands (WEBs), as shown in Figure 2.12 (c).

Although having been discussed for several decades, a plausible formation mechanism for DERs is still unavailable in the literature. Some authors argued the so-called DER ferrite results from the over-tempering of parent martensite \([12, 49]\), and the fact that DERs are never observed in the steels first tempered to a hardness lower than 700 HV \([51]\) supports this argument. However, this tempering process is definitely assisted by stress, as the temperatures of bearing operation are not sufficiently high to activate thermal growth of carbides. Swahn \textit{et al.} \([50]\) postulated that the excess carbon upon martensite-ferrite transformation gradually migrates to defects, especially dislocations, in the microstructure, but this requires an unrealistically high dislocation density of \(10^{17} \text{ m}^{-2}\) (typically it is \(10^{16} \text{ m}^{-2}\) for deformed martensite) and cannot explain the hardness decrease in DERs. Additionally, TEM investigation \([52]\) shows cellular structures in DERs, and if all the solute carbon resides around dislocations, then there should be a distinct hardness increase of the microstructure, like in WEAs, but the experimental results
Figure 2.12: OM images of DERs formed in the circumferential sections of fatigued 52100 bearing inner ring samples. (a) Sample run to $10^7$ cycles. (b) Sample run to $10^8$ cycles. The zoomed in image shows the nucleation of 30° WEBs. (c) Sample run to $10^9$ cycles. 30° and 80° WEBs are formed on the top of the DER. Images adapted from [50].

indicate the other way around. Therefore, apparently the carbon in solid solution leaves the matrix during the phase transformation, and is then captured by carbides. In this respect, the latest research on DERs by Kang et al. [16] suggests a dislocation-assisted carbide thickening mechanism with an emphasis on the formation of Cottrell atmospheres. According to the theory, gliding dislocations drag carbon atmospheres towards pre-existing carbides and assist carbide thickening, reducing the carbon content in the matrix. The postulated model managed to explain the material softening in DERs,
but the effects of stress, temperature and rotational speed could not be accurately predicted. Nevertheless, the focus on the interaction between gliding dislocations and solute carbon atoms seems to be a plausible approach to the describing the nature of such microstructural decay.

2.4.3 White etching bands (WEBs)

White etching bands (WEBs), which can also be revealed by nital solution, manifest the instability of the material at very late stages of bearing life. They are usually observed on the top of fully developed DERs, but the formation of DERs is not a necessary condition for WEB nucleation, as evidence shows WEBs formation in the absence of DERs in some steels that are softened by tempering to 670 HV [51]. Under normal rolling contact conditions, after $\sim 10^8$ cycles, a group of WEBs starts to appear at the depth of the calculated maximum shear stress with an inclination angle of $30^\circ$ to the rolling direction [13]. This group of WEBs is also referred to as low angle bands (LABs), in order to distinguish from another group of WEBs which appears after $\sim 5 \times 10^8$ cycles, inclined $80^\circ$ to the rolling direction and known as high angle bands (HABs). The appearance of these two groups of WEBs can be found from Figure 2.12 (c), and the relationship between LABs and HABs is schematically illustrated in Figure 2.13. An elegant attempt was made by Polonsky and Keer [15] to explain the unique directionality of LABs and HABs by studying the histories of applied shear stresses and normal deviatoric stresses. However, little is known about the the physics during WEB formation, much less the formation mechanism of the complex microstructu-
tural features.

Figure 2.13: Relationship between HABs and LABs under RCR. Image adapted from [53].

One of the most striking phenomena of LABs is the formation of lenticular carbides (LCs), lying along the boundaries of LABs. The typical morphology of LABs under OM is shown in Figure 2.14. It has been verified with TEM [12, 50] that the LABs with white contrast are actually carbide free ferrite bands with a cellular structure within. With increasing number of cycles, the length and width of LABs, as well as the thickness of LCs increases. A fully developed LAB can be 50 – 60 µm in length and ~10 µm in width [2], while the thickness of a LC can reach 1 µm [14]. The occurrence of LABs is detrimental to bearing’s resistance to damage, as proved by Martin et al. [12], who manually fractured a fatigued bearing inner ring sample and found that LC boundaries are favourable sites for crack propagation (Figure 2.15).

The driving force for LC formation is still unclear. Bush et al. [49] pos-
tulated a material extrusion-intrusion mechanism and argued that LCs originate from the deformation of spherical cementite particles. However, fully developed LCs are always much larger than any pre-existing cementite particles and the fact that LCs thicken with increasing number of cycles leads most researchers [14, 15, 50] to believe that LCs are actually precipitated during RCF. The excess carbon for LC precipitation, in this respect, should come from ferrite bands which are softer than the surrounding matrix [54], and this hardness drop owes to carbon depletion in ferrite bands. Swahn et al. [50]
argued that the energy increase caused by plastic deformation is the driving force for carbon flowing towards the LAB boundaries, while Buchwald and Heckel [14] attributed the outflow of carbon to both stress and carbon concentration gradients. According to their analysis, the hydrostatic pressure under Hertzian contact can be high enough to dissolve the precipitates in the matrix, resulting in a localised carbon concentration gradient. Under this concentration gradient, solute carbon diffuses towards the LAB ferrite band boundaries. The stress gradient also assists the outflow of carbon. In order to validate the proposed model, they carried out a number of RCF tests and measured the growth rate of LCs with respect to the number of cy-
cles. However, the calculation requires a subsurface temperature of \( \sim 300 \, ^\circ C \) to achieve the measured growth rate of LCs, despite the nominal operation temperature being only 100 \( ^\circ C \). Later, Polonsky and Keer [15] postulated that excess carbon is injected into the matrix due to dislocation annihilation in ferrite bands, and approximated the number of cycles \( (N_e) \) required to form a carbon free ferrite band of thickness \( \lambda \) with the following equation:

\[
N_e = \frac{\lambda^2 \dot{N}}{D},
\]

where \( \dot{N} \) is the rotational speed and \( D \) is the diffusion coefficient of carbon in \( \alpha \) iron. This model yields comparable predictions to the experimental observation by Buchwald and Heckel [14]. However, the proposed equation is rather approximate and does not take into consideration the effect of applied stress, which has been proven to have a considerable impact on the formation rate of LABs [55]. Nevertheless, the attempts made in the literature have laid the foundation for further modelling work on LAB formation.

As for HABs, they are much larger than LABs, while the resultant microstructure features are similar to those of LABs: cellular structure, carbide free and carbon free [50]. Although some researchers [50] believe a dense lamellar structure of LABs is necessary for the formation of HABs, experiment [55] shows HABs can appear prior to LABs when the contact pressure is very high (5.5 GPa). Moreover, Polonsky and Keer [15] analysed the stress history experienced by HABs and suggested a dislocation model for their formation, implying that HABs are mainly controlled by local stress state.
2.4.4 Formation of WEAs, DERs and LABs: a unified mechanism

The observed microstructural alterations, WEAs, DERs and LABs, although appearing at different stages of bearing life, show similarities that are worth noticing. Firstly, martensite is a thermodynamically unstable phase which tends to remove carbon from the solid solution and transform into ferrite. However, the operation temperature of RCF testing is usually too low to activate thermal diffusion of carbon. Therefore, the ferrite phases detected in all the three types of microstructural alterations should be stress induced. Moreover, the detected ferrite phases are always accompanied by dislocation cells, which is an indication of plastic deformation. Dislocation glide can be activated when local stress is beyond the yield limit of the steel, and the gliding dislocations tend to cluster to form a cellular structure, which is a thermodynamically more stable structure than forest dislocations. Secondly, during the martensite-ferrite transformation, carbon redistribution occurs. Carbon-depleted zones transform into ferrite while carbon-enriched zones vary with microstructural alteration types. For WEAs, the carbon enriched zones are dislocation cell walls as detected by APT and the carbon depleted zones are cell interiors. When carbides are dissolved, the excess carbon is injected into the solid solution, leading to distinct hardness increase in WEAs. For DERs, despite a cellular structure, the ferrite phase is softer than the parent martensite, indicating that carbon has left the solid solution. Therefore, the carbon-enriched zones are most likely to be pre-existing carbides which tend to continue growing, resulting in carbon depletion in the ma-
trix. For LABs, carbon is enriched at re-precipitated LCs, leaving carbon depleted ferrite bands with a decrease of hardness. To sum up, the process that all the three types of microstructural alterations have in common is strain-induced redistribution of carbon, and the whereabouts of carbon determine the observed changes in mechanical properties. The above analysis implies a possible unified mechanism that operates behind all the three types of microstructural alterations under RCF, and the key point is to solve the associated strain-induced carbon migration process.

2.5 Summary

In this chapter, the processing and resultant microstructure of the material used in this research has been reviewed, followed by an analysis of the stress state under Hertzian contact. Then the three types of subsurface microstructural alterations under RCF, WEAs, DERs and WEBs have been reviewed in detail. The phenomenology of the microstructural features was summarized and compared, along with critical discussions on their formation mechanisms. By comparison, the most significant similarity between the microstructural alterations, strain-induced carbon redistribution, has been highlighted. This is the key concept leading this research.
Chapter 3

Plasticity of 52100 bearing steels

3.1 Introduction

Before studying the microstructural alterations of bearing steels under rolling contact fatigue (RCF), it is necessary to understand the materials response to cyclic loading. The material studied in this research is 100Cr6 bearing steel. In this chapter, two types of heat treatment are applied, resulting in different microstructures for comparison. Both monotonic mechanical tests and cyclic fatigue tests are carried out, and the obtained materials parameters are vital for the modelling work in the following chapters.
3.2 Material and heat treatment

Table 3.1 lists the nominal composition of the steel used for the study in this chapter. The as-received samples were in a spheroidised condition with a hardness of $\sim 240$ HV30. During the spheroidisation process, the steel was first heated to 795 °C followed by rapid cooling to 750 °C, and was then continuously cooled to 675 °C at a rate not exceeding 6 °C/h. The resultant microstructure contains $\sim 13.4$ vol% cementite ($\text{Fe}_3\text{C}$) and $\sim 86.6$ vol% ferrite. Figure 3.1 shows an optical image of the microstructure of the as-received sample after etching with 2 % nital solution. A large amount of cementite particles with size ranging from 1 to 6 $\mu$m can clearly be seen distributed in the ferrite matrix.

Table 3.1: Nominal composition of the 100Cr6 bearing steel used in this chapter. All values are in wt % except * in ppm.

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Al</th>
<th>Ti*</th>
<th>Ca*</th>
<th>O*</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.96</td>
<td>0.34</td>
<td>0.20</td>
<td>0.007</td>
<td>0.002</td>
<td>1.44</td>
<td>0.05</td>
<td>0.22</td>
<td>0.011</td>
<td>20</td>
<td>2</td>
<td>7</td>
</tr>
</tbody>
</table>

The steel was hardened via heat treatment. Two types of heat treatment which are widely employed for martensitic bearing steels [18] are selected. As shown in Figure 3.2, for both types, the steel was first austenitized at 860 °C followed by oil quench (60 °C) and water quench (0 °C) to obtain a martensitic microstructure, whereas their tempering processes differ in time and temperature. For S1 the quenched martensite was tempered at 160 °C for 90 min while for S2 the quenched martensite was tempered at 220 °C for 240 min.
3.3 Microscopy

In order to study the microstructures of the steels after heat treatment, electron microscopic investigation was carried out. Block samples of S1 and S2 were first ground flat using SiC papers, and then polished to a mirror finish with diamond paste. 2 % nital solution was used to reveal the microstructures. Scanning electron microscopy (SEM) was performed under a FEI Nova NanoSEM 450 SEM at the secondary electron (SE) mode. Figures 3.3 (a) and (b) show the SE images of the microstructures of S1 and S2, respectively.

For both S1 and S2, large globular carbide particles of 0.2 – 0.8 µm are present. These particles are undissolved spheroidised carbides with a (Fe, Cr)$_3$C composition [56]. The size and amount of these carbide particles are usually not affected by subsequent low temperature tempering. However, the
distribution of the globular carbide particles may vary with regions of a steel sample due to inhomogeneous heating and cooling during steel-making and micro-segregation of the steel ingot [57]. This may be the reason why more globular carbide particles can be seen in Figure 3.3 (a) than in Figure 3.3 (b), although X-ray diffraction (XRD) shows that both samples contain the same amount of cementite (∼ 4 vol%). Nevertheless, tempering can alter the martensitic matrix. First of all, the carbon in the solid solution tends to be precipitated as carbides, which reduces the tetragonality of the Fe lattice [58]; also, it has been demonstrated by atom probe tomography (APT) [59] that the carbon in the solid solution of tempered martensite is strongly segregated to defects in the microstructure such as dislocations and plate boundaries. Secondly, tempering releases the residual stress of quenched martensite via dislocation rearrangement (mainly dislocation annihilation), which is manifested as plate growth. Comparing the microstructures in Figures 3.3 (a)
Figure 3.3: SE images of S1 (a) and S2 (b). Note the amounts of globular carbides are the same for both samples but very with regions.

and (b), it is observed that the martensite plates in S2 are coarser than in S1 due to higher tempering temperature and longer tempering time. Finally, tempering can result in the transformation of retained austenite (RA) into a phase with properties between ferrite and bainite [58]. For S1, according to the XRD analysis, about 10% of RA is still present while for S2, almost all the RA is eliminated. The carbon depletion in the solid solution and
dislocation annihilation caused by tempering softens the material, and this effect is basically positive correlated to tempering temperature and time.

The carbide precipitates formed during tempering are nano-sized and hence cannot be observed by OM or SEM. Therefore, transmission electron microscopy (TEM) was carried out. A FEI Tecnai F20 FEG TEM was used to characterise the microstructure of S2, and the results are shown in Figure 3.4.

In Figure 3.4 (a), a distinct plate-like martensitic structure is displayed. There is a large amount of needle-like carbide precipitates distributed in the interior of the martensitic plates. It seems that these precipitates grow along some preferred orientation such that they are roughly parallel to each other. The morphology of the precipitates in S2 agrees with that observed in S1 as reported by Barrow et al. [22, 23]. Figure 3.4 (b) shows the precipitates at a higher magnification, and a statistical analysis indicates their size to be $\sim 120 \, \mu m$ in length and $\sim 15 \, \mu m$ in width, with an aspect ratio of 8. Owing to higher tempering temperature and longer tempering time of S2, the average size of the precipitates in S2 is much larger than those in S1 ($\sim 50 \, \mu m$ in length and $\sim 7 \, \mu m$ in width, with an aspect ratio of 7). Besides, XRD analysis shows the volume fraction of the nano-sized precipitates in S2 is $\sim 10 \, \text{vol}\%$.

### 3.4 Monotonic mechanical tests on S1 and S2

To study the mechanical properties of S1 and S2, uniaxial tensile and compressive tests were performed on S1 and S2 with a Mayes 100 kN LCF me-
Figure 3.4: (a) TEM image of S2. (b) TEM image of S2 with higher magnification showing the morphology of precipitates.
chanical tester to obtain stress-strain curves at room temperature. As shown in Figure 3.5, the tensile specimens were made according to SKF standard (SS-EN 10 002-1 [60]) with the diameter of the middle cylinders being 5 mm and the length being 40 mm, and the compression specimens were cylinders of 3.1 mm in diameter and 5.3 mm in length. The size of the compressive specimens is much smaller than that of the standard specimens for other metallic materials, due to the high strength of such bearing steels and the limited load capacity of the tester.

![Figure 3.5: Tension and compression specimens.](image)

Figure 3.6 (a) shows the set-up of a tensile test. The specimen was fixed by two holders mounted at both heads of the tester. The lower head of the tester remained stationary while the upper head was able to move up and down controlled by a hydraulic system. The two hands of the strain gauge were attached to the centre of the specimen with rubber bands. Before the
test, a slight tensile load is applied to guarantee the specimen to be in contact with the holders. After the test started, the specimen was uninterruptedly pulled at a rate of 0.005 mm/s until final fracture without detectable necking developed. During the test, the strain was directly measured by the strain gauge and the tensile stress was calculated from the ratio between the measured load and the cross-sectional area of the middle cylinder.

