Mechanisms of Microstructural Damage during Rolling Contact Fatigue of Bearing Steels

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Preface

This dissertation is submitted for the degree of Doctor of Philosophy at the University of Cambridge. The research reported 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 2010 and September 2013. 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 degree, diploma or other qualification at any other university or institution. This dissertation does not exceed the word limit of 60,000 words.

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Abstract

Bearings are employed in a number of applications under extremely demanding conditions. During long operation times, the material undergoes rolling contact fatigue where microstructural damage manifests as dark-etching regions and white-etching areas, which display different properties from the surrounding region. The aim of this study is to identify the mechanisms for such damage and to suggest models that can explain the influence of the initial microstructure and test conditions.

In order to appraise the stress state in rolling contacts, two testing techniques were employed and it was examined if the testing methods could reproduce the same damage as in bearing operation. During ball-on-rod fatigue testing, microcracks were generated adjacent to inclusions and some were decorated with white-etching areas. Repetitive push tests showed a similar extent of subsurface hardening compared to the ball-on-rod tests, and allowed the strain per stress cycle to be measured.

The microstructural alterations in a white-etching area were studied both on a macroscale and on an atomic-scale. The degree of stress concentration near a microcrack was calculated employing a finite element method. The microstructure, as well as the segregation behaviour of alloying elements in the white-etching area, were investigated by employing transmission electron microscopy and atom probe tomography. A nanocrystalline structure with scattered carbide particles was observed in the white-etching area. Carbon and silicon segregation was highly pronounced in some boundaries of dislocation cell structures.

Models were suggested to account for the microstructural alterations during rolling contact fatigue. Carbide coarsening in dark-etching regions was modelled by considering how carbon diffusion is assisted by dislocation glide. The predicted hardness evolution was consistent with experimental observation. The kinetics of carbide dissolution in white-etching areas was calculated by taking two processes into account: deformation accumulation and carbon diffusion. These models suggest that the microstructural changes during bearing operation can be controlled by tailoring the initial microstructure and managing the test conditions.

Nomenclature

 ρ Dislocation density

- D_C^{α} Diffusion coefficient of C in the matrix
- n_{ion} Charge of an ion
- [F] Nodal force matrix
- [K] Stiffness matrix
- [u] Nodal displacement matrix
- α Pressure viscosity coefficient
- α_{γ} Strain enhancement factor resulting from stress concentration
- α_{θ} Fraction of U_C that can be stored at the carbide/matrix interface
- $\alpha_{\tau_{xz}}$ Stress concentration factor for τ_{xz}
- β^* Atomic attachment rate
- χ Goodness of fit in Rietveld refinement
- $\Delta A_{\alpha\theta}$ Increase in the matrix/carbide interfacial area after N
- $\Delta G_{m\theta}$ Driving force for cementite precipitation
- ΔG_{nucl} Gibbs free energy for nucleation

 Δx_{disl} Diffusion distance of a carbon atom

 $\Delta \epsilon_C$ Half normal plastic strain range per cycle

 $\Delta \epsilon_{C,gauge} \ \Delta \epsilon_{C}$ obtained from the position of the strain gauge

 $\Delta \epsilon_{C,pos} \Delta \epsilon_C$ obtained from the lower stage position

 $\Delta \gamma_C$ Half shear plastic strain range per cycle

 $\Delta \gamma_N$ Accumulated strain over N

 $\dot{\gamma}$ Strain rate

 \dot{R} Bearing operating speed in revolutions per minute

 ϵ Normal strain

 ϵ_{max} Maximum normal strain

 $\epsilon_{p,max}$ Maximum normal plastic strain

 $\gamma_{\alpha\theta}$ Matrix/cementite interfacial energy

 λ Ratio between h_{min} and $h^*_{roughness}$

 λ_C C diffusion distance during carbide dissolution

- $(\sigma_i)_r$ Residual normal stress in *i* direction
- $(\tau_{ij})_r$ Residual normal stress in *i* plane in *j* direction
- μ_0 Dynamic viscosity of contacting surfaces
- ν_i Poisson's ratio of the component *i*

 ϕ_{ikj} Profile function

 σ'_i Normal stress in *i* direction considering residual stresses

 σ_i Normal stress in *i* direction

au Shear stress

- τ'_1 Principal shear stress considering residual stresses
- τ_1 Principal shear stress
- τ_a Shear stress amplitude
- τ_{ij} Shear stress on plane *i* in *j* direction
- τ_{inc} Incubation time
- $\tau_{max,rss}$ Maximum resolved shear stress
- τ_{xz0} Value of τ_{xz} in the absence of the crack
- θ Angle between a principal shear stress plane and the surface
- θ_{crack} Angle between the crack and the surface

 \vec{F} Force

 $\vec{v_C}$ Steady drift velocity of a carbon atom

A Constant for U

a Half side length of the contact area

 $a_{\epsilon}, b_{\epsilon}$ Lattice parameter of ϵ -carbide

 $a_{\theta}, b_{\theta}, c_{\theta}$ Lattice parameter of cementite

b Magnitude of Burgers vector

 C_i^{α} Concentration of component *i* in the matrix per unit volume

 C_i^k Concentration of component *i* in the precipitate *k* per unit volume

- C_0 Carbon at% in the alloy
- C_V Carbon concentration per unit volume
- $C_{\alpha'0}$ Initial carbon mol fraction in the matrix
- $C_{\alpha'}$ C concentration in the matrix in wt%
- C_{V0} Carbon concentration in the alloy per unit volume
- $C_{V\alpha'0}$ Carbon concentration in the initial matrix per unit volume
- $C_{V\alpha'}$ Carbon concentration in the matrix per unit volume
- $C_{V\alpha,eq}$ Carbon concentration in equilibrium ferrite per unit volume
- $C_{V\alpha,eq}$ Equilibrium carbon concentration in the matrix per unit volume
- $C_{V\epsilon}$ Carbon concentration in ϵ -carbide per unit volume
- $C_{V\theta}$ Carbon concentration in cementite per unit volume
- D Diffusion coefficient of carbon
- d Arithmetic mean of two diagonals of Vickers indentation
- D_i^{α} Diffusion coefficient of component *i* in matrix
- D^{disl} Dislocation assisted diffusivity
- d_{ion} Flight distance of an ion

 d_{latt} Lattice spacing

E Elastic modulus

- *e* Elementary charge of an electron
- E^* Reduced elastic modulus

- E_i Elastic modulus of the component i
- F_i Nodal forces of a node i

 F_{kj} Structure factor

 F_{rad} Radial force applied to a rod specimen in ball-on-rod RCF test rig

 F_{result} Resultant force by F_{spring} in ball-on-rod RCF test rig

 F_{spring} Force applied by springs in ball-on-rod RCF test rig

- G^{α} Gibbs free energy of the matrix
- $G^{k,f}$ Gibbs free energy of the precipitate after adding G_{diss}
- $G^{k,i}$ Gibbs free energy of the precipitate before adding G_{diss}
- G_c Energy barrier against nucleation
- $G_{\Delta A_{\alpha\theta}}$ Gibbs free energy created by $\Delta A_{\alpha\theta}$
- G_{diss} Driving force for carbide dissolution
- G_{sys} Total Gibbs free energy of the system
- h_{min} Minimum film thickness
- $h_{roughness,i}$ Roughness of the component i
- $h^*_{roughness}$ Reduced roughness
- J Carbon flux
- J_{disl} Carbon flux made via dislocation glide
- *k* Index of overlapping neighbouring Bragg reflections
- $k^{(i)}$ Stiffness of an element *i*

k_b Boltzmann o	constant
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- k_y Yield limit of the material by shear
- K_{IIc} Mode II fracture toughness
- L Load during hardness measurement
- l_0 Initial length of the specimen
- l_c Critical size of the crack
- l_f Final length of the specimen
- l_p Spacing between precipitate particles
- L_{kj} Lorentz polarisation factor
- m, n Variable used to obtain stress components in 2D contact
- M Taylor factor
- M_p Mass of the formula unit
- $M_{\alpha\theta}$ Ferrite/cementite interface mobility

 m_{ion} Mass of an ion

 M_{kj} Multiplicity

- N Number of cycles
- N_0 Total number of possible nucleation sites
- N_{θ} Number density of cementite particles per unit volume
- N_C Number of stress cycles per revolution
- n_C Number of carbon transported by the unit length of a dislocation

 N_{form} Number of formula units per unit cell

- n_{nucl} Number of atoms in the nucleus
- n_{site} Number of octahedral interstitial sites per atomic plane normal to a dislocation

O Contact point

- p(x) Pressure distribution
- P Total compressing load
- p_0 Maximum contact pressure
- P_{kj} Preferred orientation function
- p_{max} Maximum compressive pressure
- Q Rate of G_{sys} dissipation
- q_i Independent state parameters
- R Ideal gas constant
- r Distance from a carbide particle
- R^* Reduced radius
- r_0 Initial radius of a carbide particle
- r_{θ} Particle radius of cementite
- r_d Distance between a dislocation core and a carbon atom
- R_i Radius of the component *i*
- r_k Radius of the precipitate
- $r_{\theta,max}$ Maximum radius of cementite particles

 $r_{\theta,min}$ Minimum radius of cementite particles

- $r_{\theta,m}$ Mean radius of cementite particles
- $r_{d\epsilon}$ Limiting distance within which carbon atoms are more stable with dislocation than the ϵ -carbide
- $r_{d\theta}$ Limiting distance within which carbon atoms are more stable with dislocation than the cementite
- r_{disl} Limiting distance within which carbon atoms can follow the dislocation motion during plastic deformation
- S_j Scaling factor of a phase j
- T Temperature
- t Time
- t_{ion} Flight time of an ion
- T_{oj} Overall temperature factor
- t_{pcpt} Precipitation simulation time
- U Dislocation/carbon interaction energy
- U_{ϵ} Binding energy of a carbon atom in the ϵ -carbide
- U_{θ} Binding energy of a carbon atom in the cementite
- U_C Accumulated plastic energy per each stress cycle
- u_i Nodal displacement of a node i
- V Unit cell volume
- v Velocity at contacting surfaces

- v_{θ} Cementite growth rate
- V_m Molar volume
- $v_{\theta,int}$ Velocity of interface-controlled growth

 V_{APT} Applied voltage

- v_{disl} Steady drift velocity of a carbon atom
- V_{f0} Initial volume fraction of the precipitate
- $V_{f\theta}$ Volume fraction of temper carbide
- w_C Atomic weight of C
- w_i Weighting parameter
- w_{Fe} Atomic weight of Fe
- x Distance from the contact point in the over-rolling direction
- x_0 Initial position of the upper stage during the repetitive push test

 X_C C mol fraction

- X_C^k C mol fraction in the precipitate
- $X_C^{\alpha,f}$ C mol fraction in the matrix after carbide dissolution
- $X_C^{\alpha,i}$ C mol fraction in the matrix before carbide dissolution
- x_i Instantaneous position of the upper stage during the repetitive push test
- y_i Observed intensity at a data point i
- y_{bi} Background intensity at data point *i*
- y_{ci} Calculated intensity at i

- Z Zeldovich factor
- z Depth from the surface
- APFIM Atom probe field ion microscopy
- APT Atom probe tomography
- DER Dark-etching region
- EDS Energy dispersive spectroscopy
- EHD Elastrohydrodynamic
- ELKP Elastic linear kinematic plasticity
- FEM Finite element method
- FIM Field ion microscopy
- HPT High pressure torsion
- MM Mechanical milling
- OM Optical microscopy
- RCF Rolling contact fatigue
- rpm Revolution per minute
- SEM Scanning electron microscopy
- SIM Scanning ion microscopy
- SMAT Surface mechanical attrition treatment
- SPD Severe plastic deformation
- TEM Transmission electron microscopy

TMA Thermomagnetic analysis

WEA White-etching area

- WEB White-etching band
- WEL White-etching layer
- XRD X-ray diffraction

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Chapter 1

General Introduction

Bearings are used in numerous applications from small skateboards to large industrial structures. While they operate in a variety of components for different applications, their environments are mostly unfavourable: high contact pressure and rotational speed, as well as elevated temperatures. Under such conditions, the most important issue is their finite life: when do they fail and why? Failure of bearings comes from several factors (Fig. 1.1); most stems from inappropriate conditions such as lubrication, sealing, and mounting (90%). Although all running conditions are optimised, subsurface fatigue cannot be avoided due to the innate nature of the material. This is why resistance to subsurface fatigue is significant for bearing life.

Subsurface fatigue is another way to refer to rolling contact fatigue (RCF), which is the damage coming from repetitive rolling contacts among bearing elements (balls, rollers, rings). Damage may manifest as a crack at the subsurface leading to spalling and failure. Until now, empirical relationships are used to predict bearing life by considering contact pressure, as well as various initial material conditions, such as an amount of residual austenite and carbide, grain size, hardness, as well as the magnitude of residual stresses [2].