Figure 3.6 (b) shows the set-up of the compression test. The end surfaces of the cylindrical specimen were ground to be flat and places in between two blocks made of quenched martensite with a hardness higher than 900 HV30, and a slight compressive load is applied before the test to fix the specimen. Due to the small specimen size, two 1 mm KFG-1N-120-C1-11 strain gauges produced by KYOWA were attached with super glue to opposite sides of the specimen. The average strain measured by the two strain gauges estimates the strain at the central axis of the cylinder in case the specimen is buckled during the test. After the test started, a compressive load was applied at a rate of 0.005 mm/s. The compressive stress during the test was calculated from the ratio between the measured load and the cross-sectional area of the specimen. All the compressive tests were carried out within a polymethyl methacrylate shield because the specimens were shot out upon final failure without any debris found.
Figure 3.6: Set-ups of monotonic mechanical tests. (a) Tensile test. (b) Compressive test. (c) Schematic of tensile test. (d) Schematic of compressive test.

Figure 3.7 shows the stress-strain curves of S1 and S2 under tensile and compressive loads. Each of the tests was repeated for at least three times and the most representative curve is shown. The mechanical property data of the steels are obtained from the stress-strain curves and are summarised in Table 3.2.
Figure 3.7: Results of monotonic mechanical tests.

Table 3.2: Mechanical property data of S1 and S2.

<table>
<thead>
<tr>
<th></th>
<th>$\sigma_{Y_{0.2}}$ (MPa)</th>
<th>$\sigma_F$ (MPa)</th>
<th>SDE (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1 – tension</td>
<td>885</td>
<td>1712</td>
<td></td>
</tr>
<tr>
<td>S1 – compression</td>
<td>3239</td>
<td>3839</td>
<td>2354</td>
</tr>
<tr>
<td>S2 – tension</td>
<td>1330</td>
<td>1984</td>
<td></td>
</tr>
<tr>
<td>S2 – compression</td>
<td>2911</td>
<td>3223</td>
<td>1581</td>
</tr>
</tbody>
</table>

According to the results, it is interesting that although harder than S2, S1 exhibits lower 0.2 % offset yield strength ($\sigma_{Y_{0.2}}$) under tension, whereas under compression, the yield strength of S1 is higher than that of S2. Similar phenomena are common for brittle materials such as martensitic and banatic steels [61–63]; this is because as the brittler the material is, the more sensitive to defects it is, and this effect is especially significant under tension where the tensile stress promotes crack opening. Therefore, the stress-strain curves of S1 and S2 under tension should be mainly controlled by crack development, and it is suggested that S1 is less resistant to tensile stress than S2. Nev-
Nevertheless, S1 outperforms S2 under compression due to crack closure effect of compressive stress, which also implies the stress-strain curves of S1 and S2 under compression should be mainly controlled by plastic deformation. This also explains why the steels undergo much higher strain before fracture under compression than under tension. The difference in the measured yield strengths between tension and compression is also referred to as strength differential effect (SDE) [64], which makes the material sensitive to stress mode. In the case of rolling contact, a high magnitude of hydrostatic compressive stress is present [5,24], suggesting that the mechanical properties of bearing steels measured from compressive tests are more representative; but under rolling contact, the residual stress at the subsurface can sometimes be tensile, and hence SDE must be taken into consideration in these regions.

3.5 Cyclic compressive tests on S2

The material response of S1 to cyclic loading has been extensively studied in the literature [65–67], so only S2 is presented in this section. The cyclic compressive tests were performed with the same tester on the specimens of the same type as used for the monotonic compressive tests (Figure 3.5). As shown in Figure 3.8 (a), the set-up of a cyclic test was also similar to that of the monotonic tests, with the only difference being just one strain gauge attached to the specimen. The cyclic compressive load was delivered by the upper head of the tester. The load was applied at 15 Hz following a sinusoidal pattern with a constant maximum load magnitude in each stress cycle controlled by a computer programme. The corresponding stress
history experienced by the specimen is illustrated in Figure 3.8 (b). A minimum compressive pressure ($p_{\text{min}}$) of 0.02 GPa was maintained throughout the test to avoid specimen slipping. During the test, the applied load, the strain measured by the strain gauge and the position of the upper head were recorded as a function of time. In order to study the material response to applied load, four maximum contact pressures ($p_{\text{max}}$) were selected for the tests: 1.0, 1.5, 2.0 and 2.5 GPa.

Figure 3.8: (a) Set-up of a cyclic compressive test. (b) Stress history during cyclic testing.

The strain ($\varepsilon$) experienced by the sample can be directly measured by the strain gauge, but due to the high frequency of stressing, the attachment between the strain gauge and the sample surface was gradually weakened which could result in the peeling off of the strain gauge. Sometimes the strain gauge could peel off at very early stages of the test, even at the first cycle. Therefore it is necessary to also calculate the strain using the position change ($\Delta x$) of the upper head via $\varepsilon' = \frac{\Delta x}{l_0}$, where $l_0$ is the initial height of the sample. However, the recorded $\Delta x$ includes not only the deformation of the
sample but also that of the two blocks holding the sample, which means the real strain of the sample, $\varepsilon$, is a fraction of $\varepsilon'$. Therefore, a correction factor of 0.45, which was obtained by comparing the $\varepsilon$ values and the corresponding $\varepsilon'$ values, was introduced, i.e., $\varepsilon = 0.45\varepsilon'$. The normal stress ($\sigma$) was calculated by dividing the normal load by the area of the sample.

Figures 3.9 (a) and (b) show examples of stress-strain hysteresis loops for one stress cycle with the strain measured from the strain gauge and calculated from the position change of the upper head, respectively. The loops were obtained from the sample tested at 5000 cycles with $p_{\text{max}} = 2.5$ GPa. For each loop, by drawing a horizontal line at the mean stress ($p_{\text{mean}} = \frac{1}{2}(p_{\text{min}} + p_{\text{max}})$), two intersection points can be obtained, and the strain difference between these two points is the plastic normal strain amplitude ($\Delta\varepsilon$ or $\Delta\varepsilon'$) in this stress cycle. The determination of the plastic shear strain amplitude is also illustrated in Figure 3.9.

![Figure 3.9: Hysteresis loops obtained by strain gauge (a) and the position change of the upper head (b).](image)

With increasing number of cycles, the hysteresis loops tend to become slimmer and slimmer, indicating a decrease in plastic normal strain. By
collecting $\Delta \varepsilon$ and $\Delta \varepsilon'$ values at different stages of the tests under different $p_{\text{max}}$ and plot them with respect to the number of cycles, the evolution of $\Delta \varepsilon$ and $\Delta \varepsilon'$ can be obtained, as shown in Figure 3.10, and obvious descending trends can be seen for all four $p_{\text{max}}$ values. According to classic plasticity theories [68,69], when $p_{\text{max}}$ is below the plastic shakedown limit, the material will eventually come to a plastic shakedown stage where the stress-strain curve follows a closed hysteresis loop with a constant plastic strain amplitude [66]. In order to estimate this amplitude, by referring to the analysis by Kang et al. [67] the descending trend of $\Delta \varepsilon'$ follows the relationship:

$$\Delta \varepsilon' = b_1 b_2 \log N + b_3, \quad (3.1)$$

where $b_1$, $b_2$ and $b_3$ are coefficients fitted to the experimental data.

Once the coefficients are determined, making $N \to \infty$ and considering the correction factor, $0.45 \times b_3$ becomes the normal plastic strain amplitude at the plastic shakedown stage. In the case of RCF, shear stress ($\tau$) and shear strain ($\gamma$) are more important for the formation of microstructural alterations. According to the von Mises criterion [70], the normal stress can be converted to equivalent shear stress via $\tau = \sigma/\sqrt{3}$, while the normal strain can be converted into shear strain via $\gamma = \sqrt{3} \varepsilon$. Therefore, the plastic shear strain amplitude at the shakedown stage of fatigue, termed $\Delta \gamma_s$ can be obtained:

$$\Delta \gamma_s = \sqrt{3} \times 0.45 \times b_3. \quad (3.2)$$

Figure 3.11 shows the measured evolution of $\gamma_s$ with respect to $\Delta \tau_{\text{max}}$ for S2 obtained in this study as well as for S1 obtained with the same testing
method by Kang et al. [67]. It can be seen that for both cases, $\Delta \gamma_s$ gradually increases with increasing $\tau_{\text{max}}$, and a linear equation is fitted to the data for generalization. For S2, the equation is:

$$
\Delta \gamma_s = 4.757 \times 10^{-5} \tau_{\text{max}} + 1.72 \times 10^{-5},
$$

(3.3)

where $\tau_{\text{max}}$ is in GPa. For S1, the equation is:

$$
\Delta \gamma_s = 2.105 \times 10^{-4} \tau_{\text{max}} + 1.777 \times 10^{-4}.
$$

(3.4)

The comparison in Figure 3.11 indicates that under cyclic loading, S2 un-
dergoes less plastic deformation than S1. This may be because the severer tempering on S2 leads to a higher level of dislocation annihilation such that the amount of mobile dislocation density in S2 is lower than in S1.

In the case of RCF, the magnitude of $\tau_{\text{max}}$ can be calculated from the Hertzian theory [5], and the material response of S2 can be estimated from equation (3.4).

![Graph showing the evolution of $\Delta \gamma_s$ with respect to $\tau_{\text{max}}$.](image)

Figure 3.11: $\Delta \gamma_s$ evolution with respect to $\tau_{\text{max}}$. The experimental data for S1 is obtained from [67].

### 3.6 Summary

100Cr6 bearing steel with 2 typical types of heat treatment (S1 and S2) has been investigated. The microscopic studies show distinct differences in the microstructures of S1 and S2. Owing to higher tempering temperature and longer tempering time, S2 develops coarser martensitic plates and larger precipitates than S1. Monotonic tensile and compressive tests were performed on
S1 and S2, and they both display significant SED. Under tension, S2 exhibits higher yield strength than S1 while under compression, S1 outperforms S2. Considering the compressive nature of rolling contact, the results obtained in the monotonic compressive tests can better account for the mechanical properties of the steels undergoing RCF. Furthermore, cyclic compressive tests were performed on S2, with the material response to cyclic loading obtained (Equation 3.3), and it has been found that under cyclic loading, S2 undergoes less plastic deformation than S1. The microstructural and mechanical parameters obtained in this chapter are of great importance and will be used as inputs for the microstructural alteration models in the following chapters.
Chapter 4

Dislocation-assisted carbon migration theory

4.1 Cottrell atmosphere

In BCC iron, a dislocation distorts the surrounding iron lattice and produces an elastic strain field around it. As first pointed out by Cottrell in 1948 [71], such strain field tends to attract interstitial atoms in the solid solution such as carbon and nitrogen for stress relief and thus results in an equilibrium atmosphere formed around the dislocation, which is also referred to as Cottrell atmosphere. The Cottrell atmosphere theory was later used by Cottrell and Bilby [72] to explain the yielding and strain ageing phenomena of carbon steels, obtaining good agreement between theoretical predictions and experimental results. Nevertheless, it was not until 1983 that Chang et al. [73] conducted the first direct observation of a Cottrell atmosphere using an atom probe, and validated Cottrell’s theory. With the development of
three dimensional atom probe tomography (3D APT) in recent decades, the inhomogeneous distribution of carbon in martensite has been widely studied [74–77] and it has been found that the carbon in the solid solution is always segregated at microstructural defects such as grain boundaries and dislocations where the formation of Cottrell atmospheres is playing an essential role. In the case of 100Cr6 bearing steel, given the resultant tempered martensitic microstructure which contains a large amount of dislocations and a high concentration of carbon in the solid solution, the observed RCF phenomena are highly likely to be associated with the interaction between dislocations and carbon.

4.2 Dislocation-assisted carbon flux under RCF

When a bearing inner ring is subjected to cyclic loading, RCF occurs as a consequence of micro-yielding, undertaken by the glide of mobile dislocations. However, dislocation gliding is not continuous during bearing operation as any fixed point in the fatigued region of the inner ring, as schematically illustrated in Figure 4.1 (a), experiences stress pulses with a time interval \( t_c \) when the balls roll over. A stress pulse plus the subsequent time interval forms a stress cycle. Within each stress pulse, the mobile dislocations are pulled away from their original carbon atmospheres and glide a distance \( \Delta L \) and then come to a stop until the advent of the next stress pulse after \( t_c \). This time interval allows carbon to migrate back towards those dislocations that just moved to form Cottrell atmospheres. This mechanism produces a carbon flux along with the dislocation flow, as schematically illustrated
in Figure 4.1 (b). Here the gliding dislocations act as vehicles to transport carbon atoms.

Figure 4.1: (a) Schematic of the stress history experienced by a fixed point in the stress affected region of a bearing inner ring during bearing operation. A stress pulse plus the subsequent time interval $t_c$ forms a stress cycle (b) Schematic showing the Cottrell atmospheres dragged by gliding dislocations within a stress cycle.

In order to quantify the magnitude of this dislocation-assisted carbon flux ($J_d$), it is first written as:

$$J_d = \frac{\rho_m \Delta L n_C}{t_c}, \quad (4.1)$$

where $\rho_m$ denotes the mobile dislocation density and $n_C$ denotes the number of carbon atoms being captured by a mobile dislocation of a unit length within each stress cycle. Although the magnitudes of $\rho_m$ and $\Delta L$ are difficult to estimate, the equation can still be calculated via other methods. According to the Orowan equation [78], the plastic shear strain rate $\dot{\gamma}$ is a manifestation...
of the glide of mobile dislocations with an average velocity $v$, expressed as:

$$\dot{\gamma} = b \rho_m v,$$

where $b$ denotes the magnitude of Burgers vector. In the case of RCF, $\dot{\gamma}$ is equivalent to $\frac{\Delta \gamma}{t_c}$ where $\Delta \gamma$ is the plastic shear strain per stress cycle, and $v$ equivalent to $\frac{\Delta L}{t_c}$. Therefore, the Orowan relationship can be converted to:

$$\Delta \gamma = b \rho_m \Delta L. \quad (4.2)$$

As for $n_C$, its expression can be found from the work by Cottrell and Bilby [72]:

$$n_C = 3 \left(\frac{\pi}{2}\right)^{\frac{1}{3}} \left(\frac{ADt_c}{k_B T}\right)^{\frac{2}{3}} C_{Vm}, \quad (4.3)$$

where $A$ is the interaction energy between a carbon atom and a dislocation strain field, $D$ is the diffusion coefficient of carbon atoms in $\alpha$-iron, $k_B$ is the Boltzmann constant, $T$ is the temperature and $C_{Vm}$ is the carbon concentration in the matrix per unit volume. For a bearing operated at a rotational speed of $\dot{R}$ (usually in rpm), $t_c$ can be calculated as:

$$t_c = \frac{1}{\dot{R}N_c}, \quad (4.4)$$

where $N_c$ is the number of stress cycles per revolution, which is determined by bearing geometry. Therefore, by combining equations (4.1), (4.2), (4.3) and (4.4) the final expression for $J_d$ becomes:

$$J_d = \frac{\Delta \gamma \dot{R} N_c}{b} \left[3 \left(\frac{\pi}{2}\right)^{\frac{1}{3}} \left(\frac{AD}{k_B T \dot{R} N_c}\right)^{\frac{2}{3}} C_{Vm}\right] \quad (4.5)$$

For bearings tested under various temperatures and contact pressures, the
variable $T$ can be directly measured from the fixed outer rings, whilst $\Delta \gamma$ can be estimated from other traditional fatigue testing methods. The values of the constants used in Equation (4.5) are listed in Table 4.1

Table 4.1: Values of the constants used in Equation (4.5).

<table>
<thead>
<tr>
<th>Constant</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$b$</td>
<td>0.2876 nm</td>
</tr>
<tr>
<td>$A$</td>
<td>$3 \times 10^{-30}$ N m$^2$ [72]</td>
</tr>
<tr>
<td>$D_0$</td>
<td>$6.2 \times 10^{-7}$ m$^2$ s$^{-1}$</td>
</tr>
<tr>
<td>$E$</td>
<td>80000 J mol$^{-1}$ [17]</td>
</tr>
<tr>
<td>$R$</td>
<td>8.314 J K$^{-1}$ mol$^{-1}$</td>
</tr>
<tr>
<td>$k_B$</td>
<td>$1.38 \times 10^{-23}$ m$^2$ kg s$^{-2}$ K$^{-1}$</td>
</tr>
</tbody>
</table>

Note $D_0$, the maximal diffusion coefficient for carbon, $E$, the activation energy for carbon diffusion, and $R$, the ideal gas constant are used to calculate carbon diffusivity in BCC iron at a certain temperature via $D = D_0 \exp(-E/RT)$ [17].

Equation (4.5) enables the quantification of the carbon flux induced by rolling contact. In the following chapter, the proposed dislocation-assisted carbon migration theory will be used as a powerful tool to model the formation of different types of microstructural alterations.
Chapter 5

Formation of DERs under RCF: characterisation and modelling

5.1 Introduction

Dark etching regions (DERs) are a major form of rolling contact fatigue (RCF). It occurs in bearing steels under excessive contact pressure (usually >3 GPa) [2]. Advanced characterisation has shown DER formation is in nature a martensite matrix decay process, with the consequent microstructure consisting of cellular ferritic grains and nano-sized precipitates [51–53]. The presence of a threshold stress under which DERs is not observed indicates that the microstructural decay is stress induced [49], and the appearance of dislocation cells also suggests plastic deformation in these regions. On the other hand, it has been experimentally proved through RCF tests on bearing samples that by increasing the operation temperature and contact pressure, DERs development can be accelerated [55,79], which suggests that both me-
chanical and thermal mechanisms operate during this process. The fact that the decaying matrix displays dark contrast when etched with nital is indicative of more carbide-matrix interfaces [80, 81]. A similar phenomenon has also been reported and explained in the context of “strain tempering” where the formation of Cottrell atmospheres plays an important role [16]. In this research, a multi-scale microscopic investigation on DERs has been carried out and a dislocation-assisted carbon migration model is employed to describe the formation of DERs. The modelling results are compared with the experimental observations from both the current search and the literature. The effects of modelling parameters on the predictions of DER kinetics are also discussed.

5.2 Experimental methods

5.2.1 Material and RCF testing conditions

Four through-hardened deep groove ball bearings of 6309 type were tested at SKF ERC for predetermined number of cycles to reproduce DERs at different formation stages. The geometry of a 6309 ball bearing is shown in Figure 5.1. The material of the samples is 100Cr6 with composition as listed in Table 5.1. A standard heat treatment was carried out on the material to achieve the required mechanical properties of the component: the samples were first quenched from the austenitization condition at 830 °C and then tempered at 160 °C for 90 min, followed by air cooling to room temperature. The resultant microstructure contains ~4 vol% spheroidised cementite particles
homogeneously distributed in a tempered martensitic matrix and the overall hardness is \( \sim 800 \) HV30.

Figure 5.1: Geometry of a 6309 ball bearing.

Table 5.1: Composition of 100Cr6 bearing steel (wt.%).

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>Ni</th>
<th>Mo</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.97</td>
<td>1.38</td>
<td>0.28</td>
<td>0.28</td>
<td>0.21</td>
<td>0.18</td>
<td>0.06</td>
<td>0.04</td>
</tr>
</tbody>
</table>

The RCF tests of the bearing samples were conducted on a SKF R2 RCF tester at a rotational speed of 6000 rpm. The applied radial load was 25 kN which resulted in a contact pressure of 3.2 GPa. The testing temperature was 83 °C, which was determined by measuring the outer ring temperature. Lubrication was carefully controlled during the tests to ensure an elastohydrodynamic operation condition where frictional sliding at the contact surface is eliminated. Figure 5.2 shows the schematic of a SKF R2 RCF tester. The samples were run for \( 2.6 \times 10^4 \), \( 2.6 \times 10^5 \), \( 2.6 \times 10^6 \) and \( 2.6 \times 10^7 \) revo-
lutions, respectively. As the number of cycles per revolution \( (N_c) \) for 6309 type bearings is 4.96, the actual numbers of stress cycles experienced by the samples upon the suspension of the tests were \( 1.2 \times 10^5 \), \( 1.2 \times 10^6 \), \( 1.2 \times 10^7 \), \( 1.2 \times 10^8 \) cycles, respectively. The tested bearing inner races were sent to the laboratory for further microstructural investigation.

![Figure 5.2: SKF R2 RCF tester.](image)

### 5.2.2 Microindentation and Microscopy

A bearing inner ring can be cut along the radial plane to show its axial section or through the middle of the inner ring width to show its circumferential section (Figure 2.7). After cutting, the surfaces of the sections to be investigated were carefully ground with SiC paper from P240 to P2500 to remove deep scratches, followed by polishing with 6 \( \mu m \) and 1 \( \mu m \) diamond
paste for a mirror finish. Finally, the surfaces were polished with 0.3 \( \mu \text{m} \) alumina suspension to remove the surface stress introduced during grinding and polishing. Microindentation tests were carried out using a Qness hardness tester at the circumferential sections of the samples because of the larger investigation areas, where rows of indents along the depth were applied to study hardness evolution. In order to obtain detailed and accurate hardness evolving trends, a Knoop indentation tip was employed. For each indent, the tip was loaded at 0.1 kg and held for 10 s before unloading. After the indentation, the computer automatically measured the long axes of the indents with a 650\( \times \) optical microscope to calculate the hardness values, and then directly converted the results from HK0.1 to HV0.1. For each indentation row, the indents started from 30 \( \mu \text{m} \) below the surface and extended down to a depth to 600 \( \mu \text{m} \), with the spacing between neighbouring indents of 30 \( \mu \text{m} \). At least 6 indentation rows were repeated on each sample for averaging.

After the microindentation tests, the samples were etched with 2\% nital to reveal the microstructure. The indents can thus act as markers of depth below the contact surface. The microstructural changes at the subsurface were characterised with a Leica optical microscope. Meanwhile, an FIB/TEM investigation was performed on the sample run to \( 1.2 \times 10^8 \) cycles. The TEM sample was prepared in an FEI Helios Nanolab SEM/FIB system and characterised with an FEI Technai Osiris TEM.
5.3 Experimental results

5.3.1 Optical microscopy/microhardness investigation

A preliminary OM investigation on the progress of the DER with increasing number of cycles was carried out at the axial sections of the samples. As the results shown in Figure 5.3, no DER can be observed at the early stage of RCF (1.2 $\times$ $10^5$ and 1.2 $\times$ $10^6$ cycles); whilst after 1.2 $\times$ $10^7$ cycles, dispersed dark patches start to appear at the subsurface, suggesting the onset of martensite decay, and the number density of the dark patches increases rapidly after 1.2 $\times$ $10^8$ cycles. Another noticeable feature of the DERs is the inhomogeneous distribution of these dark patches. According to the observation from Figure 5.3 (c) and (d), the dark patches are much denser in the middle of a DER than near the upper and lower edges, which is a strong indication of a shear stress dependence of DERs formation, considering a similar shear stress distribution from the Hertzian contact theory [5].

In order to quantify the evolution of dark patches with respect to depth, DER% is introduced to quantify the extent of martensite decay within a selected slice, which is defined as follows:

$$\text{DER\%} = \frac{\text{Area covered by dark patches}}{\text{Total area}}$$ (5.1)

DER% at a certain depth is determined by optical microscopy analysis using the software ImageJ. An example is shown in Figure 5.4 to illustrate the procedure of the method. According to the method, an optical image containing a part of a DER is magnified to 200× with an indentation marker.
Figure 5.3: OM images of the axial sections of the samples after etching. DER starts to appear after $1.2 \times 10^7$ cycles.

to pinpoint the depth of this part; the image is then converted into an 8-bit image to obtain the contrast with gray scale; finally, an appropriate threshold is chosen based on the level of gray associated to dark patches from the parent martensite, and then a slice parallel to the marker is selected to calculate the area fraction of DER ferrite. This method was applied to all the indents in the microindentation rows on the samples with DERs.

As for hardness measurements, it is worth noting that differences in the range of 10 – 20 HV can be considered equal as they lie within standard error values. Moreover, such scatter can be magnified with low indentation loading. Given that DERs have been observed in a wide range of steels regardless of initial hardness, the hardness change during DER formation is of
more importance than the absolute hardness values. Hence, in this research, hardness change ($\Delta HV$) is calculated for each indent by comparing it to the initial hardness obtained from the unaffected regions of the samples. The evolution of both $\Delta HV$ and DER% with respect to depth are summarised in Figure 5.5 (b) and (d). As neither significant hardness change nor microstructural evolution was detected in the samples run to $1.2 \times 10^5$ cycles and $1.2 \times 10^6$ cycles, the results are not shown here.

Based on the experimental results in Figure 5.5, several conclusions can be drawn:

- A DER is a mixture of decaying martensite (dark patches) and parent
Figure 5.5: Optical microscopy and micro-indentation on the circumferential sections of the bearing samples. (a) and (c) OM images of sample run to $1.2 \times 10^7$ and $1.2 \times 10^8$ cycles; (b) and (d) statistical data of DER% and $\Delta H V$ with respect to depth.
martensite formed at the subsurface of a bearing inner ring.

- With increasing depth from the upper edge of a DER, DER% increases first, and then peaks, followed by a gradual decrease to 0 at the lower edge.

- Negative $\Delta HV$ values at the subsurface indicate material softening in the DERs, and the evolution of the extent of softening with respect to the depth is strongly correlated to that of DER%.

- With increasing number of cycles, both DER% and the extent of softening increase at all depths as a consequence of further consumption of parent martensite.

For a fatigued bearing inner ring sample, the only variable along depth is the stress, especially the shear stress. Hence the evolution of DER% and corresponding $\Delta HV$ indicates a stress-induced mechanism for DER formation. The OM images in Figure 5.5 (a) and (c) show the early and late stages of DER development, respectively. By comparing the depth range of the DERs with the corresponding distribution of the orthogonal shear stress ($\tau_{yz}$) calculated from the Hertzian theory [5], as shown in Figure 5.6, it is obvious they both exhibit a similar evolving trend. Furthermore, a threshold orthogonal shear stress ($\tau_{yz}^{th}$) can be indexed from the DERs formation range. This critical stress can be interpreted as the onset of microyielding of this steel under RCF, for this type of steel, 0.56 GPa. Therefore, it can be expected if the contact pressure ($p_0$) increases, the formation range of DERs will be larger, and the position where DER% or $\Delta HV$ peaks will move deeper at the subsurface.
Figure 5.6: Comparison between the distribution of $\tau_{yz}$ and the formation range of DERs. The threshold stress $\tau_{yz}^{th}$ can be indexed for this material.

5.3.2 FIB/TEM investigation

In order to further study the nature of the microstructural alteration occurring during DER formation, a FIB/TEM investigation was carried out on the longest run sample. In Figure 5.7 (a), the axial section of the sample was rotated clockwise to $90^\circ$ such that the cross section of the TEM sample to be milled by FIB coincide with the circumferential section. As shown in Figure 5.7 (a) and (b), the DER displays white contrast under SEM, which indicates more interfaces within these microstructural features. The decayed martensite patches seem to be randomly shaped and distributed without any preferred orientation. The location of the TEM lamella was chosen to be the middle of the DER, $\sim 250 \mu m$ below the center of the groove where both DER% and $|\Delta HV|$ are maximised. After lifted out from the sample, the TEM lamella was then welded onto a tooth of a copper grid, as shown in Figure 5.7 (d), followed by final milling to the thickness of 100 nm.

Figure 5.8, 5.9 and 5.10 show the TEM images of the DER. An overall microstructure in the DER is shown in Figure 5.8. Micro-bands are found
Figure 5.7: FIB procedures for preparing a TEM lamella in the DER. (a) SEM image of an axial section of the sample, where the curve on the right hand side is the contact surface. Note the DER appears white contrast under SEM. (b) Pt protection location prior to FIB milling. (c) Top section of the lamella milled by FIB. (d) Cross section of the TEM lamella welded on a copper grid tooth after final milling.

in the matrix, and the initial martensite plates become unclear under RCF. Large cementite particles are distributed in the matrix and are seemingly
not affected by the breakdown of the matrix. Figure 5.9 shows the deformed matrix at a higher magnification, and cellular structures can be found. This is evidence of plastic deformation and dislocation rearrangement, which rules out the argument that DERs stem from tempering caused by subsurface temperature rise. Figure 5.10 shows the microstructure of the microbands, which also exhibit cellular structures but with identical crystal orientation inside. These micro-bands are possible nucleation of white etching bands (WEBs), considering a large amount of reports showing WEBs start to appear at the late stage of DER formation.

Figure 5.8: Overall observation of the lemella.
5.4 DER formation model

DERs in the form of inhomogeneously distributed dark patches appear at the subsurface of bearing inner rings, the region where the orthogonal shear stress ($\tau_{yz}^{th}$) exhibits a similar distribution. The initial tempered martensite microstructure of the bearing samples contains nano-sized precipitates, namely temper carbides, evenly distributed within a martensitic matrix supersaturated with carbon [22]. From a thermodynamic point of view, the solute carbon tends to cluster and form carbides given that ferrite phase can only accommodate 0.02 wt% carbon, although the kinetics are suppressed at the bearing operation temperatures. However, according to the analysis in
Chapter 4, the migration of carbon can be activated with the assistance of gliding dislocations when cyclic loading is present. In the case of DERs, the resultant softer ferrite phase containing less carbon exhibits a different etching effect compared to the surrounding matrix, and consequently appears dark. The most possible residences for the excess carbon are the nano-sized precipitates formed during steel tempering, which tend to consume carbon for further growth. Therefore, a model is established based on the dislocation-assisted carbon migration theory to describe the redistribution of carbon during DER formation.

Figure 5.11 (a) illustrates a typical microstructure before DERs formation, where a large number of carbide precipitates with favourable orienta-
Figure 5.11: Schematic showing DER formation mechanism. (a) Microstructure of tempered martensite before DER formation. (b) Thickening of pre-existing precipitates caused by dislocation-assisted carbon migration.

Precipitations are distributed in the martensitic matrix. Large globular M₃C carbides are also present. As shown in Figure 5.11 (b), the nano-sized precipitates are taken as thin plates with a half width \( r_p \), the morphology of which agrees with the characterisation by TEM [22, 23]. During DER formation, the carbon transported by gliding dislocations contributes to the thickening of a precipitate from its both sides. Therefore, there exists a carbon flux equilibrium at either proceeding precipitate-matrix interface, being \( \frac{dr_p}{dt} (C_{Vp} - C_{Vm}) = \frac{1}{2} J_d \), where \( t \) denotes the cycling time, and \( C_{Vp} \) and \( C_{Vm} \) denote the carbon concentration in the precipitates and in the matrix, respectively. The expression of \( J_d \) is given by Equation (4.5), and hence the kinetics of precipitate thickening can be obtained as follows:

\[
\frac{dr_p}{dt} (C_{Vp} - C_{Vm}) = \frac{1}{2} \frac{\Delta \gamma \dot{R} N_c}{b} \left[ 3 \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{AD}{kT \dot{R} N_c} \right)^{\frac{2}{3}} C_{Vm} \right].
\]  

(5.2)

Assuming the spacing between the precipitates to be \( l_p \), a carbon mass con-
servation equation always holds for the system:

\[ l_pC_{V0} = 2r_pC_{Vp} + (l_p - 2r_p)C_{Vm}, \quad (5.3) \]

where \( C_{V0} \) is the initial carbon concentration of the system. Equation (5.3) leads to the evolution of \( C_{Vm} \) as follows:

\[ C_{Vm} = \frac{l_pC_{V0} - 2r_pC_{Vp}}{l_p - 2r_p}. \quad (5.4) \]

It can be expected with the preceding of precipitate thickening, the carbon concentration in the matrix gradually decreases until all the carbon is consumed.