From a metallurgical point of view, there seems to be a possibility to fill the empirical gap between the initial microstructure and the bearing life by identifying mechanisms



Figure 1.1: Bearing failure modes and their frequencies [1].

of microstructural damage. This is illustrated in Fig. 1.2. During RCF of bearing steels, dark-etching regions (DERs), white-etching bands (WEBs) and white-etching areas (WEAs) are considered as different forms of the damage. They are so-called because of their different etching behaviour with respect to the unaffected region. Such damage stems from microstructural changes such as carbide coarsening/dissolution as well as the formation of cellular structures, which occurrence depends on the testing conditions (e.g. contact pressure, temperature, and speed of revolution) [3]. These induce property changes at the subsurface, which may be a crucial origin of spalling failures by influencing the condition for both initiation and propagation of a subsurface crack. As a result, they may determine the life of bearings [4]. Hence, the microstructural alterations need to be investigated in order to improve bearing life prediction. So far, the mechanisms of microstructural alteration with damage have never been modelled in association to failure, although the presence of alterations has been reported extensively in the literature [3].

This work aims to understand the origin of mechanical property alterations during RCF of bearing steels, as well as to clarify their mechanisms. Firstly, details of the stress state and the forms in which microstructural damage (DER, WEB, WEA) manifests



Figure 1.2: Causes of bearing failure by spalling in the metallurgical aspect. Some images are adopted from [5, 6].

during RCF are studied. A literature survey is presented in Chapter 2 to illustrate how the damage is related to the important microstructural changes (carbide coarsening and dissolution) and the test conditions.

Another challenge for RCF is to verify the associated microstructural transition via laboratory testing methodologies. Although standard bearing tests can simulate perfect operating conditions, they are expensive and the strain experienced during testing cannot be measured. The possibility of tests using simple sample geometry, and the way to make reliable estimation of the strain during the bearing operation will be discussed. By running selected tests, it is investigated whether the fatigue damage in real bearings can be reproduced by laboratory testing in Chapter 3. Moreover, the test results can be adopted as input parameters to models for carbide coarsening (Chapter 4) and its dissolution (Chapter 6).

Based on Chapters 2 and 3, models are suggested for evolution during RCF are presented in Chapters 4 and 6. A model for carbide coarsening is introduced in Chapter 4. The model considers that C can be transported from the supersaturated matrix to the carbide particles via dislocation glide. In Chapter 5, phenomena related to carbide dissolution are studied to support the model illustrated in Chapter 6. Stress concentration, which is the condition for carbide dissolution, is explained adopting a finite element method, and WEAs are characterised with advanced microscopy techniques. After all, the model for carbide dissolution is introduced in Chapter 6, where the dissolution is divided into two processes: strain energy accumulation and actual dissolution controlled by C diffusion. General conclusions and suggestions for future research are presented in Chapter 7.

Chapter 2

Literature Review

In order to set the scope of this study, a general description of through-hardened bearing is first introduced. Then, the stress state in bearings is reviewed to emphasise the special features of the rolling contact in their components. Microstructural damage due to rolling contact fatigue (RCF) is also introduced. Finally, an attempt to relate damage to the stress state experienced in bearings is presented.

2.1 Through-hardened bearings

Through-hardened bearings are so called because they are hardened throughout their cross section. The most popular through-hardened steel grade is 100Cr6 (Table 2.1). This study majorly considers 100Cr6 with some calculations involving other variations of 100Cr6 grade, which compositions are shown in Table 2.1. Bearing steels are classified as high carbon steels, which contain almost 1 wt%C in order to achieve high hardness. Cr, Mn, and Mo increase hardenability [7–9]. Mo not only enhances carbide stability, but also increases toughness of steels by preventing P segregation in prior austenite boundaries [10, 11]. Besides, Mn is necessary to prevent S segregation, and Si is sometimes added to control the precipitation kinetics by suppressing cementite precipitation [12].

The heat-treatment to form martensite is as follows. Partial austenitisation takes place in the austenite+cementite phase field, leaving undissolved spherical carbides which are

1			0	0	L J
	C / wt $\%$	Cr / wt%	Mn / wt $\%$	Si / wt $\%$	Mo / wt $\%$
100Cr6	0.93 - 1.05	1.35 - 1.60	0.25 - 0.45	0.15 - 0.35	$0.10 \;({\rm max.})$
100CrMo7-3	0.93 - 1.05	1.65 - 1.95	0.60 - 0.80	0.15 - 0.35	0.20 - 0.35
100CrMnMoSi8-4-6	0.93 - 1.05	1.80 - 2.05	0.80 - 1.10	0.40 - 0.60	0.50 - 0.60
100CrMo7	0.93 - 1.05	1.65 - 1.95	0.25 - 0.45	0.15 - 0.35	0.15 - 0.30

Table 2.1: Compositions of popular through-hardened bearing steels [13].

often called as residual carbides. Then, the alloy is quenched and tempered at 150–220 °C. Wear resistance can be enhanced with the residual carbides from partial austenitisation [14]. Tempering is carried out at such a low temperature to provide toughness while hardness is maintained by the substantial amount of C in solid solution, and the precipitation of transition carbides such as ϵ , η , and cementite [15]. As a result, the microstructure consists of residual carbides, nano-sized transition carbides, and retained austenite in a martensitic matrix. An example microstructure of martensitic bearing steels is shown in Fig. 2.1. Residual carbides assume a few tenths of a micrometre-sized globular particles; transition carbides as well as twins are observed within the plates. In this study, the influence of retained austenite is neglected to focus on the major phases: martensite matrix supersaturated with C, residual carbides, and tempering carbides.

2.2 Stress state during rolling contacts

A bearing is used to join two rotating parts in a machine. The cross section of a simple ball bearing is shown in Fig. 2.2. The two parts can be connected to the inner and outer ring, respectively, and rotate independently. In this way, both rings are kept in continuous contact with ball elements on which a normal force can be applied. In order to understand the stress state during rolling contact of the bearing, two analyses are addressed in this section: (i) Hertzian elastic contact theory and (ii) cyclic plasticity. These highlight the special characteristics of the deformation state during bearing operation and will be useful to not only assess the test methods that is introduced in the Section 3.1, but also to consider the mechanisms of fatigue damage discussed in the Section 2.3.



Figure 2.1: Typical microstructure of martensitic bearing steels acquired by (a) secondary electron imaging and (b) transmission electron bright field imaging.



Figure 2.2: (a) The cross section of a deep groove ball bearing. Rotation of either ring may be absent or in the opposite direction depending on the application. (b) Terminology for inner ring sections

2.2.1 Hertzian elastic contact theory

The stress state during the rolling contact can be well-estimated by Hertzian contact theory assuming linear elasticity [16]. Here, two infinite cylinders are brought into contact without traction force for a two dimensional line contact geometry (Fig. 2.3a). As a result, the contact area would be a rectangle with side lengths of 2a and infinity. The pressure distribution (p(x)) is given by Hertz as [16] (Fig. 2.3b)

$$p(x) = p_0 \sqrt{1 - \left(\frac{x}{a}\right)^2}$$
(2.1)

where x is the distance from the contact point, O, in the over-rolling direction, and p_0 is the maximum contact pressure. Taking P as total compressing load, the relationship between P, a, and p_0 can be expressed in terms of the the radii (R_1, R_2) , the elastic moduli (E_1, E_2) and the Poisson's ratios (ν_1, ν_2) of the bodies as follows [17].

$$P = \frac{1}{2}p_0\pi a \tag{2.2}$$

$$a = \sqrt{\frac{4PR^*}{\pi E^*}} \tag{2.3}$$

$$p_0 = \sqrt{\frac{PE^*}{\pi R^*}} \tag{2.4}$$

where

$$\frac{1}{E^*} = \frac{1 - {\nu_1}^2}{E_1} + \frac{1 - {\nu_2}^2}{E_2},\tag{2.5}$$

$$\frac{1}{R^*} = \frac{1}{R_1} + \frac{1}{R_2}.$$
(2.6)

Therefore, for a given load, the contact dimensions and the maximum contact pressure can be calculated.

The stress components $(\sigma_x, \sigma_z, \tau_{xz})$ and the principal shear stress τ_1 are expressed as



Figure 2.3: (a) Schematic diagram of two solids of revolution in two dimensional contact at O. (b) Pressure distribution for the contact area obtained by Hertz.

follows:

$$\frac{\sigma_x}{p_0} = -\left\{\frac{m}{a}\left(1 + \frac{z^2 + n^2}{m^2 + n^2}\right) - \frac{2z}{a}\right\},\tag{2.7}$$

$$\frac{\sigma_z}{p_0} = -\left\{\frac{m}{a}\left(1 - \frac{z^2 + n^2}{m^2 + n^2}\right)\right\},$$
(2.8)

$$\frac{\tau_{xz}}{p_0} = -\frac{n}{a} \left(\frac{m^2 - z^2}{m^2 + n^2} \right), \tag{2.9}$$

$$\tau_1 = \frac{1}{2}\sqrt{(\sigma_x - \sigma_z)^2 + 4\tau_{xz}^2},$$
(2.10)

where

$$m^{2} = \frac{1}{2} \left[\left\{ \left(a^{2} - x^{2} + z^{2}\right)^{2} + 4x^{2}z^{2} \right\}^{1/2} + \left(a^{2} - x^{2} + z^{2}\right)^{2} \right], \qquad (2.11)$$

$$n^{2} = \frac{1}{2} \left[\left\{ \left(a^{2} - x^{2} + z^{2} \right)^{2} + 4x^{2} z^{2} \right\}^{1/2} - \left(a^{2} - x^{2} + z^{2} \right)^{2} \right].$$
(2.12)

These stresses are plotted on the xz-plane in Fig. 2.4, with the angle between the τ_1 plane and the surface (θ) which is calculated by

$$\theta = \frac{1}{2} \tan^{-1} \left(-\frac{\sigma_x - \sigma_z}{2\tau_{xz}} \right).$$
(2.13)

All plots are symmetric along x = 0 except τ_{xz} and θ , which have the same magnitude with opposite signs across x = 0. Moreover, σ_x and σ_z show their maximum at z = 0,

Table 2.2: The values and locations of maximum shear stresses for two dimensional line contact [18].

	Maximum value	x	z
$ au_1$	$0.300 \ p_0$	0	0.786~a
$ au_{xz}$	$0.250 \ p_0$	$\pm~0.87~a$	$0.5 \ a$

where the maximum τ_{xz} and τ_1 points are located below the surface. The stress state of the element in the bodies can also be appraised through two kinds of diagrams: the stress distribution with respect to the depth (z) at x = 0, and that with respect to xat a certain z. The first type is shown in Fig. 2.5a. In this case, τ_1 always acts on the planes inclined 45° to the surface, since τ_{xz} is zero. Although two normal stresses (σ_x and σ_z) decrease along z, τ_1 is zero at the surface and shows its maximum at the subsurface region. For the second type of the diagram when $x \neq 0$, τ_{xz} always exists, and τ_1 changes along x/a (Fig. 2.5b,c). The values and positions of maximum τ_{xz} and τ_1 are given in Table 2.2. Each stress state at the depth of maxima is shown in Fig. 2.5b,c. Since the depths of maximum shear stresses are located closely, the stress states in Fig. 2.5b,c are similar. The magnitude of τ_{xz} and τ_1 is similar in the range of $x \leq -0.5a$ and $x \geq 0.5a$; however, τ_{xz} decreases gradually and changes its sign at x = 0, while τ_1 remains positive in $-0.5a \leq x \leq 0.5a$.

Now, considering that the upper cylinder is rolling from left to right relatively to the lower cylinder in contact, the element in the lower cylinder would experience a contact cycle in the direction from +x to -x, which is shown schematically in Fig. 2.6. It is clear that the τ_1 plane changes its direction as the cylinder passes by. θ at z = 0.786a, the depth of which contains the maximum τ_1 , is plotted in Fig. 2.7 with respect to x/a. Note that another principal shear stress plane exists orthogonal to the one shown due to force equilibrium. The magnitude of θ starts from 45° (-45°) at x = 0. It decreases gradually to zero at the point which τ_1 becomes similar to τ_{xz} . Then, it changes its sign and increases slowly.



Figure 2.4: Distribution of normalised stresses ((a) σ_x , (b) σ_z , (c) τ_{xz} , and (d) τ_1), and (e) θ , the angle of the principal shear stress plane with respect to the surface, in xz-plane for a two dimensional line contact.



Figure 2.5: Stress distribution for a line contact at (a) x = 0, (b) z = 0.786a, and (c) z = 0.500a. In (a), $-\tau_1$ has been plotted instead of τ_1 .



Figure 2.6: Schematic stress states of an element at the subsurface during over-rolling. Lower states illustrate the principal shear stress resulting from the upper states. θ is the angle of principal shear stress plane with respect to the surface.



Figure 2.7: The orientation of principal shear stress plane (θ) with respect to x at z = 0.786a.