By solving equations (5.2) and (5.4), the precipitate thickening process during DER formation can be calculated.

5.5 Modelling results and discussion

5.5.1 Determination of modelling inputs

The results of the proposed model depend upon inputs, such as material parameters and RCF testing conditions. Before the calculation, the initial microstructural information before DERs formation must be confirmed first. Referring to the work by Barrow et al. [22] and Kang et al. [16], the average half width of the precipitates \( r_{p0} \) for the steel investigated in this research was measured from TEM images to be \( \sim 7 \) nm, and the volume fraction of the precipitates \( f_{p0} \) was quantified by XRD to be \( \sim 6.7 \) vol\%. Hence, the
spacing between the precipitates can be approximated as 
\[ l_p = \frac{2r_p}{f_{p0}} \]
being \( \sim 209 \) nm. The total carbon content \( (C_0) \) of the system was calculated by Kang et al. [16] to be 3.1 at\%, which can be converted to carbon concentration through 
\[ C_{V0} = \frac{2C_0}{a_m} \]
where \( a_m \) is the lattice parameter of BCC ferrite, equal to the magnitude of Burgers vector. Besides, the carbon concentration within the precipitates, if assuming \( \theta \) type carbides, is calculated via 
\[ C_{Vp} = \frac{4}{a_\theta b_\theta c_\theta} \]
where \( a_\theta \), \( b_\theta \) and \( c_\theta \) are the lattice parameters of cementite equal to 0.45156 nm, 0.50837 nm and 0.67297 nm, respectively. Therefore, the initial carbon concentration in the matrix can thus be obtained using equation (5.4) when \( r_p = r_{p0} \), and if converted to atomic percentage, \( C_{m0} = 0.83 \) at\%. During the subsequent precipitate thickening process, \( r_p \) will gradually increase by consuming carbon in the solid solution, leading to carbon depletion in the matrix.

As for the testing conditions, \( T \), \( \dot{R} \) and \( N_c \) can be obtained from RCF tests as model inputs, but \( \Delta\gamma \) cannot be directly measured from rolling contact due to the complex stress state. Nevertheless, the material response to applied load under RCF is comparable to that under other traditional fatigue testing conditions. As shown in Figure 5.6, the plastic flow due to RCF is mainly attributed to the orthogonal shear stress component \( (\tau_{yz}) \), while the variation of \( \Delta\gamma \) under various stress levels is generally considered to be a function of the ratio between the applied shear stress and threshold shear stress [82,83]. In this respect, an equation is fitted for the experimental data taken from the work by Hahn et al. [66], who performed cyclic torsion tests under different applied stress on 100Cr6 bearing steel with the same heat treatment with this research. The fitting result is presented in Figure 5.12. Note \( \Delta\gamma \) is plotted
against $\frac{\tau_a}{\tau_{th}}$, and the fitted equation is $\Delta \gamma = 0.0003(\frac{\tau_a}{\tau_{th}} - 1)^3$. Likewise, the material response under RCF can be expressed as:

$$\Delta \gamma = 0.0003\left(\frac{\tau_{yz}}{\tau_{th}} - 1\right)^3.$$ (5.5)

Figure 5.12: Estimation of $\Delta \gamma$ as a function of $\tau_a/\tau_{th}$ fitted from the cyclic torsion data by Hahn et al. [66], and the equation follows a power law.

The proposed model yields precipitate thickening and carbon depletion in the matrix with respect to the number of cycles. It is noticeable the fraction of carbon leaving the matrix compared to the initial state, $\frac{C_m - C_{m0}}{C_{m0}}$, accounts for the extent of DER formation, which is directly proportional to DER%. This enables the outputs of the model to be compared with experimental observations. On the other hand, the model can also be employed to predict the mechanical property change, mainly the hardness change ($\Delta HV$), of the material during DER formation. As a DER is mixture of softer ferrite patches and the parent martensite, the resultant hardness at any position of a DER should be the mean hardness depending upon local DER%. According to the literature [49, 51], the hardness decrease for a fully developed DER in such
Steel is about 150 HV. Hence, the decrease of hardness within a DER mainly stems from the decrease in carbon content in the matrix. Following the rule of mixtures, \( \Delta HV = -150 \text{DER}\% = \frac{-150 \Delta C_m}{0.83 \text{ at}\%} \), which yields:

\[
\Delta HV = -180 \Delta C_m \text{ (at\%).} \tag{5.6}
\]

This relationship is plotted in Figure 5.13.

![Graph showing the linear relationship between \( \Delta HV \) and \( \Delta C_m \) obtained from the rule of mixture.](image)

**Figure 5.13**: Linear relationship between \( \Delta HV \) and \( \Delta C_m \) obtained from the rule of mixture.

### 5.5.2 Modelling results

An example of modelling results at the centre of a DER is shown in Figure 5.14. The curves were calculated using Equations (5.2) and (5.4), with RCF testing conditions: \( p_0 = 3.2 \text{ GPa} \) (\( \tau_{yz, \text{centre}} = 0.79 \text{ GPa} \)), \( T = 83 ^\circ \text{C} \), \( \dot{R} = 6000 \text{ rpm} \), \( N_c = 4.96 \). From Figure 5.14 (a) as the DER develops, the precipitates thicken with increasing number of cycles and, correspondingly, the carbon content in the matrix decreases, as Figure 5.14 (b) indicates. This process is completed when \( C_m \) reaches 0 at\%. The outputs of the model can be used...
to calculate DER% and ∆HV.

Figure 5.14: An example of modelling results at the centre of a DER with RCF testing conditions: (a) Evolution of precipitate thickness. (b) Evolution of carbon content in the matrix.

Figure 5.15 (a) and (b) present comparison between the modelling results of DER% and ∆HV with experimental observations in this research, and very good agreement can be achieved for both cases (1.2 \times 10^7 and 1.2 \times 10^8 cycles). It should be noted in each sample, the only variable along depth that results in the variation of the observables is \( \tau_{yz} \). Hence the proposed model is capable of correctly predicting DERs development under various shear stress levels.

The proposed model is also employed to predict the reported data under various RCF testing conditions from the literature. In Figure 5.16 (a), the evolution of DER% at the centre of DERs formed in the inner ring samples suspended at different number of cycles was reported by Bush \textit{et al.} [49]. The RCF tests were performed on 3208 type ball bearings at 2000 rpm, and according to the geometry of the samples, \( N_c = 5.32 \). The operation temperature was about 30 °C, and two contact pressures were utilised, 3.2 and 3.6 GPa. The model makes good predictions for both cases. In Figure
Figure 5.15: Comparison between the modelling results with experimental observations in this research for the samples run to $1.2 \times 10^7$ and $1.2 \times 10^8$ cycles. (a) DER%. (b) $\Delta$HV.

5.16 (b) and (c), predictions are made for $\Delta$HV along depth. The sample in Figure 5.16 (b), as reported by Lund [51], was a 6309 type ball bearing inner ring tested under 3.27 GPa, at 6000 rpm ($N_c = 4.96$), with the operation temperature being 60 $^\circ$C, and suspended at $10^9$ cycles. The calculated $\Delta$HV profile agrees with the measurements. The sample in Figure 5.16 (c) was one of the tests shown in Figure 5.16 (a), tested under 3.2 GPa and suspended at $1.2 \times 10^9$ cycles. Two sets of $\Delta$HV data were obtained from microindentation and residual stress measured by XRD. The measurements from XRD exhibit smoother evolution, and the predicted curve agrees better with it, although the measurements by microindentation indicate more softening effect than predicted at the centre of the DER.

5.5.3 Parametric study

The effects of testing conditions on the formation of DERs are presented in Figure 5.17. All the curves are calculated for the centre of the DERs.
Figure 5.16: Employment of the proposed model to the experiments from the literature. (a) Comparison between the calculated DER% at the centre of DERs under \( p_0 = 3.2 \) and 3.6 GPa and the data by Bush et al. [49]. (b) Comparison between the calculated ∆HV and the data by Lund et al. [51]. (c) Comparison between the calculated ∆HV and the data from both microindentation and XRD by Bush et al. [49].

As shown in Figure 5.17 (a), DER development is accelerated with increasing operation temperature. This is because DER formation is controlled by
dislocation-assisted carbon migration and, under higher temperatures, according to Equation (5.2), carbon diffusivity is increased, and therefore more carbon can be captured by gliding dislocations within a cycle (higher $n_C$), resulting in higher carbon flux. Higher stress levels, on the other hand, promote carbon flux indirectly through higher $\Delta \gamma$, which causes severer dislocation glide. Accelerated DER formation curves are presented in Figure 5.17 (b). However, higher rotational speeds delay the formation of DERs. As $t_c$ is inversely proportional to $\dot{R}$, higher $\dot{R}$ results in lower $t_c$, which means that less carbon is captured by dislocations within a stress cycle, leading to lower carbon flux (as shown in Figure 5.17 (c)). The predicted acceleration and deceleration effects of testing conditions agree with the literature [13], and the proposed model can be used to quantify these effects.

The establishment DERs formation model is crucial for industry, as the accurate prediction of DER% under various testing conditions can reduce the necessity of a large amount of time consuming full endurance RCF tests. Also, the mechanical property evolution of the material during DER formation can be predicted by combining the model with Equation 5.6, which is of importance for life predictions of bearing components. From the analysis in Figure 5.17, the main conditions that cause significant shifts of the DERs formation curves are $p_0$ and $T$, while $\dot{R}$ with the magnitude in the range of normal operation conditions is not a predominant parameter. In this respect, 3D maps for $\Delta HV$ are obtained with $p_0$, $T$ and $N$ as variables, as shown in Figure 5.18 (a). The $\Delta HV$ surfaces for $N = 10^6$, $10^7$, $10^8$ and $10^9$ cycles are illustrated, and the corresponding contour plots are presented in Figure 5.18 (b), (c), (d) and (e). With the proposed maps, $\Delta HV$ for any given testing
Figure 5.17: Effects of RCF testing conditions on the formation of DERs. (a) DERs development curves calculated under different $T$ with other testing conditions constant ($p_0 = 3.7$ GPa, $\dot{R} = 6000$ rpm, $N_c = 4.96$). (b) DERs development curves calculated under different $p_0$ with other testing conditions constant ($T = 80$ °C, $\dot{R} = 6000$ rpm, $N_c = 4.96$). (c) DERs development curves calculated under different $\dot{R}$ with other testing conditions constant ($T = 80$ °C, $p_0 = 3.7$ GPa, $N_c = 4.96$).
conditions can be easily indexed.

Figure 5.18: Maps for mechanical property evolution during the formation of DERs. (a) 3D ΔHV maps for $N = 10^6$, $10^7$, $10^8$ and $10^9$ cycles. (b), (c), (d), (e) Contour plots for the 4 surfaces in (a).
5.6 Atom probe tomographic study of DERs

Due to the magnetic nature of martensite and the highly dislocated microstructure after plastic deformation, the distribution of nano-sized precipitates in DERs is very difficult to characterise using traditional electron microscopic techniques (SEM and FIB/TEM). Therefore, the redistribution of carbon is studied using atom probe tomography (APT), a powerful tool to study material from atomic scale. The results can not only reveal the nature of DERs, but also validate the proposed DER formation model.

5.6.1 Principles of APT

APT investigation is always conducted on a needle-like specimen with a radius of $\sim 50$ nm. When an atom bonded at the surface is exposed to sufficiently high and properly directed electric field, the atom can be ionised once its bonding is overcome by the electric field, causing the ion to leave the material surface. This process is also referred to as field evaporation. The evaporated ion is subsequently accelerated by the electric field within a very short distance (few tip-radii of the specimen), and then travels with a constant velocity $v_{\text{ion}}$ from the specimen tip at a high voltage $U = V_p$ to the ground potential $U = 0$.Assuming all the kinetic energy of the ion is given by the electric filed, the following equation can be obtained:

$$\frac{1}{2}m_{\text{ion}}v_{\text{ion}}^2 = n_{\text{ion}}eV_p,$$

(5.7)
where $m_{\text{ion}}$ is the mass of the ion, $n_{\text{ion}}$ is the ionisation state of the ion and $e$ is the fundamental electric charge. The above equation gives the mass-to-charge ratio for the ion.

$$\frac{m_{\text{ion}}}{n_{\text{ion}}} = \frac{2eV_p}{v_{\text{ion}}^2}. \quad (5.8)$$

For a known flight distance $F$, the velocity of the ion can be calculated by measuring its flight time $t_{\text{ion}}$, via $v = \frac{F}{t_{\text{ion}}}$. Therefore, the mass-to-charge ratio for the ion becomes:

$$\frac{m_{\text{ion}}}{n_{\text{ion}}} = 2eV_p \left(\frac{t_{\text{ion}}}{F}\right)^2. \quad (5.9)$$

Although the charge of the ion is not known directly, it can be inferred from its spectrum. Once the ion is received by a position-sensitive detector placed at the ground potential with a distance $F$ to the specimen tip, the element type and coordinates of the ion are recorded. By this means, the element distribution of the sample tip can be obtained by three dimensional (3D) reconstruction of the collected data. Figure 5.19 shows a schematic view of a tomographic atom probe.

5.6.2 Methodology

To study the element distribution within DERs, an APT specimen was prepared from the centre of the DER formed in the bearing sample tested for $1.2 \times 10^8$ cycles, using an FEI Helios NanoLab 660 workstation. The specimen was milled and sharpened by FIB, and was finally shaped into sharp needles. All the operations were carried out at the axial section of the bearing sample.
Figure 5.19: Schematic of a tomographic atom probe. $P$ represents the intercept point of ion trajectories. Image adapted from [84]

Figure 5.20 shows the location where the APT specimen was prepared. APT analyses were performed using a Local Electrode Atom Probe (LEAP 4000X HR, Cameca Instruments) in pulsed voltage mode at pulse fraction of 15% and a pulse frequency of 200 kHz. The specimen base temperature was about 60 K. The collected raw data were reconstructed into three-dimensional (3D) atomic maps and a precise decomposition process was conducted using IVAS software 1.

5.6.3 APT results

Figure 5.21 shows an overview of the reconstructed 3D atomic maps in the dark etching region for different alloying elements. It can be seen that C

1The cutting, polishing and etching of the fatigued bearing inner ring was done in Cambridge by the author; the preparation of the APT specimen using FIB, the collection of APT data and the processing of the raw data was conducted in Aachen University (Germany) by Dr Wenwen Song. The author interpreted the obtained results and performed the discussion.
exhibits significant partition within the specimen whilst the other alloying elements such as Cr, Mn and Si are homogeneously distributed. This implies that the formation of DERs is predominantly controlled by the redistribution of carbon.

A more detailed 3D atomic map in the DER is presented in Figure 5.22 (a). The detection volume is $65 \times 65 \times 170$ nm$^3$. The non-homogeneous distribution of carbon is manifested by carbon enriched and depleted zones. It follows that the carbon depleted zones represent the DER ferrite whilst the carbon enriched zones indicate nano-precipitates, e.g. $\theta$, $\eta$ and $\epsilon$ carbides. C is also enriched at where indicated by the arrows and the dashed line in Figure 5.22 (a). The carbon content in this zone is 6 – 7 at%, agreeing with the saturation carbon content of Cottrell atmospheres. This indicates a large amount of gathering dislocations and this zone is hence identified as martensite plate boundary. Figure 5.22 (a) also shows examples of the elemental distribution in the precipitates selected and analysed at the regions

Figure 5.20: (a) OM image of the bearing axial section with a DER showing the location where the APT specimen was prepared. (b) SEM image of the DER showing the position of the APT specimen.
of interest (ROIs), with the proxigrams for different ROIs shown in Figure 5.22 (b). For each ROI, the composition profiles are obtained along the X axis. The particle with a carbon content of approximately 25 at% is identified as Fe$_3$C carbide ($\theta$), that with a carbon content of approximately 33.3 at% as Fe$_2$C carbide ($\eta$) and that with a carbon content of approximately 29.4 at% as Fe$_{2.4}$C carbide ($\epsilon$), owing to their respective stoichiometries. In total, over 15 carbon enriched zones are detected in the dataset, most of which are identified as $\theta$. Nevertheless, the presence of the transition carbides surrounded by the DER ferrite is strong evidence of carbon migration from the parent martensite towards the pre-existing tempered carbides, which is the fundamental postulate of the proposed DER formation model.
To further confirm the carbon depletion in the DER ferrite, an atomic map in DER ferrite is presented in Figure 5.23 (a) in comparison to that in parent martensite in Figure 5.23 (c). The corresponding 1D content profiles are plotted in Figure 5.23 (b) and (d), respectively. For better visualisation, the carbon atoms are shown in 0.19 nm spheres and the iron atoms are shown as points. The average carbon content in the parent martensite is approximately 2.1 at\%, whilst the carbon content in the DER ferrite is much lower, ranging from 0.02 – 0.6 at\%. Beside, it is worth noticing that Cr content in the DER ferrite (~0.66 at\%) is also lower than that in the parent martensite (~1.39 at\%), which is probably due to the redistribution of Cr in
the vicinity of the Cr-enriched globular carbides.

Figure 5.23: Comparison between the carbon distribution in DER ferrite (a) and parent martensite (c). The corresponding 1D content profiles are plotted in (b) and (d).

5.7 Summary

Modelling and characterisation on the formation of DERs under RCF was carried out in this chapter, several conclusions can be drawn from this work.

- A novel theory is postulated to explain the carbon redistribution during RCF in bearing steels. It is proposed that the carbon in solid solution
in the matrix is transported by gliding dislocations towards the pre-existing precipitates (temper carbides) which thicken as a result.

- The dislocation-assisted carbon migration theory explains DER formation. The theory is capable of quantitatively predicting the development of DERs and the corresponding change in hardness.

- RCF tests were carried out on bearing inner ring samples and DERs were successfully reproduced. The DERs were further studied by OM and microindentation tests. Statistical data of DER% and ΔHV was collected and showed to be in agreement with the modelling results.

- FIB/TEM characterisation was also performed on a DER to reveal the decayed microstructure. Cellular structures can be observed inside the DER, indicative of the occurrence of plastic deformation and dislocation rearrangement. Micro-bands were also found in the microstructure; they may constitute the nucleation sites for WEBs.

- The effects of testing conditions on the formation rate of DERs were discussed, and a 3D map was established for mechanical property evolution during DERs formation, which is of great use for industrial applications.

- APT investigation was conducted in a DER. The results show significant carbon redistribution in DERs which leads to the formation of carbon enriched and depleted zones and confirm carbon migration from the parent martensite towards the pre-existing precipitates (tempered carbides).
Chapter 6

Formation of WEBs under RCF: characterisation and modelling

6.1 Introduction

White etching bands (WEBs) appear at very late stages of bearing life, e.g., beyond $10^8$ cycles, although experimental evidence shows that increasing either contact pressure or rotational speed can significantly accelerate their development. One of the most striking phenomena along with the occurrence of WEBs is the formation plate-like lenticular carbides (LCs), adjacent to which are ferrite bands. As indicated by the literature review in Chapter 2, the formation of these microstructural features is controlled by the migration of carbon as a consequence of cyclic loading. Therefore, a systematic characterisation focused on the distribution of carbon in WEBs has been
carried out, followed by modelling work on the thickening of LCs.

### 6.2 RCF tests

Three 6309 type through-hardened deep groove ball bearings were tested to reproduce WEBs. Table 6.1 lists the composition of the bearing steel. The material was quenched in a salt bath from the austenitization temperature of 860 °C, followed by tempering at 220 °C for 240 min. The resultant microstructure is identical to that of S2 studied in Chapter 3.

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>Ni</th>
<th>Mo</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.97</td>
<td>1.38</td>
<td>0.28</td>
<td>0.28</td>
<td>0.21</td>
<td>0.18</td>
<td>0.06</td>
<td>0.04</td>
</tr>
</tbody>
</table>

The RCF tests were performed with an SKF R2 full endurance tester with a maximum contact pressure of 3.3 GPa, a rotational speed of 6000 rpm and a operation temperature of 70 °C. The tests were suspended at $5 \times 10^8$, $10^9$ and $2 \times 10^9$ cycles.

After the RCF tests, the fatigued bearing inner rings were cut to show their circumferential and axial sections, followed by careful surface preparation and nital etching for subsequent microscopic investigation.
6.3 Microscopic investigation

6.3.1 OM

WEBs were observed at about 200 – 500 µm below the contact surface, the same region where shear stress peaks. If observed from the axial section, as Figure 6.1 shows, the WEBs are horizontal, whilst Figures 6.1 (c), (d) and (e) show the WEBs in the circumferential sections have an inclination angle of 20° – 30° to the over-rolling direction. Therefore, WEBs in three dimensions should be thin plates. With increasing number of cycles, the number density of WEBs increases and the depth range where WEBs are formed also becomes wider, indicating more WEBs nucleated with prolonged cycling. Moreover, the length and thickness of WEBs increases with increasing number of cycles as well.

Besides, microindentation tests using a Knoop tip were carried out along the depth in the circumferential sections of the bearing samples (Figures 6.1 (c), (d) and (e)). The spacing between neighbouring indents is 30 µm. Figure 6.2 shows the evolution of hardness change (ΔHV) with respect to depth at different formation stages of WEBs. For all the three samples, material hardening peaks at ∼ 150 µm below the contact surface, and when the number density of WEBs is still low (5 × 10^8 and 1 × 10^9 cycles) the ΔHV curves do not show much difference. It is worth noticing that the magnitude of maximum hardness increase (∼ 25 HV) agrees with that measured from the samples after repetitive push tests in Chapter 3, which indicates that the material response under RCF is similar to that under repetitive push. Nevertheless, when WEBs are populated (2 × 10^9 cycles), the softening effect
Figure 6.1: OM images of WEBs in the bearing samples. (a) Schematic of a bearing inner ring showing the locations where OM observation was carried out. (b) OM image of WEBs observed from the axial section. (c), (d) and (e) OM images of WEBs observed from the circumferential sections of the samples tested for different numbers of cycles.

of WEBs can be observed, and significant hardness decrease occurs in the depth range of WEB formation.

6.3.2 SEM

SEM investigation was carried out under an FEI Nova NanoSEM 450 SEM, and energy-dispersive X-ray spectroscopic (EDS) mapping was also performed on a WEB. The characterisation results reveal that a WEB consists of a ferrite band with LCs adjacent to it (Figures 6.3 (a) and (b)). LCs can appear on either or both sides of a ferrite band, but the former case is more
Figure 6.2: $\Delta HV$ evolution along the depth for the fatigued bearing inner ring samples. The dashed line represents $\Delta HV = 0$.

often observed. A very fine microstructure is found within the deformed ferrite bands compared with the surrounding matrix, as shown in Figure 6.3 (c). In addition, Figure 6.3 (d) illustrates when a developing WEB is encountered with pre-existing $\text{M}_3\text{C}$ particles, the latter can be dissolved. Similar phenomena have also been observed by other authors [53].

SEM characterisation also indicates gradual thickening of LCs with increasing number of cycles. Hence a statistical investigation was carried out with the thickness of at least 50 LCs from the most populated region measured and averaged for each sample. The average LC thickness ($l_{LC}$) values are shown in Table 6.2.

Table 6.2: LC thickness evolution with increasing number of cycles.

<table>
<thead>
<tr>
<th>$N$/cycles</th>
<th>$5 \times 10^8$</th>
<th>$10^9$</th>
<th>$2 \times 10^9$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$l_{LC}/\mu m$</td>
<td>0.12 ± 0.04</td>
<td>0.24 ± 0.07</td>
<td>0.43 ± 0.06</td>
</tr>
</tbody>
</table>

Figure 6.4 (c) – (e) presents the results of chemical mapping obtained from EDS. A WEB consisting of a LC and a ferrite band can be seen from
Figure 6.3: SEM images of WEBs from the circumferential section of the sample run for $2 \times 10^9$ cycles. (a) A set of WEBs found at the subsurface. (b) A WEB consisting of a deformed ferrite band and LCs. (c) very fine microstructure within a deformed ferrite band. (d) M$_3$C particles dissolved by a WEB.

the SEM image in Figure 6.4 (a). The size of the LC is much larger than the spherical M$_3$C particles. Comparing the distributions of iron in Figure 6.4 (c) and carbon in Figure 6.4 (e), it is obvious that carbon is enriched in the LC and the M$_3$C particles while the concentration of iron is lower in these regions. Moreover, in Figure 6.4 (e), carbon depletion is found in the ferrite band (region “A”) adjacent to the LC, compared with the uniformly spread carbon in the surrounding matrix (region “B”). This indicates that carbon is segregated from the ferrite band to the LC during WEB formation. Figure 6.4 (d) shows chromium is strongly segregated to the M$_3$C particles but not to the LC. Chromium enrichment in the M$_3$C particles can be expected as
these particles are formed at the spheroidisation stage [18] and the diffusion of chromium during the subsequent RCF test is negligible given such low diffusion coefficient of chromium at the bearing operation temperature. Carbon, on the other hand, somehow migrates towards LCs as a consequence of cyclic loading. Besides, the fact that chromium enrichment is not found in the chromium rules out the argument that LCs originate from the deformation of the pre-existing chromium-enriched M₃C particles [49], and hence LCs are in nature iron carbides.

Figure 6.4: Results of EDS chemical mapping on a WEB. (a) SE image of the EDS area containing a WEB. (b) Spectrum of the EDS test showing the detected elements from the area. The Kₐ peaks of each element are also marked. (c) – (e) Chemical mapping results of Fe, Cr and C, respectively.
6.3.3 TEM/FIB

TEM lamellea were prepared from the severest fatigued sample (2 × 10⁹ cycles) using FIB in a FEI Helios NanoLab workstation. The milled lamella contains a cross section along the width of a WEB. TEM observations were carried out in a FEI Osiris TEM/STEM 80-200 TEM where electron energy loss spectroscopy (EELS) was also performed to quantify the carbon content of the LCs.

Figure 6.5 is a TEM image of a FIB lamella. A part of the Pt protection was removed during final milling, indicating that the sample thickness had reached the lower limit. But due to the wedge-shape nature of the lamella, the optimum electron transparency is always the region near the Pt protection. From Figure 6.5, the cross section of a WEB can be seen in the middle of the lamella, with a ferrite band in between two LCs. An obvious cellular microstructure can be found inside the ferrite band, which is an indication of plastic deformation and consequent dislocation rearrangement, while the surrounding matrix has a distinctly different microstructure. It should also be noted that spherical M₃C particles can be found in the matrix but absent in the WEB. Figure 6.6 presents the boundary of the WEB at higher magnification and a LC lies in between the ferrite band and the matrix. The LC exhibits a columnar structure with units of about 100 nm in width. A more detailed microstructure image of the LC is shown in Figure 6.7. A similar microstructure was also observed by Lund *et al.* [51] and was described as “rung-like carbide disks”. Since the LCs are re-precipitated, one plausible explanation to this specific microstructure, in agreement with the analysis by
Swahn et al. [50], is that each small columnar unit follows the nucleation of a carbide crystallite, and when carbon is transported to the boundary to participate into further growth of the crystallites, they grow towards the inner part of the ferrite band at a constant pace. Therefore, the interfaces between the columns are actually grain boundaries. The fact that these grain boundaries are parallel to each other and perpendicular to the WEB boundary is a strong indication of carbon flux in the direction of crystallite growth, which is most likely to be perpendicular to the WEB boundary.

The ferrite band, as shown in Figure 6.6, contains finely distributed
Figure 6.6: TEM image showing a LC lying between the ferrite band and the matrix.

equiaxed dislocation cells, in agreement with the descriptions from the literature [52], and the sizes of the cells are in the range of 100 – 200 nm. Note that dislocation cells can also be found in the matrix near the LC. The formation of dislocation cells in martensite under RCF is a result of local plastic deformation, but what is observed in WEBs is different from that in white etching areas (WEAs) – another type of microstructural alteration under RCF that also exhibits similar martensite decay with dislocation cell formation, carbide dissolution and white contrast under visible light after etching with nital [85]. It is generally thought that WEAs are always ac-
accompanied by cracks initiated from foreign inclusions and form in a butterfly wing shape. Extensive characterisation and analysis on WEAs [29,30,37,86] has led to the conclusion that WEAs stem from the repetitive heating and rubbing of the crack surfaces and the corresponding microstructural change is of the same nature as that induced by severe plastic deformation (SPD). Additionally, the stress concentration around inclusions plays an important role in the formation of WEAs [39,87]. In the case of WEBs, however, microstructural changes occur in the absence of any cracks or observable stress concentrators, and the dislocation substructure is coarser than the ferrite grains in WEAs (100 – 200 nm vs. 10 – 50 nm). Besides, WEBs appear much later than WEAs under the same condition \((10^4 \text{ cycles} \text{ vs.} \ 10^8 \text{ cycles})\). More importantly, in spite of carbide dissolution in both cases, the dissolved carbon in WEAs is found segregated to dislocation cell walls [38] which leads to a distinct increase in hardness, whereas the dissolved carbon in WEBs is transported across the length of a few microns and re-precipitated as LCs along the boundaries leaving a softer ferrite band. Therefore, the formation of WEBs is more likely to be a gradual matrix degradation accompanied by carbon redistribution rather than the drastic microstructural transition caused by SPD.

In order to determine the carbon concentration in LCs, EELS was carried out. As shown in Figure 6.8 (a), a region containing a LC in the middle was selected for subsequent mapping for carbon. A spherical carbide particle with known composition \((M_3C, M = \text{Fe or Cr})\) was also included for reference. After careful processing of the spectrum, a carbon map was obtained, as presented in Figure 6.8 (b). Carbon enrichment can be clearly seen in both
Figure 6.7: High magnification image showing the unique microstructure of a LC. The long and thin LC consists of small carbide blocks, which are carbide crystallites with clear grain boundaries between each other.

The M$_3$C particle and the LC, which agrees with the finding from EDS. The quantitative analysis performed in the areas marked the dash-line rectangles yields a carbon content in the LC of about 23 at%, very close to the expected 25 at% in M$_3$C. Hence LC crystal structure is expected to be cementite.

The microstructure of the matrix surrounding the WEB was also studied. As shown in Figure 6.9, some needle-like precipitates are found in the matrix, with their elongation directions roughly perpendicular to the elongation direction of a large martensite plate. This agrees with the microstructural
Figure 6.8: EELS result on the LC. (a) Energy filtered TEM image of the LC with a $M_3C$ particle for reference. (b) C map obtained from the EELS test. The dash-line rectangles mark where quantitative analysis was carried out. The two images are of the same magnification and share the same scale bar.

description of tempered martensite [22], but there also exists a large number of much finer carbide fragments, suggesting the onset of precipitate dissolution before WEB nucleation. The plastic deformation in the matrix can be visualised by looking at a spherical $M_3C$ particle, as shown in Figure 6.10, where dislocation loops are generated from the sources at the particle-matrix interface and propagate into the particle.