2.2.2 Cyclic plasticity

Although the elastic theory provides a plausible estimate for the stress state during rolling contact, it should be borne in mind that the material hardens once it starts to deform. Therefore, this estimate of stress state may only be valid for the first cycle; in the subsequent cycles, one has to consider plasticity which produces residual stresses modifying the previous stress state.

There are four kinds of the response when a material undergoes cyclic loading: (i) perfectly elastic, (ii) elastic shakedown, (iii) cyclic plasticity (plastic shakedown), and (iv) incremental collapse (ratchetting) (Fig. 2.8) [19]. The activating response is determined by the load factor, p_0/k_y , where k_y is the shear yield stress. If the load is too low for yield to occur, the cyclic loading would be "perfectly elastic". With increasing stress, yield takes place and residual stress develops. In subsequent cycles, the residual stress is added to the applied stress. If the material does not have to deform plastically but deforms elastically under the combined stress, it is called "elastic shakedown". If the combined stress is large enough to cause yield continually, the plastic response occurs in either two ways: (i) "cyclic plasticity" or "plastic shakedown" with closed stress-strain hysteresis


Figure 2.8: Material response to cyclic loading adopted from [19].

loop, (ii) "residual strain accumulation" or "ratchetting" with the open loop resulting in unidirectional strain accumulation.

The response is determined by several factors such as the contact pressure, traction coefficient, and contact geometries (two/three dimensional contact). These effects are shown in shakedown maps (Fig. 2.9) assuming symmetrical loading during each cycle. Without traction, the material is expected to go through the elastic limit, elastic shakedown, plastic shakedown, and subsurface flow in sequence. Plastic shakedown is observed at one particular point ($p_0 = 4k_y$) in two dimensional contact, while it is present in the load range ($p_0 \ge 4.7k_y$) in three dimensional contact [17]. These values have been obtained from the criterion that maximum τ_1 needs to exceed k_y for yield to occur. It is also interesting that surface flow will happen instead of plastic shakedown if either traction or load is high, i.e. in the right upper corner of the map.



Figure 2.9: Shakedown maps of (a) 2D line contact [19] and (b) 3D elliptical contact [20]. In (a), "flow" means forward flow which is caused by alternating plasticity.

2.3 Microstructure damage during rolling contact fatigue

Microstructural damage during fatigue is mostly observed in the inner ring, since its possible contact area is smaller than that of the outer ring (Fig. 2.2). The occurring damage can be classified as a dark-etching region (DER), white-etching bands (WEBs), and white-etching area (WEA) due to their contrast under optical microscope. The damage is only observed under the ideal conditions; that is, without lubrication faults or contamination. In order to eliminate confusion, microstructural "damage" is differentiated from microstructural "changes" or "alterations" in this study. Damage occurs at a larger scale owing to the microstructural changes/alterations pertaining to specific phases, e.g. carbide coarsening and dissolution.

2.3.1 Dark-etching regions

First observed by Jones, a DER is manifested as dark contrast by optical microscopy when etched with nital and picral [21]. A DER can be clearly observed in axial and



Figure 2.10: Optical micrographs of a dark-etching region in (a) axial and (b) circumferential sections of the inner ring of a martensitic bearing steel. (c) Secondary electron images of the dark-etching region in the same sample.

circumferential sections of the inner ring (Fig. 2.2b, 2.10a,b). It is often seen at a depth from several hundred of micrometres under the surface [5, 21, 22] which corresponds to the depth of maximum shear stresses (Table 2.2). Its typical width is \sim 2 mm and increases with running time and contact pressure [5, 21, 23]. Its boundary with the undeformed matrix is diffuse; the etching gets heavier and then lighter along the depth across it. Higher resolution reveals that it consists of scattered deformed patches (Fig. 2.10c) and the etching strength depends on the concentration of these patches.

Test conditions determine DER formation time. The reported testing conditions are arranged in Table 2.3 and plotted in Fig. 2.11 in terms of p_0 and the number of cycles (N). DERs start to form at $\sim 10^7 - 10^8$ cycles under $\sim 3-4$ GPa. They form faster with higher pressure. Note that the operation speed also plays a role; a DER did not form at 3.37 GPa with a lower speed (124 Hz = 1500 rpm).

The DER formation has been known to be caused by tempering [21, 23, 29]. While

Table 2.3: Reported microstructural damage and corresponding bearing test conditions $(p_0: \text{ maximum contact pressure, } N: \text{ number of cycles})$ for 52100 steel at the room temperature. Note that the operating speed is given by the number of cycles per second (Hz). $[d_{inner}, d_{outer}]$ in Fig. 2.2 is [40 mm, 80 mm] and [45 mm, 100 mm] for 3208 and 6309 bearings, respectively [24]. (DER: dark-etching region, WEB: white-etching band.)

Damage type	p_0 / GPa	N / cycles	Speed / Hz	Bearing type	Reference
-	2.62	2.55×10^9	177	3208	[23]
-	2.90	6.37×10^8	177	3208	[23]
-	3.27	7.44×10^8	124	6309	[25]
DER	3.24	6.37×10^7	165	6309	[23]
DER	3.28	1.44×10^8	496	6309	[26]
DER	3.28	$> 4.96 \times 10^8$	496	6309	[5]
DER	3.59	$6.37 imes 10^7$	177	3208	[23]
DER	3.72	$> 1.49 \times 10^7$	496	6309	[5]
DER+WEB	3.24	1.27×10^9	177	3208	[23]
WEB	3.27	4.81×10^8	802	6309	[27]
WEB	3.27	1.90×10^9	802	6309	[27]
DER+WEB	3.27	1.34×10^9	802	6309	[25]
DER+WEB	3.27	1.51×10^9	802	6309	[25]
$DER+30^{\circ} WEB$	3.28	2.26×10^8	496	6309	[26]
$DER+30^{\circ} WEB$	3.28	$> 1.49 \times 10^9$	496	6309	[5]
$DER+30^{\circ} WEB$	3.72	$>4.96 imes10^8$	496	6309	[5]
$DER+30^{\circ}/80^{\circ} WEB$	3.28	4.15×10^8	496	6309	[26]
$DER+30^{\circ}/80^{\circ} WEB$	3.28	$> 4.96 \times 10^9$	496	6309	[5]
$DER+30^{\circ}/80^{\circ} WEB$	3.72	$> 1.49 \times 10^9$	496	6309	[5]
DER $+30^{\circ}/80^{\circ}$ WEB	3.80	3.27×10^9	496	6309	[28]



Figure 2.11: Maximum contact pressure and number of cycles under which microstructural damage is observed in the full endurance bearing tests given in Table 2.3.

Jones suggested that it was caused by repeated heating on cyclic pressure [21], plastic flow seems to be the most reasonable origin, since DER formation is never observed at the surface and its threshold stress exists (Fig. 2.11) [23, 29]. Moreover, Johnston *et al.* reported that DERs always occurred in the bearing tests with a grooved inner ring while they did not appear often with a cylindrical inner ring [30]. Considering that similar amount of heat would be generated in both cases, it implies that DER formation is attributed to plastic deformation, rather than temperature increase during the test. During tempering, carbide particle size increases by the transport of C atoms in the matrix, which brings the increase in matrix/carbide interfacial area. Since DERs manifest themselves with both picral and nital, which are known to reveal carbide/ferrite interfaces [31], it implies that stronger etching can be attributed to larger carbide. In addition, it results in a reduction of C concentration in solid solution which explains the observed hardness decrease in such regions [5, 21, 23, 26]. Based on these suggestions, hardness evolution in a DER is modelled within the framework of dislocation assisted tempering

Table 2.4: Dimensions of white-etching bands [5].

Type	Length / μm	Width / μm	Spacing / μm
30°	5 - 30	0.1 – 0.5	0.5 - 10
80°	100	10	5 - 50

in Chapter 4. It should be noted that the carbide size increase is referred to as carbide "coarsening" in this study. However, this is different from the particle coarsening in the classical diffusional precipitation in that carbide volume fraction is not constant in this case; the size of all particles increases, resulting in larger volume fraction of carbide in a DER.

2.3.2 White-etching bands

WEBs are characterised by a light contrast when examined under an optical microscope and a smooth protrusion by a scanning electron microscope (Fig. 2.12b) [5, 21, 23, 25, 29]. If shaped in three-dimensions, each band assumes a plate which is horizontal in the axial section and inclined to the over-rolling direction in the circumferential section [5, 21, 23, 25, 26, 28, 29]. WEBs can be classified into two types by their angle to the surface: 30° (in the range of 20° – 35°) and 80° (in the range of 65° – 85°). The dimension of a 80° WEB is larger than that of a 30° (Table 2.4) and these "bands" have a large aspect ratio as their name suggests. An overall morphology of both WEBs is schematically shown in Fig. 2.12a.

As seen in Table 2.3 and Fig. 2.11, WEBs are usually observed after DER formation. In this case, WEBs lie within the DER and 80° WEBs form by interpenetrating the 30° WEBs (Fig. 2.12). However, a DER does not have to precede them; Lund claimed that WEBs occur on the soft matrix and the DER formation is not a necessary condition for WEB generation in an initially soft microstructure [26].

It is well known that WEBs consist of cellular structures with size of 50–100 nm [25, 32] without carbide particles [25, 27, 33]. Another interesting feature is that carbides form an elongated thin layer around the bands [5, 25, 32].



Figure 2.12: (a) A schematic showing an overall morphology of white-etching bands. (b) An optical micrograph of white-etching bands with a dark-etching region adapted from [5].

Since WEBs only form after long operation, their formation mechanism has not been included in this study.

2.3.3 White-etching areas

WEAs form partially enveloping and spreading from nonmetallic inclusion particles and cracks (Fig. 2.13) [22, 34]. The WEA around an inclusion is often called 'butterfly' due to its shape. Unlike DERs or WEBs, WEAs are scattered right from the surface to 1.5 mm underneath, which further grows with the running time. Butterflies are inclined ~45° from the surface towards the over-rolling direction and their size is mostly between 5 and 15 μ m [22]. The formation of WEAs is not chronologically related to that of DERs and WEBs; it can be as early as 10⁵ cycles under 3.43 GPa while the DER and WEBs occur only after 10⁷ and 10⁸ cycles, respectively [35].

As in WEBs, the nanocrystalline structures without carbide particles are observed in



Figure 2.13: (a) A scanning electron micrograph of white-etching area around an Al_2O_3 inclusion highlighting a butterfly feature adopted from [34]. (b) An optical micrograph of white-etching area adjacent to the crack.

WEAs [34, 36]. The cell size depends on the distance from the crack defining one of the butterfly boundaries; cells are 10–50 nm near the crack and become 500–1000 nm away from it [37].

Interestingly, Sugino *et al.* have claimed that the microcracks are prerequisite of WEA formation [22]. In this sense, a WEA seems to form adjacent to microcracks due to stress concentration. Cellular structures by dynamic recovery can take place in the presence of high degree of deformation. Moreover, the carbides can become unstable and dissolve due to the deformation. It is supported by the fact that butterfly formation is suppressed in DERs where stress concentration and microcrack formation can be reduced due to their softness [22].

It is in this context that the stress concentration at the cracks and inclusions and a detailed WEA characterisation are studied in Chapter 5. In addition, two models explaining carbide dissolution are suggested in Chapter 6.

2.4 Stress state and microstructural damage

Although there have been a number of suggestions about the responsible stress for microstructural damage in bearings [38], they have only taken the elastic contact theory (Section 2.2.1) into account. To consider cyclic plasticity, other studies used the finite element method, which demands extensive calculations [39, 40]. In this section, a simple reasoning has been made to clarify the material response during bearing operation. Moreover, it is intended to relate the stress state to microstructural damage in bearings.

For a long time, there has been controversy in determining the responsible stress for the microstructure damage observed in bearings [5, 23, 38, 41]. However, it could not be verified by experiments because the depths of the possible stresses were so close with each other and the damage appears in such a large depth range that they cannot be corresponded. Based on the literatures reviewed above, it is claimed that the responsible stress can only be explained when residual stresses are considered; residual stresses should be added to the stress component in Eqs. 2.7-2.9 and new τ_1 calculated with these stresses will be the responsible stress for the damage [17, 18]. To follow this scheme, the residual stresses in 2D contact system (Fig. 2.3) are [17]

$$(\sigma_x)_r = (\sigma_x)_r (z), \ (\sigma_z)_r = (\tau_{xz})_r = 0,$$
 (2.14)

assuming no traction, and steady and continuous deformation for the flat surface. Therefore, Eqs. 2.8 and 2.9 remain the same, while Eq. 2.7 becomes

$$\sigma'_x = \sigma_x + (\sigma_x)_r \,. \tag{2.15}$$

By replacing σ_x with σ'_x in Eq 2.10, modified principal shear stress, τ'_1 is obtained. For example, the stress state along x-axis in the case of $(\sigma_x)_r = 250$ MPa and $p_0 = 3.3$ GPa at z/a = 0.51 is shown in Fig. 2.14b. Compared with the stress state without $(\sigma_x)_r$ (Fig. 2.14a), τ'_1 has the maximum value of 0.26 p_0 at x = 0.70a with $\theta = 12.6^{\circ}$ and 77.4° , while τ_1 has the maximum value of 0.28 p_0 at x = 0.40a with $\theta = 29.6^{\circ}$ and 60.4° . In other words, the principal shear stress during the contact has been reduced accompanying direction change of the principal shear stress plane. In this way, the material can minimise the principal shear strain with the aid of residual stresses; however, the limit would be



Figure 2.14: Variation of stresses at a constant depth z/a = 0.51 (a) without residual stresses, (b) with $(\sigma_x)_r = 250$ MPa under $p_0 = 3.3$ GPa.