6.4 LC thickening model

The dislocation-assisted carbon migration mechanism proposed in Chapter 4 can be applied to the growth of LCs. Prior to the precipitation of LCs, ferrite bands are formed under cyclic subsurface shearing. The shear stress can be so severe that the parent martensite loses the initial plate-like mi-
Figure 6.9: Martensitic matrix surrounding the WEB containing finely distributed nano-sized carbides.

crostructure and decays into a type of deformed ferrite, during which process a large amount of mobile dislocations are created and forced to glide to undertake plastic deformation. Martensite itself can be regarded as a carbon-supersaturated ferrite phase which tends to remove carbon from the solid solution to form carbides, due to the solubility of carbon in ferrite phase being very low (< 0.02 wt%) at the bearing operation temperature [18]. The dislocation gliding along with a high level of local hydrostatic compression stress causes the dissolution of both globular and tempered carbides [14], which adds to the supersaturation of carbon in the ferrite bands. Therefore,
the formation of precipitates becomes thermodynamically favourable, but the operation temperature of rolling contact fatigue tests (usually < 100 °C) is too low to cause noticeable thermal diffusion of carbon such that the kinetics of carbide formation is suppressed. In the case of LCs, however, the carbon flux caused by gliding dislocations from the carbon-supersaturated ferrite band towards the ferrite band boundaries contributes to such kinetics. This is the reason why LCs are only observed along deformed ferrite bands where very significant dislocation glide occurs. Figure 6.11 illustrates the three formation stages of a LC. At stage I, as pointed out by some authors [50, 52],
the boundary between a ferrite band and the matrix becomes favourable for carbide nucleation, and therefore numerous carbide nuclei are formed first along the ferrite band boundary. With further carbon transportation towards the ferrite boundary, these nuclei grow into carbide crystallites, which then coalesce into a long and thin LC at stage II. At stage III, the carbide crystallites continue growing towards the interior of the ferrite band, leading to thickening of the LC at the microstructural level. It should be noted that the proposed LC formation mechanism is different from diffusion-controlled carbide precipitation during heat treatment of the steel, as the lenticular morphology of LCs results from the coalescence of numerous carbide crystallites which nucleate immediately once a deformed band is formed, rather than through continuous lengthening along ferrite band boundaries. In a word, the length of a LC is dependent upon the length of the ferrite band, while its thickness gradually increases until all the carbon in the ferrite band is consumed.

With the dislocation transported carbon contributing to the proceeding LC front, there exists an equilibrium between the carbon flux of LC thickening and the dislocation-assisted carbon flux. The expression for the latter can be found from Equation (4.5), while the former, similar to the DER formation model, can be expressed as \( \frac{dh_{LC}}{dt} (C_{V\theta} - C_{Vb}) \), where \( C_{V\theta} \) and \( C_{Vb} \) denote the carbon concentration within the LC and within the ferrite band, respectively. The equilibrium equation thus becomes:

\[
\frac{dh_{LC}}{dt} (C_{V\theta} - C_{Vb}) = \frac{\Delta \gamma \dot{R} N_c}{b} \left[ 3 \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{AD}{k_B T \dot{R} N_c} \right)^{\frac{2}{3}} C_{Vb} \right].
\]

(6.1)
Figure 6.11: Schematic of LC growth as a result of dislocation-assisted carbon migration mechanism. A three-stage mechanism is suggested.

Note $t$ is always converted to the number of cycles via $N = t \dot{R} N_c$ during calculation. Furthermore, a carbon mass conservation holds for the system:

$$\lambda C_{V0} = l_{LC} C_{V\theta} + (\lambda - l_{LC}) C_{Vb}, \quad (6.2)$$

where $\lambda$ denotes the thickness of a fully developed WEB, which is estimated to be 9 $\mu$m [14, 15]. The initial carbon concentration of the system, $C_{V0}$, is equal to the carbon concentration of the alloy due to complete carbide dissolution in ferrite bands.
6.5 Modelling results and discussion

6.5.1 Modelling results

In order to carry out the calculation, a key input that describes the material response to cyclic loading, $\Delta \gamma$, is determined by equation (3.4) obtained in Chapter 3. The magnitude of $\tau_{\text{max}}$ is calculated from the contact pressure $p_0$ via $\tau_{\text{max}} = 0.3p_0 [5]$. The values of the other modelling parameters are listed in Table 4.1 in Chapter 4.

By substituting Equation (6.2) into Equation (6.1) and solving the differential equation, the evolution of $l_{\text{LC}}$ and $C_{\text{Vb}}$ with respect to $N$ can be obtained. The modelling results are compared with experiments from this research (Table (6.2)) and Buchwald and Heckel [14] with the same steel. Table lists the testing conditions for both RCF tests.

<table>
<thead>
<tr>
<th></th>
<th>This research</th>
<th>Buchwald and Heckel</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T$</td>
<td>75 °C</td>
<td>105 °C</td>
</tr>
<tr>
<td>$\tau_{\text{max}}$</td>
<td>1 GPa</td>
<td>1 GPa</td>
</tr>
<tr>
<td>$R$</td>
<td>6000 rpm</td>
<td>9600 rpm</td>
</tr>
<tr>
<td>$N_c$</td>
<td>4.96</td>
<td>4.96</td>
</tr>
</tbody>
</table>

By inputting the corresponding testing conditions, the calculated $l_{\text{LC}}$ thickening and $C_{\text{Vb}}$ depletion curves are shown in Figures 6.12 (a) and (b), respectively. For both cases, the observed LC thickening rates are well predicted, suggesting the accuracy of the proposed mechanism and model. Besides, the carbon depletion in the ferrite bands during thickening explains the detected material softening. Moreover, with the same material and contact pressure, LC thickening is faster in this research than in Buchwald and
Heckel’s research because of higher operation temperature, despite the de-
celeration effect from higher rotational speed.

Figure 6.12: Modelling results for (a) LC thickness evolution and (b) carbon depletion.

6.5.2 Parametric study

The testing conditions that control the formation rate of LCs are contact pressure, operation temperature and rotational speed. Higher operation temperature enhances carbon diffusion in the matrix, and thus enables more carbon to be captured by dislocations, accelerating LC thickening (Figure 6.13 (a)). Higher contact pressure, on the other hand, results in higher maximum shear stress at the subsurface and consequently, higher plastic shear strain amplitude. According to the proposed mechanism for carbon migration, dislocations act as vehicles transporting carbon in solid solution during dislocation glide, so severer plastic deformation, undertaken by higher mobile dislocation density and/or longer mean glide distance, definitely promotes this type of dislocation flow and the corresponding carbon flux (Figure 6.13 (b)). Increasing rotational speed, however, has a negative effect on carbon
migration as higher rotational speed decreases the time interval for carbon segregation to dislocations in each stress cycle, which reduces the amount of carbon in the carbon flux (Figure 6.13 (c)). The acceleration effect of operation temperature and deceleration effect of rational speed on WEB formation agrees with the argument by Polonsky and Keer [15]. Nevertheless, the predictions made by the present model are more accurate than any previous models. Figure 6.12 (a) demonstrates the model is capable of predicting the trend of LC formation under various testing conditions, which is crucial to industry and thus reduce the necessity of carrying out a large number of time consuming RCF tests.

6.6 Summary

WEBs were successfully reproduced in bearing samples after RCF tests, and the occurrence of WEBs softens the material. High resolution characterisation was also carried out on WEBs. SEM imaging reveals the structure of WEBs, with distinct features compared to the parent martensite; pre-existing spherical M₃C carbide particles can be dissolved by developing WEBs, rather than being deformed; EDX mapping effectively visualizes the elemental distribution within a WEB, and confirms the carbon migration towards LCs; FIB/TEM shows the cellular structure within the deformed ferrite bands, which is evidence for dislocation glide. Higher magnification TEM shows unique morphology of LCs consisting of carbide crystallites with the growth direction of LCs in agreement with the proposed mechanism. EELS confirms LCs to be θ-type carbides.
Figure 6.13: Effects of RCF testing conditions on LC thickening. (a) Thickening curves under increasing $T$ with other conditions constant ($\Delta \gamma = 6 \times 10^{-5}$, $\dot{R} = 6000$ rpm); (b) thickening curves under increasing $\Delta \gamma$ with other conditions constant ($T = 100$ °C, $\dot{R} = 6000$ rpm); (c) thickening curves under increasing $\dot{R}$ with other conditions constant ($T = 100$ °C, $\Delta \gamma = 6 \times 10^{-5}$).

The proposed dislocation-assisted carbon migration mechanism was applied to LC formation in bearing steels under RCF. The growth of LCs con-
sumes the carbon within adjacent ferrite bands and thus WEBs are formed. The special stress history of cyclic rolling contact triggers a dislocation-assisted carbon migration mechanism, with the magnitude of carbon flux dependent upon the operation conditions of RCF tests. The LC thickening process with increasing number of cycles is described by two key equations, the carbon flux equilibrium at the LC-ferrite interface and carbon mass conservation. The calculated LC thickening rates were compared with the experimental results and good agreement was achieved. It has been demonstrated that the model can accurately predict LC thickening (WEB formation) under various RCF testing conditions.

The formation of WEBs is a detrimental microstructural transition during bearing fatigue controlled by the migration of carbon. The proposed model can potentially be extended to other strain-induced microstructural decay which involves elemental redistribution.
Chapter 7

Formation of WEAs under RCF: modelling carbon segregation in dislocation cells

7.1 Introduction

In rolling contact fatigued bearing inner rings or rods, white etching areas (WEAs) are formed adjacent to cracks initiated from non-metallic inclusions or carbides. TEM investigations [29, 37, 40] have shown that inside a WEA the parent martensite matrix completely transforms into a ferritic dislocation cell structure. Gliding dislocations cluster to form cell walls, leaving dislocation depleted cell interiors (Figure 7.1). This type of dislocation rearrangement is a thermodynamically favourable process under plastic deformation as the resultant dislocation distribution possesses a lower energy configuration compared to a dislocation forest with evenly distributed dislocations [88–90].
Another interesting phenomenon in WEAs is the redistribution of carbon. Atom probe tomographic (APT) studies [38,41] have shown that the carbon in the solid solution is strongly segregated to dislocation cell walls while the carbon concentration in cell interiors is much lower. Also, the dissolution of carbides can increase the amount of carbon in the solid solution and thus adds to such carbon segregation. The above features in WEAs, namely carbide dissolution, dislocation cell formation, and carbon segregation to dislocation cell walls, have also been observed in steels undergoing severe plastic deformation (SPD) such as cold drawing [42, 43], mechanical milling [45, 46] and high pressure torsion [44]. Therefore, it is reasonable to assume that the formation of WEAs is also caused by SPD which may stem from the repetitive beating and rubbing of crack surfaces when cyclic loading is present [35].

![Image](image.png)

Figure 7.1: Formation stages of ferrite-nano grains in WEAs. Image adapted from [85].
Despite the extensive characterisation on WEAs, it is still unclear the nature of the driving force for such carbon migration, although it is generally thought that the formation of WEAs is controlled by the redistribution of carbon [38, 41]. Nevertheless, experimental results indicate a strong correlation between dislocation rearrangement and carbon segregation to dislocation cell walls, and thereby the postulated dislocation-assisted carbon migration mechanism, which has successfully explained the formation of DERs (Chapter 5) and WEBs (Chapter 6) under RCF, is applied to account for the microstructural alterations in WEAs.

7.2 Carbon redistribution during dislocation cell formation

When a martensitic matrix is subjected to cyclic loading, a large amount of dislocations may be generated. These dislocations glide and cluster to form dislocation cell walls, along which carbon in Cottrell atmospheres around gliding dislocations may be transported to cell walls. Based on this mechanism, a simplified model is proposed to describe the formation process of a dislocation cell. As the schematic in Figure 7.2 shows, a dislocation cell is assumed to be composed by two concentric spheres, where the inner one represents the cell interior and the outer layer represents the cell wall. If the radius of the cell is termed $r_c$, and the cell wall thickness termed $h$, an equation of carbon mass conservation of this system can be obtained as
follows:

\[ \frac{4}{3} \pi r_c^3 C_0 = \frac{4}{3} \pi (r_c - h)^3 C_i + \frac{4}{3} \pi [(r_c^3 - (r_c - h)^3)] C_w, \]  

(7.1)

where \( C_0 \) is the total carbon content of the system which is equal to that of the alloy by assuming complete carbide dissolution, \( C_w \) and \( C_i \) are the average carbon content in the cell wall and interior, respectively. The above equation yields the cell wall thickness as a function of carbon distribution.

\[ h = \left(1 - \sqrt{\frac{C_w - C_0}{C_w - C_i}}\right) r_c. \]  

(7.2)

The carbon transported to the cell wall should be anchored to atmospheres around the dislocations that stay immobile. Therefore, it is reasonable to assume that with the dislocation-assisted carbon migration mechanism operating, \( C_w \) is constant while \( C_i \) gradually decreases until all the carbon in the cell interior is consumed. This leads to gradual thickening of the cell wall (Equation (7.2)), and the formation of the dislocation cell is completed when all the carbon is segregated to the cell wall.

The final thickness of a dislocation cell wall \( (h_f) \) for a given \( r_c \) can be calculated when \( C_i = 0 \). \( h_f \) is used to estimate the dislocation densities in either the cell interior or in the cell wall by adopting the annihilation criterion by Essmann and Mughrabi [91]. In the absence of external stresses, the attractive shear stress between two dislocations of opposite sign is \( \frac{Gb}{2\pi y} \), where \( G \) is the shear modulus of the steel, \( b \) is the Burgers vector and \( y \) is the distance between dislocations. The two dislocations will annihilate spontaneously once the shear stress required for dislocation glide is exceeded. Hence there exists a critical dislocation spacing \( (y_c) \) above which the spontaneous
Figure 7.2: Schematic of proposed dislocation cell formation model.

dislocation annihilation cannot occur. For steels, the shear stress for the on-
set of dislocation glide is equal to the Peierls-Nabarro stress of body centred
cubic iron ($\tau_p$), which was calculated by Kashyap et al. [92] to be 0.11143
GPa for the most favourable slip system $\{1 1 0\} \langle 1 1 1 \rangle$. Therefore $y_c$ is
expressed as:

$$y_c = \frac{Gb}{2\pi \tau_p}.$$  \hspace{1cm} (7.3)

Referring to Essmann and Mughrabi, the dislocation density in a cell wall
with a thickness $h_f$ is given by:

$$\rho_w = \frac{1}{h_f y_c}.$$  \hspace{1cm} (7.4)
Likewise, the dislocation density in the cell interior becomes:

$$\rho_i = \frac{1}{2(r_c - h_f)y_c}. \quad (7.5)$$

According to the composite model of dislocation by Mughrabi [93], a dislocation cell can be treated as a composite consisting of a hard phase (cell wall) and a soft phase (cell interior), and the flow stress increment due to dislocation generation in each phase can be estimated using the Taylor’s relationship [94] for ferrite as 

$$\Delta \tau_w = 0.25Gb(\sqrt{\rho_w} - \sqrt{\rho_0}) \quad \text{and} \quad \Delta \tau_i = 0.25Gb(\sqrt{\rho_i} - \sqrt{\rho_0}),$$

where \(\Delta \tau_w\) and \(\Delta \tau_i\) are the flow stress increment in the cell wall and in the cell interior, respectively, \(\rho_0\) is the initial dislocation density of the martensitic matrix. Thus the mean flow stress increment in a dislocation cell can be obtained following the rule of mixtures:

$$\Delta \tau_m = f_w 0.25Gb(\sqrt{\rho_w} - \sqrt{\rho_0}) + f_i 0.25Gb(\sqrt{\rho_i} - \sqrt{\rho_0}), \quad (7.6)$$

where \(f_i = \frac{(r_c - h_f)^3}{r_c^3}\) and \(f_w = 1 - f_i\) are the volume fractions of the cell interior and the cell wall, respectively.

As for the formation kinetics of a dislocation cell, on the one hand the carbon flux caused by wall thickening is expressed as \(\frac{dh}{dt}(C_{Vw} - C_{Vi})\), where \(t\) is the cycling time and \(C_{Vw}\) and \(C_{Vi}\) are the carbon concentration in the cell wall and in the cell interior, respectively. On the other hand, the dislocation-assisted carbon flux \(J_d\) can be found from Equation (4.5). Therefore, the carbon equilibrium at the proceeding cell wall front leads to the following
equation:

$$\frac{dh}{dt}(C_{Vw} - C_{Vi}) = \frac{\Delta \gamma \dot{R}N_c}{b} \left[ 3 \left( \frac{\pi}{2} \right)^{\frac{3}{2}} \left( \frac{AD}{k_bT\dot{R}N_c} \right)^{\frac{3}{2}} C_{Vi} \right].$$

(7.7)

In addition, the carbon mass conservation in the form of Equation (7.1) yields the evolution of $C_{Vi}$ as a function of $h$:

$$C_{Vi} = C_{Vw} - \left( \frac{r_c}{r_c - h} \right)^3 (C_{Vw} - C_{V0}),$$

(7.8)

where $C_{V0}$ is the total carbon concentration of the system. By solving equations (7.7) and (7.8), the formation process of a dislocation cell in a WEA can be obtained.

### 7.3 Modelling results and discussion

#### 7.3.1 Determination of input parameters

**Dislocation cell radius – $r_c$**

The size of the dislocation cells in WEAs reported in the literature show in a wide range of values. This is because the cell size is strongly dependent upon local stress state, and the WEAs reported in different papers were formed under different contact pressures and were chosen from different positions of the fatigued samples where the local stress state can differ greatly. Moreover, FIB/TEM characterisation of the dislocation cells is usually carried out in very a small region compared to the whole WEA, while the distribution of the cell size, as suggested by some authors [30, 32], is inhomogeneous in a
WEA. Table 7.1 summaries the dislocation cell size in WEAs reported in the literature. The measured hardness values are also presented.

Table 7.1: Reported cell size and hardness in WEAs.

<table>
<thead>
<tr>
<th>Ref</th>
<th>Cell size</th>
<th>Hardness of matrix</th>
<th>Hardness of WEAs</th>
</tr>
</thead>
<tbody>
<tr>
<td>[37]</td>
<td>$\sim 50$ nm</td>
<td>$820 - 850$ HV</td>
<td>$\sim 1000$ HV</td>
</tr>
<tr>
<td>[95]</td>
<td>$\sim 10$ nm</td>
<td>$\sim 800$ HV</td>
<td>$1040 - 1200$ HV</td>
</tr>
<tr>
<td>[29]</td>
<td>$-$</td>
<td>$770 - 830$ HV</td>
<td>$1000 - 1200$ HV</td>
</tr>
<tr>
<td>[36]</td>
<td>$-$</td>
<td>$1091 - 1213$ HV</td>
<td>$1234 - 1275$ HV</td>
</tr>
<tr>
<td>[86]</td>
<td>$50 - 100$ nm</td>
<td>$-$</td>
<td>$-$</td>
</tr>
</tbody>
</table>

Table 7.1 indicates the size range of dislocation cells in WEAs is $10 - 100$ nm, which means $r_c$ ranges 5 to 50 nm.

**Dislocation cell wall carbon content $- C_w$**

As argued in the literature [13], it has been widely accepted that WEAs are formed by localised severe plastic deformation (SPD) of steels, during which process the carbon segregation to dislocation cell walls is a common phenomenon. In this respect, a large number of atom probe tomography (APT) investigations have been carried out to study such carbon redistribution in high carbon steels after SPD, and the comparison of the reported experimental results leads to a surprising finding that regardless of the initial states of the Fe-C systems, the final states always consist of a cellular structure with roughly constant carbon content in dislocation cell walls ($C_w$). Table 7.2 summaries the APT results from the literature.

Table 7.2 shows that irrespective of initial state and SPD method, the reported $C_w$ is always around 7 at%, which also agrees with the theoretical saturation carbon content in Cottrell atmospheres [97,98]. This finding
Table 7.2: APT results on carbon redistribution of steels undergoing SPD.

<table>
<thead>
<tr>
<th>Ref</th>
<th>Steel type</th>
<th>SPD method</th>
<th>dislocation cell size (nm)</th>
<th>$C_w$ (at%)</th>
<th>$C_i$ (at%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>45</td>
<td>Fe – 0.8 wt% C alloy powder</td>
<td>mechanical milling</td>
<td>450 – 900</td>
<td>7</td>
<td>0.5</td>
</tr>
<tr>
<td>42</td>
<td>Fe – 0.81 wt% C pearlitic steel</td>
<td>cold drawing</td>
<td>&lt; 10</td>
<td>7.5</td>
<td>-</td>
</tr>
<tr>
<td>44</td>
<td>Fe – (0.6 – 0.8) wt% C pearlitic steel</td>
<td>high pressure torsion</td>
<td>20</td>
<td>6</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>43</td>
<td>Fe – 0.9 wt% C pearlitic steel</td>
<td>cold drawing</td>
<td>-</td>
<td>9</td>
<td>0.5 – 1</td>
</tr>
<tr>
<td>46</td>
<td>Fe – 0.9 wt% C pearlitic steel</td>
<td>mechanical milling</td>
<td>8 – 15</td>
<td>6.7</td>
<td>0.64 – 1.5</td>
</tr>
<tr>
<td>41</td>
<td>100Cr6 martensitic bearing steel</td>
<td>rolling contact fatigue</td>
<td>10</td>
<td>4.4 – 8</td>
<td>-</td>
</tr>
<tr>
<td>96</td>
<td>Fe – 0.7 wt% C pearlitic railway steel</td>
<td>rolling contact fatigue</td>
<td>&lt; 1000</td>
<td>7 – 9</td>
<td>-</td>
</tr>
</tbody>
</table>
supports the proposed dislocation cell formation theory in this research that the wall thickness and the corresponding dislocation density is controlled by carbon distribution. Therefore, the value of 7 at% is used for the following calculation. It is also worth noticing that the reported carbon content in the cell interiors ($C_i$), although significantly lower than the normal carbon content of the materials, does not decrease to zero. The possible reason is that unlike RCF, the traditional SPD methods produce the nanocrystallized ferrite grains within a relatively short time such that a complete carbon segregation cannot occur, whereas in WEAs the material is repetitively loaded for millions of cycles and thus the fully formed dislocation cells can be carbon free in the cell interiors.

**Plastic strain amplitude near inclusions – $\Delta \gamma$**

Due to the stress concentration effect of impurities and cracks, the strain amplitude for WEA formation is different from that for the matrix decay (DERs or WEBs). It is very difficult to measure this value using experimental methods. Nevertheless, Freitas *et al.* [99] calculated the normal plastic strain amplitude distribution around an Al$_2$O$_3$ inclusion using finite element method (FEM), showing that under a typical Hertzian stress (3.5 GPa), $\Delta \gamma$ around the inclusion located at the maximum shear stress region is of the order of $10^{-2}$. The FEM result indicates that $\Delta \gamma$ within WEAs can be 2 – 3 orders of magnitude higher than that in the matrix. Therefore, $\Delta \gamma$ is estimated to be $10^{-2}$ for the proposed dislocation cell formation model.
7.3.2 Modelling results

Calculations on fully formed dislocation cells in WEAs were carried out for a typical 100Cr6 bearing steel where $C_0 = 3.63$ at%. The evolution of $h_f$ with respect to $r_c$ is plotted in Figure 7.3 (a). As equation (7.2) indicates, $h_f$ increases linearly with $r_c$ when $C_i = 0$. Figure 7.3 (b) shows the evolution of the dislocation densities, $\rho_i$, $\rho_w$ and $\rho_m$, with respect to $r_c$. It can be seen that the magnitudes of the dislocation densities are more sensitive to cell radius when the cell size is small ($r_c < 20$ nm), and increase rapidly with decreasing cell radius. The predicted magnitude of $\rho_m$ varies in the range of $10^{15} - 10^{16}$ m$^{-2}$, agreeing with the analysis of other authors [29].

![Figure 7.3: Modelling results of the evolution of cell wall thickness (a) and the evolution of dislocation densities (b) with respect to cell radius.](image)

The hardness increase in WEAs was calculated from the mean flow stress increment as a consequence of cell formation, considering that the predicted dislocation density in WEAs can be 1 order of magnitude higher than in the parent martensite ($\sim 10^{15}$ m$^{-2}$ [100]). It is generally accepted that the hardness of a metal ($H$) is related to its yield strength via $H = 3\sigma_Y$, where both $H$ and $\sigma_Y$ are in GPa [101–103]. However, this relationship becomes very
inaccurate for high strength steels [104]. Therefore by fitting to experimental data, an alternative equation was proposed by Pavlina and Tyne [104] to define the Vickers hardness in terms of the yield strength as follows:

\[
H = 0.4(\sigma_Y + 110),
\]

(7.9)

where \( H \) is in HV and \( \sigma_Y \) is in MPa. Hence the hardness change in HV (\( \Delta HV \)) due to work hardening becomes \( 0.4\Delta \sigma_Y \), where \( \Delta \sigma_Y \) can be converted from the mean flow stress increment through \( \Delta \sigma_Y = \sqrt{3}\Delta \tau_m \) and \( \Delta \tau_m \) can be calculated from Equation (7.6). The predicted \( \Delta HV \) with respect to \( r_c \) is plotted in Figure 7.4 in comparison to the reported data listed in Table 7.1, and good agreement is obtained.

![Figure 7.4](image)

Figure 7.4: Calculated hardness increase in the cellular structure with respect to cell radius in comparison with reported \( \Delta HV \) data from Becker et al. [37], O’Brien et al. [86], Grabulov et al. [29] and Solano-Alvarez et al. [34].

Figures 7.5 (a), (c) and (e) show the calculated wall thickening curves of dislocation cells with three typical cell radii reported in the literature. It can be seen that for each curve, there is a major cell formation stage where the cell
wall thickens rapidly, and then the curve comes to a plateau, indicating the end of the cell formation process. The number of cycles at which this process ends \( N_e \) was estimated and marked for each cell radius, and it is obvious that larger cells require a higher number of cycles to form. This finding is very important because if the cell size distribution in a WEA is inhomogeneous, the growth of a WEA may become inclined to the propagating major crack depending upon local cell size. This could be the origin of the butterfly wing shape of WEAs. Accordingly, the calculated carbon depletion curves in cell interiors are plotted in Figures 7.5 (b), (d) and (f). For the purpose of facilitating the comparison, the progress of cell formation is quantified by the completion percentage of cell wall thickening \( \left( \frac{h}{h_f} \times 100\% \right) \) or carbon depletion \( \left( \frac{C_0 - C_i}{C_0} \times 100\% \right) \), termed Cell %. Figure 7.6 shows the formation curves with the three cell radii, and the gray area shows the reported range where WEAs start to appear [18], roughly overlapping the calculated major formation stages.

### 7.3.3 Parametric study

As the model suggests, dislocation cell formation in WEAs is a carbon controlled process, the rate of which is dependent upon the factors affecting dislocation-assisted carbon migration. Figure 7.7 shows the effects of RCF testing conditions on the formation of dislocation cells as calculated from the proposed model. Figure 7.7 (a) indicates that, as temperature controls the amount of carbon that can be captured by gliding dislocations, higher operation temperatures can promote carbon migration, accelerating cell for-
Figure 7.5: Calculated dislocation cell wall thickening curves for (a) $r_c = 5$, (c) $r_c = 25$ nm and (e) $r_c = 50$ nm, and the corresponding carbon depletion curves in cell interiors as respectively shown in (b), (d) and (f).

Plastic strain amplitude increases the number and speed of mobile dislocations, the vehicles for carbon migration. Therefore, as shown in Figure 7.7 (b), the formation curve is shifted towards lower number of cycles...
Figure 7.6: Cell formation curves with the three cell radii calculated from the modelling results in Figure 7.5. The gray area represents the reported $N$ range where WEAs start to appear [18].

with increasing plastic strain amplitude. As for the rotational speed, Figure 7.7 (c) indicates that the cell formation is decelerated by higher rotational speeds, owing to the shorter interval time allowing for Cottrell atmosphere formation.

### 7.3.4 Butterfly wing shape of WEAs

WEAs are also referred to as “butterflies” due to their unique morphology. As the calculation results in Figure 7.5 indicate, the possible cause of the butterfly wing shape is the difference in $N_e$ for different cell radii. In this case, a WEA growth simulation is carried out on the basis of the proposed dislocation cell formation model. As suggested by some authors [30], the dislocation cell size varies with respect to the distance from the main crack. The cells close to the main crack are very fine ($\sim 10$ nm in size), and the
Figure 7.7: Effects of RCF testing conditions on dislocation cell formation. (a) Thickening curves under increasing $T$ with other conditions constant ($\Delta \gamma = 1 \times 10^{-2}$, $\dot{R} = 6000$ rpm); (b) thickening curves under increasing $\Delta \gamma$ with other conditions constant ($T = 65 \, ^\circ C$, $\dot{R} = 6000$ rpm); (c) thickening curves under increasing $\dot{R}$ with other conditions constant ($T = 65 \, ^\circ C$, $\Delta \gamma = 1 \times 10^{-2}$)
size gradually increases towards the other boundary (boundary b as shown in Figure 2.8). Figure 7.8 schematically shows such cell size variation within a WEA. For simplification, it is assumed that the cell size increase follows a linear relationship with respect to the distance from the main crack. Assuming the crack length to be \( l \) and the WEA thickness to be \( w \), the minimum cell radius \( (r_{c, \text{min}}) \) is located adjacent to the main crack while the maximum cell radius \( (r_{c, \text{max}}) \) is located at the maximum thickness of a WEA \( (w_{\text{max}}) \). Therefore, the distance \( (d_r) \) of a cell with radius \( r_c \) from the main crack can be calculated from the following equation:

\[
d_r = \frac{r_c - r_{c, \text{min}}}{r_{c, \text{max}} - r_{c, \text{min}}} \times w_{\text{max}}
\] (7.10)

According to the literature, \( r_{c, \text{min}} = 5 \) nm, \( r_{c, \text{max}} = 250 \) nm, and \( w_{\text{max}} = 20 \mu m \). During crack propagation, the material below any point on the main crack is loaded for a certain number of cycles, which corresponds to \( N_e \) of the largest cell that can be formed. Hence, the distance of the largest cell from the main crack calculated from equation (7.10) defines the thickness of the WEA below this point, namely \( w = d_{r, \text{max}} \). With this method, the WEA boundary can be obtained by linking the thickness defined by each point along the main crack. Figure 7.9 (a) shows the SEM image of a WEA formed after \( 1.3 \times 10^8 \) cycles by Grabulov et al. [30]. Using the same testing conditions as inputs \( (T = 25 \degree C, \dot{R} = 15000 \text{ rpm, } N_c = 2) \), the simulated WEA shape is presented in Figure 7.9 (b), with the growth history also shown. For calculation convenience, the main crack was assumed to propagate at a constant speed. It should also be noted that the input \( \Delta \gamma \) was
Figure 7.8: (a) Schematic of a pair of WEAs formed at the subsurface of a rolling contact fatigued sample. (b) Schematic of the cell size distribution within a WEA. Images adapted from [85].

estimated to be one order of magnitude lower than that predicted by Freitas et al. [99] with a contact pressure of 3.5 GPa, as the contact pressure used in [30] is only 2.7 GPa. Comparing Figures 7.9 (a) and (b) it can be seen the simulation result agrees well with the experiment observation, suggesting a good accuracy of the proposed model.

7.4 Summary

The formation of WEAs under RCF has been studied. The analysis of the relationship between the formation of dislocation cells in WEAs, and the
segregation of carbon at their walls, leads to the suggestion that WEA formation is carbon controlled. Using the proposed dislocation-assisted carbon migration theory, a dislocation cell formation model has been established.

First of all, the model is capable of predicting the evolution of some key microstructural parameters in WEAs as a function of cell radius, such as cell wall thickness, dislocation density, flow stress and hardness. The predicted parameters were compared to literature measurements obtaining good agreement. Secondly, the model can predict the kinetics of dislocation cell formation, and for the cell radii of reasonable values, the predicted major cell formation stages lie within the observed range of number of cycles where WEAs appear. Additionally, the model considers the RCF testing conditions that can affect the formation kinetics of dislocation cells, temperature, plastic strain amplitude and rotational speed and the effects of these external factors has been discussed. Finally, a WEA growth model has been presented on the basis of the proposed dislocation cell formation model, and the growth model.
successfully predicted the butterfly wing shape of a WEA, which agrees well with microscopic observations.