 τ_{xz} which cannot be reduced by residual stresses.

Adopting this approach, the residual stress reported according to the running revolutions in [4] are added to the stress to calculate the change in τ'_1 and θ as in Fig. 2.15. Note [4] employed a ball bearing and discrepancy may occur since the calculation is based on 2D contact geometry. In addition to the fact that residual stress dramatically changes with the occurrence of damage, θ stays near to 0° until the damage appears in both cases. θ being ~0° implies that τ_1 near the origin has been effectively suppressed by the residual stress. When the sign of θ changes to negative, DERs start to be observed from 10⁸ and 10⁷ revolutions for $p_0 = 3.2$ GPa and 3.7 GPa, respectively. It seems that in the early stages of rolling contact fatigue, residual stress develops up to the level which moves the x coordinate of the maximum τ'_1 location from 0 to near that of maximum τ_{xz} . However, as the residual stress further develops, the maximum τ'_1 location passes this point and decreases θ below zero. Interestingly, when WEBs form, residual stress stops dropping down causing τ'_1 decreases near to maximum τ_{xz} again, while without WEBs formation, residual stress is dramatically reduced resulting in increasing τ'_1 .

Now, the material response during rolling contact fatigue can be determined. Before the DER formation, τ'_1 stays near the maximum $\tau_{xz} = 0.25p_0$ (Table 2.2). This becomes 0.80 GPa for $p_0 = 3.2$ GPa and 0.93 GPa for $p_0 = 3.7$ GPa. k_y for 52100 martensitic steel



Figure 2.15: The evolution of (a,b) residual stress $((\sigma_x)_r)$ [4], (c,d) principal shear stress (τ'_1) , (e,f) the angle between the principal shear plane and the surface at z = 0.20 mm. (g,h) is microstructural damage occurring over the depth [4]. (a,c,e,g) is for $p_0 = 3.2$ GPa and (b,d,f,h) is for $p_0 = 3.7$ GPa. LAB and HAB corresponds to 30° and 80° WEB, respectively.

Table 2.5: Yield stress, hardness, shear yield stress (k_y) , and elastic shakedown limit $(4.7k_y)$ of 100Cr6 martensitic steel.

Reference	Proof stress / GPa	k_y / GPa	$4.7k_y$ / GPa
[15]	1.5-1.7 (at 0.2% strain)	0.87 – 0.98	4.1 - 4.6
[40]	$1.66 \ (at \ 0.2\% \ strain)$	0.958	4.50

can be estimated from its yield stress and relevant values are shown in Table 2.4 [42]. It is seen that τ'_1 is lower than k_y in both cases; therefore, it can be said that materials show elastic shakedown when DERs have not occurred (Fig. 2.8). When DERs form, the response of the material becomes different in each case; τ'_1 is increased dramatically exceeding k_y and this results in plastic shakedown for $p_0 = 3.2$ GPa (Fig. 2.9b,2.15c). On the other hand, τ'_1 is increased and decreased again for $p_0 = 3.7$ GPa, i.e. the material experiences plastic shakedown and comes back to elastic shakedown (Fig. 2.9b,2.15d). This behaviour seems to be caused by WEB formation (Fig. 2.15f). In this study, it is assumed that the material undergoes plastic shakedown during RCF when damage forms, and τ'_1 at this point corresponds to the maximum $\tau_{xz} = 0.25p_0$ (Table 2.2).

When the material is subjected to plastic shakedown, it shows closed stress-strain loop (Fig. 2.16). There are two kinds of strain of opposite direction with equal magnitude in it. Despite the absence of macroscopic strain in principle, the material experiences plastic deformation inducing microstructural changes. Since plastic strain is the result of dislocation motion, some of forward motion may be reversed by opposite motion. Therefore, the plastic strain range per cycle will be less than \overline{AB} , but greater than zero. Here, the half shear plastic strain range per cycle is defined as $\Delta \gamma_C$ which is equal to $\frac{1}{2}\overline{AB}$ in Fig. 2.16 and this is considered as the strain responsible for the plastic deformation. The value of $\Delta \gamma_C$ is very important when the Orowan equation is adopted to relate macroscopic strain to microscopic dislocation glide. In this respect, a mechanical test to determine $\Delta \gamma_C$ using a laboratory fatigue tester is introduced in Section 3.3.



Figure 2.16: Closed shear stress (τ) -strain (γ) loop showing plastic shakedown for symmetrical loading. τ_a is shear stress amplitude.

2.5 Summary

For through-hardened bearings, martensitic 100Cr6 steel has been most popularly employed. During their operation, cyclic rolling contact occurs and the corresponding stress state can be best described when Hertzian elastic contact theory is combined with cyclic plasticity. As a result, subsurface damage such as DERs, WEBs, and WEAs appear, which can promote spalling at failure. Such damage forms are related to carbide coarsening and dissolution as well as dislocation generation/rearrangement in the microstructural scale.

However, studies on the stress state and microstructural changes have not been related closely. Here, it is suggested that the one way to connect them is via residual stress developed during the bearing operation. By considering the residual stress, it is seen that the magnitude of the responsible shear stress (τ'_1) can be reduced to the maximum orthogonal shear stress (τ_{xz}). Moreover, the material response including its hysteresis loop for cyclic stress-strain can be comprehended and further be related to microstructural changes.

Chapter 3

Laboratory testing strategies

The main aim of this chapter lies on introducing bearing testing strategies, and on examining the possibility to appraise the material behaviour in applications subjected to rolling contact fatigue (RCF). Furthermore, the fatigue strain is measured, as this value can be employed as an input to the microstructural alteration models. In Section 3.1, the test methods which are most commonly used for for RCF testing are introduced. In Section 3.2, a ball-on-rod RCF test is presented, which is compared with a repetitive push test introduced in Section 3.3.

3.1 Test methods for rolling contact fatigue

In order to assess the life of bearing steels, a number of test methods are suggested. The best would be the one that can represent the real bearing operation conditions appropriately. During bearing operation, subsurface spalls are initiated after long periods of rolling contact, often up to several years [1], which is too long for the testing of various specimens. In this context, accelerated tests have been introduced, adopting either higher contact pressures or higher rotational speeds than normal operating conditions. During testing, the most important issue is to induce damage at the subsurface, preventing that initiated from the surface. To take this into account, the elastohydrodynamic lubrication condition is assessed by λ , the ratio between the minimum film thickness (h_{min}) and the

sample roughness $(h_{roughness}^*)$, which is given as [43]

$$\lambda = \frac{h_{min}}{h_{roughness}^*},\tag{3.1}$$

where

$$h_{min} = 1.38 \left(R^*\right)^{0.27} \left(\alpha \mu_0 v\right)^{0.73} \left(\frac{p_0}{E^*}\right)^{-0.19}.$$
(3.2)

Here, the expression is for elliptical contact surface of a Polymet RCF rig [44]; E^* and R^* are the reduced elastic modulus and radius, which are defined in Eqs. 2.5 and 2.6, respectively; α is the pressure viscosity coefficient, μ_0 is the dynamic viscosity of contacting surfaces, v is the velocity at the surfaces, p_0 is the maximum contact pressure, and $h^*_{roughness} = \sqrt{h^2_{roughness,1} + h^2_{roughness,2}}$ where $h_{roughness,i}$ is the roughness of each body, i = 1, 2, in contact. With the calculated λ value, the lubricated contact condition is determined [11]; Regime I ($\lambda < 1$) is where the two bodies come into partial metal to metal contact. Regime II ($1 < \lambda < 3$) is where the degree of metal to metal contact is limited. Regime III ($\lambda > 3$) is where metal to metal contact is avoided, and it is this region that subsurface RCF is most likely to occur. Based on Eqs. 3.1 and 3.2, it is seen that high speed would actually result in decelerating the test; the test lasts even longer because of larger λ [1]. Therefore, only high pressure is used in order to accelerate the tests.

A good way to simulate the conditions experienced by real bearings is by a full-scale endurance test; e.g. SKF R2 rigs (Fig. 3.1). It can simulate axial/radial loading conditions with various operating speeds. Despite its ability to reproduce the real bearing conditions, it is a very expensive and time-consuming method due to the high quality level of the test bearings. Since the lives of bearings are scattered, a large number of tests should be carried out for reliable statistical analysis, which makes the full scale test more costly. In this perspective, bearing element tests are performed, employing a simple specimen geometry. Popular tests include (i) five-ball fatigue test [45], (ii) flat washer test [46], (iii) Polymet RCF test [44, 47], and (iv) ball-on-rod RCF test [48].



Figure 3.1: A schematic cross section of an SKF R2 endurance test rig [49]. Test bearings are coloured.

As its name suggests, the test specimen assembly of a five-ball fatigue tester is composed of five balls; a cross section is shown in Fig. 3.2. Four balls are positioned equivalently, being spaced by a separator and with one ball being positioned against them at a contact angle, β . The upper ball is rotated by the drive spindle and failure usually occurs at the upper ball, since its stress frequency is higher than the lower balls. Loading is controlled by a dead weight and the lubrication is maintained by a tube directed to the upper ball. One advantage is that the testing temperature can be controlled; it can be varied from -184 °C up to 1093 °C [45]. It is reported that β influences the test results (life) via sliding, traction force, and λ [45]. If β increases, the sample life tends to decrease while the operating temperature increases.

In the flat washer test, flat washers with 74.6 mm-outside diameter, 50.8 mm bore, and 6.35 mm width are used as test components. Acting as a pair, one slave washer and one test washer is assembled with 16 balls of 7.94 mm-diameter (Fig. 3.3) [46]. Loading is by a dead weight system through a ball sunk into a hole, and the lubrication is fed by



Figure 3.2: A schematic cross section of a five-ball fatigue tester [45, 49]. The contact angle (β) between the test ball (red) and lower balls (blue) is shown.

a pump. The test is stopped automatically when vibration is higher than the set level.

A cylindrical sample with 9.53 mm diameter and 76.2 mm length is tested in the Polymet RCF test [47]. The sample drives two rollers whose edges are hemispherical as shown in Fig. 3.4. These rollers apply load onto the sample and lubrication is dropped from the top of the test rig. The generated spalls are found to be similar to the ones shown in full scale endurance testing [44, 47].

In the ball-on-rod RCF test, a cylindrical sample is radially loaded by three balls which are spaced by a separator (Fig. 3.5), which enables to test both the rod and balls. The sample diameter is the same with the one in the Polymet RCF testing for the rod and the diameter of the balls is 12.7 mm. In order to reduce the testing time reasonably and prevent the ball failure, the balls are purposely roughened to 0.089 μ m [48]. Spalling is detected by monitoring the vibration and lubrication is drip fed as in Polymet RCF testing.

Common conditions for these test methods are given in Table 3.1. Comparing with



Figure 3.3: A flat washer test assembly [46]. The test washer is located on the central driven shaft.



Figure 3.4: (a) The cross section of a Polymet rolling contact fatigue tester and (b) a roller used in the tester [47, 49]. A rod specimen is coloured in red in (a).



Figure 3.5: Schematics of (a) an upper view and (b) a side view of a ball-rod rolling contact fatigue tester [48].

Testing Method	p_0 / GPa	Speed / Hz	Reference
Five-ball fatigue testing	5.52	$\sim 250, \sim 500$	[45, 47]
Flat washer testing	4.17	200	[46]
RCF (Polymet) testing	4.83	333 - 1667	[44]
Ball-on-rod RCF testing	5.52	143	[48]

Table 3.1: Normal conditions for bearing element testing methods. (p_0 : Maximum contact pressure)

the conditions for bearing endurance tests for microstructural alteration in Table 2.3, the operation speed is lower for flat washer and ball-rod RCF tests, while others are comparable. A lower speed may result in smaller λ and metal to metal contact [1]. Therefore, appropriate lubrication selection is essential to control λ . Moreover, it is clear that all tests are based on high p_0 to accelerate the tests, which implies that the raceway develops a plastically deformed region during testing, which is not normally the case with full-scale bearing tests, and may consequently cause different results.

It seems that the application of these tests is limited to the initial screening of either materials or running conditions. Aspects of the materials reflected by the test duration are hardness, composition, heat treatment and cleanliness; whereas the running conditions include lubrication type, additives, and temperature [1, 44–46]. However, the accuracy in life rank has always been into question since performance results may not be correlated among different testing strategies [1].

Although many RCF test results have been reported, only a few have focused on microstructural damage. Sugino *et al.* reported butterfly formation during rolling contact between two tapered disks under 4.9 GPa-contact pressure [22]. Johnston *et al.* pointed out that the dark-etching region is not often seen during testing with a cylindrical inner ring without grooves. If seen, it appears much later than that in deep groove bearings [30].

It is in this context that microstructural damage during the ball-on-rod RCF test is studied in the next section. If the testing method can reproduce the test conditions in real bearings, dark-etching regions as well as white-etching bands/areas would appear. Such systematic testing would also clarify which features cause subsurface spalls in the specimens.

3.2 Ball-on-rod rolling contact fatigue test

The ball-on-rod RCF test is one of the bearing element tests described in the previous section. The possibility for obtaining microstructural damage similar to the actual bearing operation is discussed.

3.2.1 Experimental

3.2.1.1 The test rig

The ball-on-rod RCF test rig employed in this study was supplied by Delta Research Corporation. Four stations are available in the machine (Fig. 3.6a). Photographs of its several components are shown in Fig. 3.6b–f, and cross sections of the main part are presented in Fig. 3.7.

Each station consists of a collet and a main part, which are separated by a spacer (Figs. 3.6b–d, 3.7a). The collet is attached to the station and fixes the rod sample which is driven by a motor under the station. The main part is composed of (i) an upper cup housing, (ii) a spring retainer plate, and (iii) a lower cup housing (Figs. 3.6d–f, 3.7b). Three balls in a retainer are placed between the cups of their upper and lower housings. The lower cup housing, the spring retainer plate, and three springs are attached to each other by bolts. The spring retainer plate has a hole in the middle, which is matched to the cup of the lower cup housing (Fig. 3.6f). When the upper cup housing is properly fitted along with the balls and retainers to the main part by another set of bolts, the balls are equivalently in contact with the cups and a gap is created between the lower cup housing and the spring retainer plate. If the rod specimen is correctly positioned, it is radially loaded by these three balls via spring force as shown schematically in Fig. 3.8.

The geometry enables the springs to apply a vertical force, F_{spring} , on the cups, which



Figure 3.6: (a) Overview of the ball-on-rod RCF test rig [50], (b) collet, (c) spacer, (d) main part, (e) an assembled station, and (f) main part without the upper cup housing. Four stations are marked in yellow in (a).



Figure 3.7: (a) Cross sections of the ball-on-rod rig main part and collet. (b) Component details of the main part [48].



Figure 3.8: (a) A schematic showing the force applied to the cups and the sample by springs. (b) Results of the spring force [48]. F_{spring} is the applied spring force; F_{result} is the resultant force; F_{rad} is the radial force on a rod sample.

is the vertical component of F_{result} . As a result, its horizontal component, F_{rad} , which is the force acting on the rod sample, is calculated by,

$$F_{rad} = \frac{F_{spring}}{\tan\left(25.083^\circ\right)}.\tag{3.3}$$

Assuming (i) perfectly smooth contact surfaces, (ii) isotropic linearly elastic bodies, and (iii) no frictional forces, the total load (P) on the rod from one ball equals to $2F_{rad}$ and the contact pressure (p_0) can be obtained from Hertzian contact theory as [17],

$$p_0 = G_3 \left(\frac{6PE^{*2}}{\pi^3 R^{*2}}\right)^{\frac{1}{3}} = G_3 \left(\frac{12F_{rad}E^{*2}}{\pi^3 R^{*2}}\right)^{\frac{1}{3}},$$
(3.4)

where G_3 is a geometry factor which is the function of radius of the rod and balls; here it is taken to be 1.0226 [17]. E^* and R^* are the reduced elastic modulus and radius; while E^* is given from Eq. 2.5, R^* is calculated differently from Eq. 2.6 in this case considering the contact between a cylinder and a sphere with [17],

$$\frac{1}{R^*} = \sqrt{\frac{1}{R_1^2} + \frac{1}{R_1 R_2}},\tag{3.5}$$

where R_1 and R_2 are the radii of the the balls and rod sample, which are 6.35 mm and 4.75 mm, respectively. Since both the sample and the balls are steels, the elastic moduli and Poisson ratios are taken to be $E_1 = E_2 = 210$ GPa and $\nu_1 = \nu_2 = 0.3$. *P* is adjusted with the load cell installed on the test rig. The load cell measures the total load applied by three springs on one cup, which is equal to $F_{cell} = 3F_{spring} = 3F_{rad} \tan (25.083^{\circ})$. Therefore, Eq. 3.4 can be rewritten as:

$$p_0 = G_3 \left(\frac{4F_{cell} E^{*2}}{\pi^3 R^{*2} \tan\left(25.083^\circ\right)} \right)^{\frac{1}{3}},\tag{3.6}$$

and p_0 is plotted with respect to F_{cell} in Fig. 3.9. In this plot, the lower axis is F_{cell} given in N while the upper axis is in lb which is the number seen in the actual screen of the test rig. Since p_0 is proportional to $F_{cell}^{\frac{1}{3}}$, p_0 varies strongly with F_{cell} up to ~3.5 GPa. Considering that the spring load may oscillate over the long test period, a low contact pressure ($p_0 < 3.5$ GPa) is not recommended.

3.2.1.2 Microscopy

Before and after the tests, the sample microstructures are studied by optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The test produces grooves on the surface of the rod samples, which represent the area in contact with the balls (Fig. 3.10). To investigate the unaffected region, the end of the rod was cut into a plate, one side of which was around 15 mm. To obtain the deformed area, the volume including the groove was cut and both axial and circumferential sections were observed as shown in (Fig. 3.11). After being cut, the samples were cold-mounted, mechanically ground using silicon carbide paper, and polished with 6 μ m and 1 μ m diamond suspensions. Then, the final polishing was carried out with colloidal silica before



Figure 3.9: Sensitivity of the contact pressure (p_0) to the load on screen (F_{cell}) . etching with 2% nital.

For OM and SEM, a Leica DM 2500M microscope and a JEOL 5800LV were employed, respectively. Secondary electron imaging with SEM employed a 10 keV electron beam under working distance of 5–10 mm. In some micrographs, the angle of the cracks adjacent to the inclusion was measured with the respect to the contact surface (Fig. 3.12). When multiple cracks were observed around one inclusion, all angles were counted separately, while the ones with the same angle were considered to be a single crack. Moreover, residual cementite volume fraction was determined by the point counting method from a SEM image covering $100 \times 120 \ \mu m^2$.

Energy dispersive spectroscopy (EDS) in SEM was used to identify the inclusion type with a 15 keV-electron beam and a 10 mm-working distance. EDS has some limitations which may affect the results during quantitative analysis. When the electron beam reaches the sample, the electrons interact with an interaction volume as shown in Fig. 3.13, and the penetration depth depends on both electron beam energy and the specimen atomic number as shown in Fig. 3.14. EDS adopts characteristic X-rays coming from the largest part of the interaction volume; for 15 keV, the penetration depth is approximately 500 nm.



Figure 3.10: Ball-on-rod RCF tested samples. The grooves due to testing are clearly observed.



Figure 3.11: The sample preparation procedure for metallography. Possible affected regions are coloured with grey.



Figure 3.12: Determination of the crack angle (θ_{crack}) with respect to the specimen surface on the circumferential section. Both examples of positive and negative angles are shown.



Figure 3.13: Schematic illustration of the volume of material that is probed by an incident electron beam together with the volumes from which X-rays and backscattered Auger/secondary electrons emanate [51].

Hence, if the analysed particle is smaller than 500 nm, some part of the signal may come from the surrounding matrix. Another limitation is that C cannot be quantitatively analysed owing to C contamination in the chamber. Hydrocarbon in the vacuum pump attached to SEM can be decomposed into H and C by the high electron energy, resulting in C atoms being accumulated on the specimen during the analysis.

The unaffected regions were observed by TEM. Samples were firstly cut into thin plates with ~0.6–1 mm-thickness. Then, 3 mm-diameter discs were obtained via electrical discharge machining. The discs were ground to 80–90 μ m and subsequently electropolished with 15% perchloric acid with ethanol at 15 °C. The microstructure was observed with a Philips CM30 with a 300 keV-electron beam.

3.2.1.3 X-ray Diffraction

The XRD technique was employed in order to identify the amount of austenite in the undeformed region. The samples were prepared by grinding and polishing in the same way as described in the previous section, but without etching. The data were collected by



Figure 3.14: The penetration depth of electrons (a) with respect to the electron energy in a stainless steel and (b) with respect to the atomic number of the sample for 10 keV, 20 keV and 30 keV electron beams [51].

Philips PW1820 using Cu K α radiation at an operation voltage of 40 kV and a current of 40 mA. The 2 θ range was 35°–125° with a 0.04° step size and 5 s-dwell time per step. The austenite phase fraction was then estimated by Rietveld refinement.

The principle of Rietveld refinement is to acquire the best fit of the data by refining a number of experimental parameters step by step. The goodness of the fit is assessed by χ^2 , which is calculated as:

$$\chi^{2} = \sum_{i} w_{i} \left(y_{i} - y_{ci} \right)^{2}, \qquad (3.7)$$

where y_i is the observed intensity at a data point *i*, y_{ci} is the calculated intensity at *i*, and w_i is a weighting parameter which is given as the inverse of the variance of y_i . y_{ci} is expressed as:

$$y_{ci} = \sum \left\{ S_j \left| F_{kj} \right|^2 \phi_{ikj} \left(2\theta_i - 2\theta_k \right) L_{kj} M_{kj} T_{oj} P_{kj} \right\}^2 + y_{bi}$$
(3.8)

where k is the index of overlapping neighbouring Bragg reflections, S_j is the scaling factor for phase j, F_{kj} is the structure factor, ϕ_{ikj} is the profile function, L_{kj} is the Lorentz polarisation factor, M_{kj} is the multiplicity, T_{oj} is the overall temperature factor, P_{kj} is the preferred orientation function, and y_{bi} is the background intensity at data point *i*. When refinement is completed, F_{kj} can be used to determine the crystallography including lattice parameters and atomic positions; ϕ_{ikj} is adopted in analysis of particle size and strain. S_j is used to determine the weight fractions of the phases, W_p , as follows:

$$W_p = \frac{S_p N_{form} M_p V}{\sum_j S_j (N_{form} M_p V)_j}$$
(3.9)

where N_{form} is the number of formula units per unit cell, M_p is the mass of the formula unit, and V is the unit cell volume.

The refinements were carried out by FullProf version 0.50 software. Since the scan rate was not enough to quantify carbide phases, only austenite and martensite were introduced during the refinement. All lattice and profile parameters of both phases were refined.

3.2.1.4 Microindentation hardness

Microstructural damage induces the changes in mechanical properties including hardness. Since microstructural damage is expected to occur in a small area during RCF testing, microindentation hardness is a good and simple method to track the alteration in mechanical properties.

A 0.3–1 mm-thick groove is detected by the naked eye after each test (Fig. 3.10). The size of the contact area (contact thickness) can be estimated from Hertzian elastic theory. For two-dimensional line contact, the contact thickness (2a) is estimated from Eqs. 2.3, 2.4 as,

$$2a = \frac{4p_0 R^*}{E^*},\tag{3.10}$$

while that in three-dimensional contact for the pressure applied to circular contact region

can be expressed as [17],

$$2a = \frac{\pi p_0 R^*}{E^*}.$$
 (3.11)

By employing Eqs. 3.10 and 3.11, the contact thickness is plotted with respect to p_0 in Fig. 3.15a. Considering that the contact area nature of the ball-on-rod RCF test would be between two and three dimensional, and $p_0=3.5-6$ GPa, the estimated contact thickness lies between 0.40–0.85 mm which is consistent with the observed groove thickness. Moreover, depths of maximum shear stresses are calculated with the aid of Eqs. 3.10, 3.11, Table 2.2, and [17], and the results are plotted in Fig. 3.15b. Considering $p_0=3.5-6$ GPa, maximum τ_1 occurs at a depth of 200–340 μ m and 95–160 μ m for two and three dimensional contact, respectively; and the maximum τ_{xz} occurs at 125–215 μ m depth for two dimensional contact. With the calculated contact thickness and the depth of the maximum shear stresses, the possible damaged area is schematically shown by the grey area in Fig. 3.16. In order to get the hardness evolution under the surface with the indentation spacing of 50 μ m in depth direction up to 750 μ m below the surface, fifteen indentations smaller than 50 μ m have been made in three columns and five rows.

For a given Vickers hardness, the relationship between the load (L) and indent size (d) is expressed as [52]

Vickers hardness =
$$\frac{2L\sin\left(\frac{136^\circ}{2}\right)}{d^2}$$
, (3.12)

where d is an arithmetic mean of two diagonals of the indentation, and is plotted in Fig. 3.17 for a Vickers hardness of 600–850. From the plot, $d = 20-50 \ \mu\text{m}$ can be obtained with $L \sim 200-800$ g. Since larger indents can give higher accuracy, 500 g is chosen, which results in $d \sim 30-40 \ \mu\text{m}$. Note that the minimum recommended spacing between the indents are $2.5 \times d$ [52], which is satisfied in this case.