The study on WEAs in this chapter is an insight into the microstructural alterations in bearing steels undergoing severe plastic deformation, which provides a potential solution to the observed phenomena in other Fe-C systems tested with various types of severe plastic deformation methods.
Chapter 8

Overall microstructural kinetics under RCF

The proposed dislocation-assisted carbon migration theory has been successfully applied to explain and predict different types of microstructural alterations throughout bearing life. The instability of bearing steels under RCF is a consequence of strain-induced carbon redistribution, manifested as the formation of carbon enriched and depleted regions. Carbon segregation in martensitic steels is a thermodynamically favourable process, due to the metastable nature of the phase. In spite of the insufficient carbon diffusivity under normal bearing operation temperature, applied stresses enhance such carbon migration via the interaction between gliding dislocations and carbon atoms. Owing to the special stress history of rolling contact where the time intervals between stress pulses allow for the formation of Cottrell atmospheres around mobile dislocations, a dislocation-assisted carbon flux \( J_d \) is produced. Therefore, the kinetics of such carbon segregation can be
described as long as a carbon flux equilibrium is found, and along with a carbon mass conservation equation, the development of the carbon-enriched features, whether pre-existing precipitates, lenticular carbides or dislocation cell walls, can be predicted. Figure 8.1 shows how the proposed theory is applied to the three types of microstructural alterations, and the two key equations are given as follows:

\[
\frac{d(r_p, l_{LC}, h)}{dt}(C_{\text{VII}} - C_{\text{VI}}) = J_d; \quad (8.1)
\]

\[
V_0C_{\text{V0}} = V_{\text{II}}C_{\text{VII}} + (V_0 - V_{\text{II}})C_{\text{VI}}, \quad (8.2)
\]

where \(C_{\text{V0}}, C_{\text{VI}}\) and \(C_{\text{VII}}\) represent the carbon concentration in the whole system, in the carbon depleted zones and in the carbon enriched zones, respectively, and \(V_0, V_{\text{I}}\) and \(V_{\text{II}}\) represent the volume of the whole system, of the carbon depleted I zones and of the carbon enriched II zones, respectively.

Based on this mechanism, three microstructural alteration models have been established.

Different types of microstructural alterations occur at different stages of RCF. Figure 8.2 shows an example of the calculated formation curves of the three types of microstructural alterations in a tested bearing inner ring throughout its lifetime. Comparing the curves, it can be found that the major factor that controls the formation rate of each type of microstructural alteration is the size of the carbon segregation system, which determines how far carbon needs to be transported. For WEAs, the carbon migration distance is the radius of a dislocation cell, typically tens of nanometres; while for DERs, the carbon in the solid solution needs to travel hundreds of nanome-
Figure 8.1: Basic concepts of the microstructural alteration models based on the dislocation-assisted carbon migration theory.

The proposed models can be used to generate tools to evaluate the microstructural damage in bearing steels. Figure 8.3 shows the microstructural alterations maps where the lines are plotted by linking the calculated numbers of cycles of 100% completion of each type of microstructural alteration under certain RCF testing conditions, and the enclosed regions define the microstructural alterations that are fully developed. The maps can be used...
Figure 8.2: Formation curves of WEAs, DERs and WEBs in a rolling contact fatigued 6309 bearing inner ring sample made of 100Cr6 bearing steel with the heat treatment of S1 in Chapter 3. The testing conditions used for the calculation are: $T = 80 \, ^\circ\text{C}$, $p_0 = 3.5 \, \text{GPa}$ and $R = 6000 \, \text{rpm}$. Note WEB% is defined as the ratio between the current LC thickness and the maximum LC thickness.

to index the fatigue status of a bearing component for any given number of cycles, and thus can reduce the necessity of carrying out large numbers of time consuming full endurance RCF tests.

Subsurface initiated RCF of bearing steels causes micro-pitting and spalling at the surface. The description of the subsurface microstructural alterations is crucial to the prediction of bearing life. WEAs, though always viewed as symptoms of crack propagation, could still affect crack propagation speed as the resultant microstructure in WEAs has distinctly higher hardness compared to the parent martensite. The formation of DERs, however, leads to material softening and thus reduces the load capacity of the material. Therefore crack propagation is expected to be faster in a DER than in the parent
martensite. WEBs produce large-sized inhomogeneities in the microstructure, soft ferrite bands and hard cementite plates, the interfaces between which could initiate cracks. In summary, given that the occurrence of the microstructural alterations has significant influences on the material damage development at all stages, the proposed quantitative models are ready to be incorporated with a variety of life prediction models and increase their accuracy.
Figure 8.3: Microstructural alterations maps. (a) Map for various temperatures with other conditions constant ($\Delta \gamma = 10^{-4}$, $\dot{R} = 6000$ rpm, $N_c = 4.96$). (b) Map for various plastic strain amplitudes with other conditions constant ($T = 70^\circ C$, $\dot{R} = 6000$ rpm, $N_c = 4.96$). (c) Map for various rotational speeds ($T = 70^\circ C$, $\Delta \gamma = 10^{-4}$, $N_c = 4.96$). Note the strain amplitudes used for WEAs are assumed to be 2 orders of magnitude higher than that for DERs and WEBs, and the cell radius is assumed to be 50 nm for detectable WEAs.
Chapter 9

General conclusions and suggestions for future work

The formation of three major types of microstructural alterations observed in martensitic 52100 bearing steels under rolling contact fatigue (RCF) – dark etching regions (DERs), white etching bands (WEBs) and white etching areas (WEAs) – was systematically investigated, with both model predictions and experimental validations having been presented. The major achievements that have been gained in this research are listed as follows:

- Two types of standard heat treatments were applied to 52100 bearing steel, where S1 was tempered at 160 °C for 90 min and S2 tempered at 220 °C for 240 min. The differences in the sample microstructures were characterised by electron microscopy. The strength differential effect (SED) was detected by uniaxial tension and compression tests in both S1 and S2 samples, with this effect being more pronounced in harder (brittler) material. Subsequently, a series of repetitive push
tests were performed on S2 compression samples with various maximum pressures. The plastic response of the material to cyclic loading was obtained, which was used as an important input for the modelling work of WEBs.

- As implied by extensive characterisation in the literature, the key factor that controls the formation of the microstructural alterations is the strain-induced redistribution of carbon. Therefore, a novel theory was postulated that gliding dislocations act as vehicles transporting carbon in the solid solution towards carbon absorbers. The theory combines the Cottrell atmosphere theory with the Orowan equation, which enables to quantify the dislocation-assisted carbon flux given the unique stress history of rolling contact. The presence of such carbon flux eventually leads to carbon segregation at carbon-enriched zones, leaving behind carbon-depleted zones.

- The postulated dislocation-assisted carbon migration theory was successfully applied to the formation of DERs. It was presumed that the thickening of pre-existing carbide precipitates leads to the carbon depletion in DER ferrite. The proposed model is capable of making predictions on the progress of DER formation and the corresponding hardness decrease with respect to the number of cycles under various RCF testing conditions, which agrees well with the experimental observations from both this research and the literature. Furthermore, the atom probe tomographic study confirmed the existence of carbon-enriched and carbon-depleted zones in the DER specimen, and the
detected transition carbides also confirms the pre-existing precipitates to be the carbon absorbers.

- The postulated dislocation-assisted carbon migration theory was successfully applied to the formation of WEBs. Long and thin lenticular carbides (LCs) are precipitated along deformed ferrite bands, and the growth of LCs gradually consumes the carbon in ferrite bands. The proposed model was used to predict the thickness of LCs with respect to the number of cycles under various testing conditions, with good agreement obtained. The EDS and EELS techniques were employed to map carbon distribution in WEBs, confirming the carbon enrichment in LCs and the carbon depletion in ferrite bands.

- The postulated dislocation-assisted carbon migration theory was successfully applied to the formation of WEAs. WEAs consist of nano-sized dislocation cells with significant carbon segregation at cell walls detected by atom probe tomography. Hence a model was established to describe the formation of dislocation cells in terms of carbon redistribution, and the predicted major formation stages of the cells with various radii agree well with the range of number of cycles where WEAs start to appear. The model also managed to predict the hardness increase in WEAs as reported in the literature. Moreover, a WEA growth simulation was carried out and the unique butterfly wing shape of WEAs was successfully described.

- It was indicated by this research that the major three types of microstructural alterations formed in bearing steels under rolling contact
fatigue, despite the differences in morphology, size and stage of occurrence, all stem from the same mechanism underneath: carbon redistribution as a consequence of dislocation glide. Chapter 8 showed an overview of how different types of microstructural alterations are formed one after another and presented powerful tools – microstructural alterations maps – to evaluate the extent of microstructural damage in a bearing component throughout its lifetime.

Future work is suggested as follows:

- The proposed models require two types of inputs for calculation, RCF testing conditions and microstructure parameters. The former can be directly obtained from testing whilst the latter, at the current stage, can only be obtained through experiments. Future work is suggested on the general description of the microstructures of bearing steels with various compositions and heat treatments. The material response to cyclic loading, which is highly dependent upon the microstructure, should also be modelled universally.

- The proposed models are capable of predicting the evolution of mechanical properties of the material during microstructural alteration formation, which has great influence on crack nucleation and propagation, and subsequent bearing failure. Therefore, future work can be done to embed the microstructural alteration models in various life prediction models to increase their accuracy.

- The impact of the postulated dislocation-assisted carbon migration theory can be far-reaching. It not only leads to great progress in un-
derstanding the strain-induced carbon redistribution in bearing steels which has puzzled researchers for several decades, but also can be further extended to other scenarios with similar phenomena such as severe plastic deformation and hydrogen embrittlement.
Appendix A

Stress components under elliptical contact

This part shows the equations used for the calculation of the stress components \((\sigma_{xx}, \sigma_{yy}, \sigma_{zz}, \tau_{xy}, \tau_{xz}, \tau_{yz})\) in a contact body under elliptical contact. Detailed analysis of the stress state can be found from [5] and [24]. Note that all the coordinates used in the equations are normalised with respect to the half long axis of the contact ellipse in the \(x\) direction, \(a\), where the definition of the coordinates is identical to that shown in Figure 2.5. Hence here \(x\), \(y\) and \(z\) stand for \(x/a\), \(y/a\) and \(z/a\). To carry out the calculation, the ellipticity of the contact ellipse \(k\) (< 1) and the Poisson’s ratio of the material \(\nu\) must be known.

First of all, the value of \(s\) (a parameter used in the expressions of the
stress components) is given by the positive root of the following equation:

\[ s^6 + s^4[1 + k^2 - x^2 - y^2 - z^2] + s^2[k^2 - y^2 - z^2 - k^2(x^2 + z^2)] - z^2k^2 = 0 \quad (z > 0). \]  

(A1)

The stress components related to \( z \) are given by:

\[ \frac{\tau_{zx}}{p_0} = \frac{-kxzL}{1 + s^2}, \]  

(A2)

\[ \frac{\tau_{yz}}{p_0} = \frac{-kyzL}{k^2 + s^2}, \]  

(A3)

\[ \frac{\sigma_{zz}}{p_0} = \frac{-kz^2L}{s^2}, \]  

(A4)

where

\[ L = z/\left[ s^3H\sqrt{(1 + s^2)(k^2 + s^2)} \right], \]  

(A5)

\[ H = \left( \frac{x}{1 + s^2} \right)^2 + \left( \frac{y}{k^2 + s^2} \right)^2 + \left( \frac{z}{s^2} \right)^2. \]  

(A6)

While the other three stress components are given by:

\[ \frac{\sigma_{xx}}{p_0} = \frac{\nu}{\pi p_0} \frac{\partial \psi}{\partial z} - \frac{1 - 2\nu}{2\pi p_0} \frac{\partial^2 \psi_1}{\partial x^2} - \frac{z}{2\pi p_0} \frac{\partial^2 \psi}{\partial x^2}, \]  

(A7)

\[ \frac{\sigma_{yy}}{p_0} = \frac{\nu}{\pi p_0} \frac{\partial \psi}{\partial z} - \frac{1 - 2\nu}{2\pi p_0} \frac{\partial^2 \psi_1}{\partial y^2} - \frac{z}{2\pi p_0} \frac{\partial^2 \psi}{\partial y^2}, \]  

(A8)

\[ \frac{\tau_{xy}}{p_0} = \frac{1 - 2\nu}{2\pi p_0} \frac{\partial^2 \psi_1}{\partial x \partial y} - \frac{z}{2\pi p_0} \frac{\partial^2 \psi}{\partial x \partial y}. \]  

(A9)

The derivatives of \( \psi \) and \( \psi_1 \) are given by:

\[ \frac{z}{p_0} \frac{\partial^2 \psi}{\partial x^2} = -2\pi k \left[ zI_1 - \left( \frac{xs}{1 + s^2} \right)^2 L \right], \]  

(A10)
\[
\frac{z}{p_0} \frac{\partial^2 \psi}{\partial y^2} = -2\pi k \left[ z I_2 - \left( \frac{y s}{k^2 + s^2} \right)^2 L \right], \quad (A11)
\]

\[
\frac{z}{p_0} \frac{\partial^2 \psi}{\partial x \partial y} = \frac{2\pi k x y L s^2}{(1 + s^2)(k^2 + s^2)}, \quad (A12)
\]

\[
\frac{1}{p_0} \frac{\partial^2 \psi_1}{\partial x^2} = -2\pi k \left[ z I_1 - \frac{1}{k'^3} \left( y \theta_1 + x \theta_2 + k' - \frac{z k'}{s} \sqrt{\frac{s^2 + k^2}{s^2 + 1}} \right) \right], \quad (A13)
\]

\[
\frac{1}{p_0} \frac{\partial^2 \psi_1}{\partial y^2} = -2\pi k \left[ z I_2 + \frac{1}{k'^3} \left( y \theta_1 + x \theta_2 + k' - \frac{z k'}{s} \sqrt{\frac{s^2 + 1}{s^2 + k^2}} \right) \right], \quad (A14)
\]

\[
\frac{1}{p_0} \frac{\partial^2 \psi_1}{\partial x \partial y} = \frac{2\pi k}{k'^3} (x \theta_1 - y \theta_2), \quad (A15)
\]

where \( k' = \sqrt{1 - k^2} \). The auxiliary angles \( \theta_1 \) and \( \theta_2 \) are given by:

\[
\frac{\theta_1}{k'^3} = -\frac{\tan^{-1}(ny/D)}{n^3}, \quad (A16)
\]

\[
\frac{\theta_2}{k'^3} = -\frac{\tanh^{-1}(nx/C)}{n^3}, \quad (A17)
\]

where

\[
n = \sqrt{1 - k^2}, \quad (A18)
\]

\[
C = 1 + s^2 + N, \quad (A19)
\]

\[
D = k^2 + s^2 + N, \quad (A20)
\]

\[
N = \frac{G z}{s}, \quad (A21)
\]

\[
G = \sqrt{(1 + s^2)(k^2 + s^2)}. \quad (A22)
\]
The three elliptic integrals $I_1$, $I_2$ and $I_3$ can be calculated via:

\begin{align}
I_1(k, s) &= \frac{1}{k^2}[F(\phi, k') - E(\phi, k')], \\
I_2(k, s) &= \frac{1}{k^2} \left[ \frac{E(\phi, k')}{k^2} - F(\phi, k') - \frac{1}{k^2s} \sqrt{\frac{k^2 + s^2}{1 + s^2}} + \frac{1}{s} \sqrt{\frac{1 + s^2}{k^2 + s^2}} \right], \\
I_3(k, s) &= \frac{1}{k^2} \left[ \frac{1}{s} \sqrt{\frac{k^2 + s^2}{1 + s^2}} - E(\phi, k') \right],
\end{align}

where $E(\phi, k)$ and $F(\phi, k)$ are the standard incomplete elliptic integrals and $\tan \phi = 1/s$. 

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