All microindentation hardness data were obtained employing a Mitutoyo MVK-H2 tester with a 15 s dwell time. A total four sets of measurements were made for each test



Figure 3.15: (a) The contact thickness and (b) the depth for maximum shear stresses for two and three dimensional (2D and 3D) contact based on Hertzian elastic contact theory.



Figure 3.16: Microindentations made under the surface of the axial section. A possible RCF affected zone is shown in grey.



Figure 3.17: The indent size (d) range with respect to the load (L) of Vickers hardness. The number adjacent to the line corresponds to the hardness value.

condition.

3.2.1.5 Test conditions

The steels used in the testing were 100Cr6 (Table 2.1) and provided by SKF. They were partially austenitised at 860 °C for 20 minutes and quenched in oil at 60 °C for 10 minutes before rinsing in cold water. Then, tempering was carried out at 160 °C for 90 minutes.

A rotation speed of 3600 rpm and a lubrication feed rate of 10 drops per minute were kept as reported by Glover [48]. Since the specimen experiences 2.389 cycles per revolution [48], the test speed corresponds to 143.3 Hz. The balls were changed after each test, and the cups were changed after every four tests. The balls are made of 100Cr6 with an arithmetic average roughness of 0.020 μ m, resulting in a root mean square roughness of <0.025 μ m. Such roughness is lower than that in [48], which is intended to prevent surface initiated failure. The root mean square roughness of the rod sample was measured by a Veeco Dektak 6M stylus profiler with a 12.5 μ m-diameter diamond tip and 10 mgforce. Four measurements were made; each measuring roughness of a 0.5 mm-line in the longitudinal direction of the rod and the roughness was 0.090 \pm 0.020 μ m. For lubrication, BP Turbo Oil 2380 with a density of 9.749 × 10² kg m⁻³ at 15 °C was used.

Table 0.2. The Histophy property of Di	10100 011 20	ee [ee, er]:
Property	at 40 $^{\circ}\mathrm{C}$	at 100 $^{\circ}\mathrm{C}$
Pressure viscosity coefficient (α) / Pa ⁻¹	1.50×10^{-8}	1.17×10^{-8}
Kinematic viscosity / $mm^2 sec^{-1}$	24.2	4.97



Table 3.2: The viscosity property of BP Turbo Oil 2380 [53, 54]

Figure 3.18: The ratio (λ) between the minimum lubrication film thickness and the sample roughness with respect to contact pressure in the ball-on-rod rolling contact fatigue testing.

With the viscosity property information given in Table 3.2, and the dynamic viscosity (μ_0) calculated via (kinematic viscosity)×(density), λ can be obtained through Eqs. 3.1 and 3.2 with respect to the maximum contact pressure (p_0) , and is plotted in Fig. 3.18. If p_0 increases from 3.5 to 6 GPa, λ decreases from 1.25 to 1.14 at 40 °C. Since λ is larger than 1 but smaller than 3, a small degree of metal to metal contact is expected (Section 3.1). Note that λ is sensitive to the testing temperature; it can be reduced far below 1 to ~ 0.3 , when the temperature reaches 100 °C.

Tests were carried out at $p_0 = 3.7, 4.2, 4.7, 5.2, \text{ and } 5.6 \text{ GPa}$. 5.6 GPa was the usual contact pressure for the ball-on-rod RCF test in [48], which is much higher than p_0 for the full scale endurance tests (Table 2.3). Therefore, lower pressure conditions have been included in this study. To maintain the stability of p_0 during the test, p_0 was kept greater



Figure 3.19: The initial microstructure of the ball-on-rod RCF test sample after martensitic heat-treatment by (a) secondary electron imaging, and (b) transmission electron bright field imaging.

than 3.5 GPa (Fig. 3.9). For each p_0 condition, samples were run for 10^7 and 10^8 cycles, which took ~19 hours 22 minutes and ~193 hours 47 minutes, respectively. An additional test was performed under $p_0 = 5.6$ GPa for 2.43×10^8 cycles, i.e. 471 hours 18 minutes.

3.2.2 Results

The initial microstructure before the tests was tempered martensite with twinning and temper carbides (Fig. 3.19). It contained 5.1 ± 0.8 vol% residual cementite and 12.8 ± 0.2 wt% retained austenite. χ^2 of Rietveld refinement for austenite was 2.348. Its hardness after the heat treatment was 834 ± 10 HV0.5.

No indication of overall microstructural change has been observed in either axial sections or circumferential sections by OM and SEM. In other words, decay of martensite plates and residual cementite was not detected with OM and SEM.

However, a number of microcracks initiated from inclusions were observed in the cir-



Figure 3.20: Relative population of inclusions with microcracks with respect to depth for each maximum contact pressure. The number of cracks counted for plotting is given in parenthesis in the legend.

cumferential sections for all test conditions. The inclusions with microcracks were counted and their relative population is plotted against depth for each p_0 in Fig. 3.20. Crack distribution is shown in Fig. 3.21 in terms of the depth and angle with respect to the surface; some typical crack morphologies are shown in Figs. 3.22 and 3.23. Some general features observed on the microcracks are:

- The maximum frequency of inclusions bearing microcracks moves to deeper regions with increasing p_0 (Fig. 3.20).
- The cracks are scattered from the surface down to a depth of 600 μ m (Fig. 3.21).
- Most cracks lie within -10° to 20° from the surface. The angle does not seem to be related to crack depth (Fig. 3.21).
- The smallest crack observed is ~1 μm (Fig. 3.22a). However, this might be a small part of the large crack since only two dimensional sections have been investigated. The large crack size is ~15 μm (Fig. 3.22b).


Figure 3.21: Distribution of the cracks initiated from inclusions according to their angle and depth with respect to the surface.

- Cracks are usually created on both sides of the inclusions. However, a crack may be observed on only one side of the inclusion as in Fig. 3.22c, which may be due to the sectioning effect. Sometimes, multiple cracks appear on one side of the inclusion (Fig. 3.22d,e).
- Cracks on either side of the inclusion are sometimes connected by a crack going through the inclusion as shown in Fig. 3.22f.
- White-etching areas (WEAs) are observed adjacent to some cracks (Fig. 3.23); this morphology resembles the butterfly in Fig. 2.13a.
- The inclusion type and size do not seem to be related to crack formation. The composition of the inclusions in Fig. 3.22e,f,g is given in Table 3.3. Cracks have been observed adjacent to MnS, oxysulfide, spinel, and oxide particles irrespective of their size.
- For adjacent inclusions, one of them may be decorated by a crack, while the rest do not (Fig. 3.22h,i).



Figure 3.22: Representative features of microcracks initiated from inclusions during ballon-rod RCF testing. (a,e,f,g) are secondary electron images and (b,c,d,h,i) are optical micrographs. Images are tilted so that the surface lies horizontally in the figure. (p_0 : the maximum contact pressure, N: the number of cycles, z: depth)



Figure 3.23: Enlarged images of Fig. 3.22 b,c showing white-etching areas.

Larger cracks which size exceeds 100 μ m are observed for $p_0 = 5.2, 5.6$ GPa, $N = 10^7$ cycles, and $p_0 = 4.7$ GPa, $N = 10^8$ in the circumferential section. They are straight and mostly parallel to the surface at a depth of ~100–200 μ m (Fig. 3.24a). These cracks may penetrate prior austenite grains but bypass residual cementite particles (Fig. 3.24b). Sometimes, relatively thick cracks occur as shown in Fig. 3.24c. In this case, a larger crack lies parallel to the surface while smaller cracks are not.

Although microstructural alteration was localised adjacent to inclusions, a gradual

1				
	Fig. 3.22e	Fig. 3.22f	Fig. 3.22g-1	Fig. 3.22g-2
Fe / wt%	$2.7{\pm}0.3$	26.3 ± 0.4	$7.7 {\pm} 0.3$	51.2 ± 0.4
Cr / wt%	1.1 ± 0.2	$0.9 {\pm} 0.2$	-	$0.9 {\pm} 0.1$
Mn / wt%	51.5 ± 0.3	$34.9 {\pm} 0.4$	-	-
S / wt $\%$	40.2 ± 0.3	34.5 ± 0.3	-	-
O / wt $\%$	-	$2.9 {\pm} 0.5$	44.7 ± 0.3	$7.9 {\pm} 0.2$
Al / wt $\%$	-	-	$35.3 {\pm} 0.3$	19.2 ± 0.2
Mg / wt%	$2.8 {\pm} 0.1$	$0.5 {\pm} 0.1$	12.4 ± 0.2	20.8 ± 0.4
Ca / wt $\%$	$1.6 {\pm} 0.1$	-	-	-
Type	MnS	Oxysulfide	Spinel	Spinel

Table 3.3: Composition of some inclusions shown in Fig. 3.22 by EDS and their expected inclusion types.



Figure 3.24: Cracks created in the circumferential section during the ball-on-rod RCF testing with (a) $p_0=4.7$ GPa for 10^8 cycles, (b,c) $p_0=5.2$ GPa for 10^7 cycles. Images are tilted so that the surface lies horizontally in the figure.

change in microindentation hardness with depth (z) has been observed. The effect of N and p_0 is shown in Figs. 3.25 and Fig. 3.26, respectively. The trend in hardness evolution is similar for all test conditions. The hardness reaches its maximum at $z \sim 100$ μ m, then decreases to the value of the unaffected region at $z > 350 \ \mu$ m. The maximum hardness depth corresponds with the depth showing either maximum τ_1 for the three dimensional contact or maximum τ_{xz} for the two dimensional contact (Fig. 3.16). For all p_0 conditions, the effect of N is negligible (Fig. 3.25), which suggests that hardness increases at its maximum stress at the early stages before $N = 10^7$ cycles, and is retained throughout the test. In Fig. 3.26, it is clearly shown that the maximum hardness value increases according to p_0 ; when $p_0 = 5.6$ GPa, HV0.5 reaches up to ~900. In addition, the range showing hardness augment is broadened with increasing p_0 . This is attributed to the fact that greater p_0 causes larger contact area dimension (a) (Fig. 3.15a) and the stress affected region is extended deeper under the surface (Fig. 2.4).

3.2.3 Discussion

Dark-etching region (DER) formation has not been observed during the ball-on-rod RCF testing. One reason might be higher pressure than that for the full bearing endurance test (Table 2.3, Fig. 2.11). Another reason could be the lower testing speed (143.34 Hz); it has been reported that testing with lower speed did not produce DER formation [25].

Contrary to lower hardness resulting from DER formation, higher hardness was obtained in the tested specimens. Based on the results of microindentation hardness, the origin of hardness increase without apparent microstructural change can be explained by residual stress development [55] due to: (i) dislocation density increase/rearrangement (strain hardening) and (ii) stress induced martensitic transformation of retained austenite. Dislocation density increases at the early stage of deformation and saturates (steady state) [56]. Likewise, it was previously reported that some amount of retained austenite is transformed to martensite at $N \leq 4.96 \times 10^3$ cycles and then the rest is maintained up to large N (4.96×10^5 – 4.96×10^8 cycles depending on p_0) [4]. Therefore, the evolution



Figure 3.25: Vickers hardness profiles with depth under the raceway for $p_0 = (a)$ 3.7 GPa, (b) 4.2 GPa, (c) 4.7 GPa, (d) 5.2 GPa, and (e) 5.7 GPa. Two grey horizontal lines in each plot represent the hardness range of the unaffected region.



Figure 3.26: Vickers hardness profiles with depth under the raceway for $N = (a) 10^7$ cycles and (b) 10^8 cycles. Two grey horizontal lines in each plot represent the hardness range of the unaffected region.

of both dislocation density and transformed austenite amount agrees with the observed hardness increase.

The microcracks seem to be initiated from inclusions and they may be the precursor of WEAs, since WEAs are never observed without microcracks in this work. Microcracks without WEA were observed more frequently; only 10–20% of the observed microcracks are accompanied by WEAs for all test conditions. This observation is consistent with the study employing thrust type testers where microcracks are claimed to be the prerequisite for WEAs [57]. Also, the fact that these cracks were not observed in the axial section but in the circumferential section suggests that cracks form parallel or slightly inclined with respect to the over-rolling direction. Therefore, only narrow cracks which are not connected to inclusions would appear in the axial sections as shown in Fig. 3.27, which makes it difficult to distinguish them from other features. Most microcracks showed a positive angle with respect to the over-rolling direction, which is the same with the WEA [22, 37]. However, the observed orientation of the majority of the microcracks was parallel to the surface in contrast with the microcracks observed in [57] or commonly observed butterflies [36], which develop at an angle of ~45°. This suggests that the stress state in the ball-on-rod RCF test may be different from that of other test methods, which



Figure 3.27: Schematics showing the sectional view of microcracks initiated from inclusions.

may be the reason that a DER does not occur in the ball-on-rod RCF tests.

3.3 Repetitive push test

During bearing operation, the inner ring experiences compressive load cycles as the bearing elements roll over it, and the stress state varies depending on the position with respect to a reference point in the subsurface. The strain values cannot be measured directly from normal RCF testing methods, representing a challenge in relating macroscopic material response to microscopic dislocation movement. This section deals with a repetitive push test employing classical mechanical testing equipment, which enables the strain experienced per cycle during the test to be measured.

3.3.1 Experimental

The cyclic stress state during bearing operation is simulated by the repetitive push test with a Mayes 100 kN mechanical tester as in Fig. 3.28. Firstly, a specimen is loaded up to the maximum compressive pressure (p_{max}) during 30 s. Then, sinusoidal compressive



Figure 3.28: The schematic test plan of the repetitive push test.

stress cycles are applied for 5×10^5 times with a frequency of 15 Hz. During this step, a minimum compressive pressure is kept compressive (-0.04 GPa) to prevent the specimen from slipping. Finally, the specimen is released by decreasing the pressure during 1 s. Note that the employed frequency is much lower than that in RCF testing (Tables 2.3 and 3.1); however, 15 Hz was the maximum frequency that can stably apply load in such equipment. p_{max} was varied from 1 to 3.5 GPa in 0.5 GPa steps. According to [58], the maximum resolved shear stress ($\tau_{max,rss}$) is equal to $\frac{1}{M}p_{max}$, where M is Taylor factor which is 2.9 for randomly oriented body centred cubic metals. Since τ'_1 can be reduced down to maximum τ_{xz} (Section 2.4), the corresponding contact pressure in the RCF testings (p_0) is calculated assuming $\tau_{max,rss}$ is 0.25 p_0 (Table 2.2) and the values are listed in Table 3.4.

The sample was a 100Cr6 steel with composition given in Table 3.5. It was heat treated in the form of a $15 \times 15 \times 25$ mm³ specimen. Firstly, it was partially austenitised at 860 °C for 30 minutes in a Ar tube furnace and quenched in oil at 70 °C. After 10 minutes in oil, the sample was rinsed in cold water. Then, it was tempered at 160 °C for 15 minutes. The sample was finally cut into cylinders of 3.43 mm in diameter and

p_{max} / GPa	$\tau_{max,rss}$ / GPa	p_0 / GPa
1.0	0.34	1.38
1.5	0.52	2.07
2.0	0.69	2.76
2.5	0.86	3.45
3.0	1.03	4.14
3.5	1.21	4.82

Table 3.4: Corresponding contact pressure (p_0) for the maximum pressure (p_{max}) in the repetitive push test.

Table 3.5: Composition of the 100Cr6 steel for the repetitive push test (all in wt%). Fe content is balanced accordingly.

С	Cr	Mn	Si	Cu	Ni	Mo	Al
0.97	1.38	0.28	0.28	0.21	0.18	0.06	0.04

5.00–5.21 mm in length. Since only compressive pressure is applied, the specimens do not need the gripping part which would generate stress concentration. Each specimen was polished flat on both top and bottom surfaces and a strain gauge was attached on its side by the CC-33A strain gauge cement (cyano-acrylate base). The smallest Kyowa strain gauge available, KFG-1N-120-C1-11, was adopted and its dimension is shown in Fig. 3.29.

The test setup is shown in Fig. 3.30. The cross section of the lower stage is a 76 mmsided square and that of the upper stage is a 30 mm-diameter circle. Supporters are used in order to prevent denting on the stages during the test. These supporters are the 100Cr6 steel heat-treated the same step as the samples for the test but with a longer tempering time (30 min). They are $\sim 20 \times 20 \times 13$ mm³ blocks and were stuck to the stages before



Figure 3.29: A schematic diagram of the strain gauge used in the study [59].



Figure 3.30: The setup schematic for the repetitive push test.

each test. During loading, only the upper stage moved for loading while the lower stage remained stationary.

During the tests, two data sets were recorded. One set was obtained from the attached strain gauge. The data were recorded directly as microstrain and a typical example is shown in Fig. 3.31a. The other set was recorded from the upper stage position. Since the supporters have similar hardness and larger cross sectional area than the specimen, their denting and strain during the tests was ignored. Thus, the normal strain (ϵ) was obtained from the initial (x_0) and instantaneous position (x_i) of the upper stage and the initial specimen length (l_0) as,

$$\epsilon = \frac{x_0 - x_i}{l_0},\tag{3.13}$$

with one example being plotted in Fig. 3.31b. From these two plots, the normal plastic strain range per cycle $(2\Delta\epsilon_C)$ is obtained by drawing a horizontal line passing through $p_{mean} = \frac{1}{2}(0.04 + p_{max}) \ (p_{mean} \text{ and } p_{max} \text{ in GPa})$ and taking the difference of the intersecting strain values as shown schematically in Fig. 3.32. Then, these were plotted with respect to the number of cycles (N); one example for $p_{max} = 3.0$ GPa is given in Fig. 3.33. The data from the upper stage position $(2\Delta\epsilon_{C,pos})$ seem to show the decay over



Figure 3.31: Examples of the data obtained from (a) the strain gauge and (b) the upper stage position in the repetitive push test for $p_{max} = 1.5$ GPa, N = 10.

logarithmic N in all cases; therefore, the data are fitted employing the curve fitting tool in MATLAB R2011a via

$$2\Delta\epsilon_{C,pos} = b_1 b_2^{\log N} + b_3, \tag{3.14}$$

where b_i (i = 1, 2, 3) are the fitting parameters. Note that $0 < b_2 < 1$ for the decaying trend.

Unfortunately, the strain gauges were not always attached during the testing due to the small specimen dimension and the amount of the data obtained from the gauges was not sufficient to produce reliable fitting results. Hence, a proportionality coefficient of 0.45 was multiplied to Eq. 3.14 in order to scale the data from the strain gauges $(2\Delta\epsilon_{C,gauge})$;

$$2\Delta\epsilon_{C,gauge} = 0.45 \times 2\Delta\epsilon_{C,pos} = 0.45 \left(b_1 b_2^{\log N} + b_3 \right). \tag{3.15}$$

Here, we regard $\Delta \epsilon_{C,gauge}$, as the responsible strain for plastic deformation as explained



Figure 3.32: A schematic diagram showing the acquisition of normal plastic strain range $(\Delta \epsilon_{C,pos}, \Delta \epsilon_{C,gauge})$ and the maximum strain (ϵ_{max}) from the repetitive push test.



Figure 3.33: An example $2\Delta\epsilon_{C,pos}$ and $2\Delta\epsilon_{C,gauge}$ data obtained from the repetitive push test data for $p_{max} = 3.0$ GPa.

in Section 2.4.

The decaying strain stems from the material hardening and after a long time, the strain range approaches an asymptotic value, which corresponds to the plastic strain responsible for plastic deformation (RCF damage) in long run bearings. This strain range, $\Delta \epsilon_C$, can be obtained by taking $N = \infty$ in Eq. 3.15;

$$\Delta \epsilon_C = \left(\Delta \epsilon_{C,gauge}\right)_{N=\infty} = 0.225 \ b_3. \tag{3.16}$$

Finally, $\Delta \epsilon_C$ is converted into the half shear plastic strain range $(\Delta \gamma_C)$ via M,

$$\Delta \gamma_C = M \Delta \epsilon_C. \tag{3.17}$$

Not only the strain range $(\Delta \gamma_C)$ but also the maximum plastic strain $(\epsilon_{p,max})$ have been plotted per each cycle. To acquire $\epsilon_{p,max}$, the total maximum strain (ϵ_{max}) was obtained directly from the strain gauge data as shown in Fig. 3.32. Then, only the plastic contribution was calculated by excluding the elastic strain;

$$\epsilon_{p,max} = \epsilon_{max} - \left(-\frac{p_{mean}}{E}\right),\tag{3.18}$$

where the elastic modulus (E) was taken as 210 GPa. Note that p_{mean} is defined to be positive (Fig. 3.32).

After the tests, specimens were cut in their longitudinal direction, ground, and polished as described in Section 3.2.1.2, 9×4 indents were then made along as shown in Fig. 3.34. Since the final length of the specimen was in the range of 4.95–5.20 mm, the spacing between the indents was ~500 μ m. Considering the minimum recommended spacing between the indents (Section 3.2.1.4) and employing Eq. 3.12, the possible load range is 13–17 kg for 600–850 Vickers hardness. Therefore, the possible maximum load of 2 kg with 15 s-dwell time was adopted by Mitutoyo MVK-H2. Finally, the sample was etched with 2% nital and investigated by OM and SEM.



Figure 3.34: A schematic diagram showing the sample preparation for microstructural analysis and microindentation hardness after the repetitive push tests. l_f is the final length of the specimen.

Table 3.6: Fitting parameters for analysing the repetitive push test data according to Eqs. 3.14 and 3.15.

p_{max}	b_1	b_2	b_3
1.0	3.625×10^{-4}	4.978×10^{-1}	3.225×10^{-4}
1.5	8.244×10^{-4}	1.934×10^{-1}	4.308×10^{-4}
2.0	1.393×10^{-3}	1.961×10^{-1}	5.431×10^{-4}
2.5	1.177×10^{-3}	4.010×10^{-1}	5.258×10^{-4}
3.0	1.114×10^{-3}	$5.063 imes10^{-1}$	$5.315 imes 10^{-4}$
3.5	1.381×10^{-3}	4.283×10^{-1}	6.294×10^{-4}

3.3.2 Results

Two data sets for $2\Delta\epsilon_C$ with respect to the number of test cycles are shown in Fig. 3.35 for all conditions. The data are fitted according to Eqs. 3.14 and 3.15, and with the parameters given in Table 3.6. $2\Delta\epsilon_{C,pos}$ are well fitted, which enables to find reasonable trend lines for $2\Delta\epsilon_{C,gauge}$. The number of $2\Delta\epsilon_{C,gauge}$ data points decreases significantly with p_{max} due to the large deformation of the specimen. With increasing p_{max} , the relatively large difference is observed in the initial $2\Delta\epsilon_C$ values; after ~10³ cycles, $2\Delta\epsilon_C$ approaches an asymptotic value.

The estimated $\Delta \gamma_C$ values from the results are plotted in Fig. 3.36. They are all in the range of approximately 2.0×10^{-4} – 3.5×10^{-4} for $p_{max} = 1.0$ –3.5 GPa, i.e. $p_0 \sim 1$ – 5 GPa. It is interesting that $\Delta \gamma_C$ increases in the lower pressure regime and saturates



Figure 3.35: $2\Delta\epsilon_{C,pos}$ and $2\Delta\epsilon_{C,gauge}$ data obtained from the upper stage position and the strain gauge in the repetitive push tests for different p_{max} . Trend lines are also shown.



Figure 3.36: $\Delta \gamma_C$ with respect to p_{max} . Corresponding p_0 is given in the upper axis according to Table 3.4 assuming $\tau_{rss} = \tau_{xz}$.

when $p_0 \ge 2.5$ GPa. The most important feature is that under the pressure for the full scale endurance tests, i.e. $p_0 = 2.5-4$ GPa (Table 2.3), $\Delta \gamma_C$ can be approximated to be 3.5×10^{-4} .

In Fig. 3.37 where the maximum normal plastic strain ($\epsilon_{p,max}$) is plotted, $\epsilon_{p,max}$ decays towards zero in all cases, suggesting volume expansion due to martensitic transformation of retained austenite. Since the greater decay took place for larger p_{max} , it suggests that the amount of the transformed austenite depends on the applied pressure. The $\epsilon_{p,max}$ reduction rate was also observed to depend on p_{max} , resulting in $\epsilon_{p,max} \sim 0.006$ at $N \sim 4 \times 10^2$ cycle for all conditions except $p_{max}=3.5$ GPa. Insufficient data in the case of $p_{max}=3.5$ GPa is attributed to too high pressure to keep the strain gauge attached for a long time.

The results of microindentation hardness after the tests are shown in Fig. 3.38. The tests with $p_{max} = 1.0$ and 1.5 GPa do not show significant hardness change compared to the untested condition. Nonlinear increase of hardness is observed as p_{max} increases; the



Figure 3.37: Maximum normal plastic strain $(\epsilon_{p,max})$ with respect to N.

hardness change is greater in the higher p_{max} regime, while that for $p_{max} = 2.0$ and 2.5 GPa are similar.

The initial microstructure before the test was tempered martensite with 4.0 ± 0.2 vol.% residual cementite, 2.4 ± 0.2 vol.% transition carbides, and 11.1 ± 0.2 vol.% austenite [15]; the corresponding optical micrographs are shown in Fig. 3.39. The hardness of the matrix was 877 ± 8 HV2. Small microcracks and inclusions are observed. The microcracks are limited within a prior austenite grain and do not extend to the grain next to it. Large inclusions are elongated in the length direction of the specimen.

After testing, microcracks are observed more often (Fig. 3.40a). However, no indication of martensite/residual carbide decay or stress concentration near inclusions was observed (Fig. 3.40b). Under higher pressure ($p_{max} = 3.0$ and 3.5 GPa), it is apparent that elongated inclusions are broken into pieces (Fig. 3.41). However, this needs further study to confirm that it is a real effect, which can be achieved by milling with a FIB to ensure the alleged cracks have a depth.



Figure 3.38: Vickers hardness profiles according to the normalised depth (z) with the final specimen length (l_f) . Two grey horizontal lines represent the hardness range of the untested specimen.

3.3.3 Discussion

The plastic strain ranges obtained by the repetitive push tests and other studies are plotted in Fig. 3.42 for comparison. Since the testing methods are different for all studies, the reported values are converted to $\Delta \gamma_C$. In [60], cyclic shear stress-shear strain hysteresis loops has been calculated assuming elastic linear kinematic plasticity (ELKP) with the parameters shown in Fig. 3.43a; $\Delta \gamma_C$ has been obtained from the stabilised loop for $p_0 = 3.5$ GPa. In [61, 62], the cyclic stress-strain hysteresis loop itself has been assumed with parameters shown in Fig. 3.43b, enabling the direct calculation of $\Delta \epsilon_C$ which can be converted into $\Delta \gamma_C = \sqrt{3}\Delta \epsilon_C$. Hahn *et al.* [40] obtained $\Delta \epsilon_C$ via cyclic torsion testing with constant stress amplitude; this data has also been converted into $\Delta \gamma_C = \sqrt{3}\Delta \epsilon_C$. Note that this conversion is different from Eq. 3.17 due to the consistency with the von Mises assumption made in the original literature. In the case of data acquired from symmetric push-pull fatigue testing in [63], the testing stress amplitude was assumed to be equivalent to p_{max} in the repetitive push test and the corresponding p_0 and $\Delta \gamma_C$ were



Figure 3.39: Optical micrographs of (a) microcracks and (b) the microstructure adjacent to an inclusion in the specimen before testing.



Figure 3.40: Optical micrographs of (a) microcracks in the specimen tested with $p_{max} = 1.0$ GPa and (b) the microstructure adjacent to an inclusion in the specimen tested with $p_{max} = 1.5$ GPa.



Figure 3.41: Cracks (pointed with arrows) within an inclusion observed in the specimen tested with $p_{max} = 3.5$ GPa.

calculated as explained in Section 3.3.1.

In Fig. 3.42a, all $\Delta \gamma_C$ resulted in the order of 10^{-4} . The symmetric push-pull fatigue tests by Christ *et al.* [63] resulted in low $\Delta \gamma_C$; this may be due to the testing involving tensile loading, which needs further study. The data by Bhargava *et al.* [61, 62] shows relatively large values $\sim 10^{-2}$ (Fig. 3.42b, which suggests that $\Delta \gamma_C$ may depend significantly on the test/calculation methods. Here, it is concluded that $\Delta \gamma_C$ in bearing operation would be in the range of 10^{-4} – 10^{-3} which is supported by the most researchers [40, 60].

The difference in hardness before and after the test (Δ HV) has been plotted in 3.44 for both repetitive push tests and ball-on-rod RCF tests. The data for the repetitive push tests were averaged for $0.2 \leq z/l_f \leq 0.8$ in Fig. 3.38 due to the low hardness close to the surface. Δ HV seems very similar in both cases. As mentioned in Section 3.2.3, Δ HV is likely to be originated from the residual stress development during testing. Then, the low hardness near the surface may be due to the relaxation of residual stress due to the material surface. Moreover, it suggests that the degree of the residual stress developed during the tests is very similar in both tests. Since residual stress originates from the material response to stress, such as dislocation generation and rearrangement as well as stress induced martensite formation, it suggests that the material response may be similar in both cases.



Figure 3.42: Comparison of $\Delta \gamma_C$ values obtained in the repetitive push test with those by Warhadpande *et al.* [60], Hahn *et al.* [40], Christ *et al.* [63], and Bhargava *et al.* [61, 62]. Note that $\Delta \gamma_C$ -scale is different in (a) and (b).



Figure 3.43: Material parameters used for the elastic linear kinematic plasticity assumption by (a) Warhadpande *et al.* [60], and (b) Bhargava *et al.* [61, 62].



Figure 3.44: Hardness increase experienced in the repetitive push testing after $N = 5 \times 10^5$ cycles, and the ball-on-rod RCF testing after $N = 10^8$ cycles.

3.4 Summary

Ball-on-rod RCF tests were carried out with $p_0 = 3.7$ -5.6 GPa, and $N = 10^7$, 10^8 cycles to study microstructural change. The formation of DER was not observed; however, a number of microcracks initiated from inclusions, which were identified for all test conditions. 10–20% of the cracks were observed to be decorated by WEAs, which suggests that microcracks may be the precursor of WEAs. The microcracks are observed only in the circumferential section at 0–600 μ m under the surface with most of them within -10° to 20° from the surface. The cracks were formed adjacent to inclusions with different types and sizes; the depth corresponding to the largest number of inclusions bearing microcracks matched with the τ_1 variation with depth. Larger cracks (> 100 μ m) were seen at a depth of 100–200 μ m when tested with $p_0 \ge 4.7$ GPa. They lie parallel to the surface, penetrating prior austenite grain boundaries. Hardness increase was detected for all conditions. The hardness reaches a maximum at 100 μ m under the surface and decreases to the undeformed matrix value for depths beyond 350 μ m. The extent of the increase depends on p_0 , but not on N, implying that strengthening is attained at early rolling stages.

Repetitive push tests were carried out in order to acquire the strain range $(\Delta \gamma_C)$ responsible for the plastic deformation during bearing operation. The maximum testing compressive pressure (p_{max}) was 1.0–3.5 GPa, which corresponds to $p_0 = 1.38-4.82$ GPa. $\Delta \gamma_C$ increases from $p_0 \approx 1.5$ to 2.5 GPa and saturates when $\Delta \gamma_C \sim 3.5 \times 10^{-4}$. Another interesting result is that the plastic strain decays to zero which suggests the transformation of retained austenite into martensite under stress. Although microstructural changes of the matrix could not be detected with OM and SEM, the breaking of inclusions was observed at $p_{max} = 3.0$ and 3.5 GPa, i.e. $p_0 = 4.1$ and 4.8 GPa.

The hardness increase during the test was similar to the ball-on-rod RCF test. This suggests that the microstructural changes induced by both tests are equivalent.

Chapter 4

Coarsening of carbide particles

One of the forms of microstructural damage during rolling contact fatigue (RCF) is darketching regions (DERs), which occurrence has been outlined in Section 2.3.1. It has also been pointed out that their formation is attributed to tempering, during which supersaturated C in the matrix migrates to existing carbide particles and assists their coarsening. As a result, the etching behaviour and hardness gradually change. The tempering is suggested to be a consequence of the plastic flow during RCF rather than heating [29].

In this chapter, tempering assisted by deformation is suggested to result from dislocation glide. Since a C atmosphere can form around a dislocation strain field, dislocations may glide along with their atmospheres and carry C atoms to the carbide particles. As this process continues, the matrix becomes C-depleted and the carbide coarsens, resulting in hardness reduction. This concept will be explained with derived mathematical expressions. The new formulation is applied to some run bearings, where the hardness evolution is successfully predicted.

4.1 Model for dislocation assisted tempering

In this section, the concept of C being transported via moving dislocations is introduced to show that dislocation assisted diffusion is possible. As a result, the hardness evolution during rolling contact is predicted, and the effects of parameters related to the initial microstructure and operating conditions are examined.

For microstructure evolution computations, the following assumptions are made;

- (i) Residual cementite effects are ignored. However, the matrix composition is determined by considering 4 vol% of residual cementite, as shown in the previous work [15]. MatCalc version 5.50 (rel 1.007) with mc_fe v.2.0 thermodynamic database are employed to calculate the fractions and compositions in the initial microstructure.
- (ii) DER formation does not form along with other damage such as WEAs or WEBs.
- (iii) Dislocation-C interaction energy is independent of alloy composition and dislocation density.
- (iv) The amount of C in solid solution is the main factor which determines hardness [64].The effect of precipitate growth and dissolution to hardening/softening is negligible.
- (v) Dislocations are generated up to a density of 10^{16} m⁻², and the steady-state is reached at an early operation stage.
- (vi) Carbide coarsening is a diffusion controlled phenomenon assisted by dislocations.
- (vii) The effect of retained austenite in dislocation assisted tempering and hardness evolution is neglected.

In addition, it is worth emphasising that the size as well as the volume fraction of carbide increase during dislocation assisted tempering. Hence, the carbide coarsening in this study is different from the classical coarsening [65] where constant carbide volume fraction is maintained by larger carbide particle growing at the expense of smaller one.

4.1.1 Carbon transport

Consider a stationary dislocation in a matrix supersaturated with C. When the dislocation strain field attracts C atoms, the corresponding interaction energy (U) can be expressed as [66, 67]:



Figure 4.1: Interaction energy (U) between dislocation and C atoms with respect to the distance (r_d) between them. U_{θ} and U_{ϵ} are the binding energies of a C atom in the cementite and ϵ -carbide, respectively, and $r_{d\theta}$ and $r_{d\epsilon}$ are obtained with Eq. 4.1.

$$U = \frac{A}{r_d/b},\tag{4.1}$$

where r_d is the distance between the dislocation core and a C atom, b is the magnitude of Burgers vector, and A is a constant equal to 0.5 or 0.46 eV [66, 68]; here, we take A = 0.46eV following Kalish and Cohen [68]. The variation of U with r_d is plotted in Fig. 4.1. However, not all C is trapped within dislocation atmospheres, as C atoms may be more stable within carbides, or their diffusivity may not keep up with dislocation glide.

The tendency of cementite to incorporate C atoms can be assessed from its binding energy with C (U_{θ}) . If $r_{d\theta}$ is defined to be the C-dislocation distance when $U = U_{\theta}$, it becomes the distance which limits the stability of C atoms within a dislocation strain field against cementite. C atoms with $r_d > r_{d\theta}$ would lower its energy within cementite. Therefore, as a dislocation with a C atmosphere meets a cementite particle, C atoms located at $r_d > r_{d\theta}$ can leave the dislocation and be incorporated into the cementite. This explanation applies similarly to the other carbide species. With $U_{\theta} = 0.523-0.542$ eV [69–71] and the binding energy of C in ϵ , $U_{\epsilon} = 0.27$ eV [72], $r_{d\theta}$ and $r_{d\epsilon}$ are 0.85b–0.88b and 1.7b, respectively (Eq. 4.1). Therefore, more C atoms would be trapped by cementite rather than by ϵ in its proximity.

C atoms in the Cottrell atmosphere can be dragged by gliding dislocations only if they have sufficient mobility. This can be quantified by comparing the thermal velocity of the C atom with the gliding velocity of the dislocation. Following Cottrell [66], the steady drift velocity $(\vec{v_C})$ of a C atom moving by thermal activation under the force \vec{F} is given as

$$\vec{v_C} = \frac{D}{k_b T} \vec{F},\tag{4.2}$$

where D is the diffusion coefficient of C, k_b is the Boltzmann constant, and T is temperature. Since \vec{F} comes from dislocation-C interaction,

$$F = -\left(\frac{\partial U}{\partial r_d}\right) = \frac{A_b}{\left(\frac{r_d}{b}\right)^2},\tag{4.3}$$

$$\therefore v_C = \frac{D}{k_b T} \cdot \frac{A/_b}{\left(\frac{r_d}{_b}\right)^2}.$$
(4.4)

Only C atoms with v_C larger than or equal to the dislocation velocity (v_{disl}) can keep up with the dislocation during plastic deformation. Therefore, the limiting distance (r_{disl}) of C transported by a dislocation can be obtained by substituting v_C by v_{disl} in Eq. 4.4;

$${}^{r_{disl}}/{}_{b} = \sqrt{\frac{D\left({}^{A}/{}_{b}\right)}{k_{b}Tv_{disl}}}.$$

$$(4.5)$$

 v_{disl} can be expressed with strain rate $(\dot{\gamma})$ via Orowan equation: $\dot{\gamma} = \rho b v_{disl}$, where ρ is the dislocation density. For a bearing rotating at \dot{R} revolutions per minute (rpm) and N_C stress cycles per revolution,

$$\dot{\gamma} = \frac{\dot{R}}{60} \cdot N_C \cdot \Delta \gamma_C, \qquad (4.6)$$



Figure 4.2: The change in r_{disl} according to the operating temperature for $\Delta \gamma_C = 3.5 \times 10^{-4}$.

where $\Delta \gamma_C$ is the shear strain range per cycle, which is explained in Section 2.4 and obtained in Section 3.3. Hence,

$$v_{disl} = \frac{\dot{R}N_C \Delta \gamma_C}{60\rho b}.$$
(4.7)

$$\therefore \ ^{r_{disl}}/_{b} = \sqrt{\frac{60DA\rho}{\dot{R}N_{C}\Delta\gamma_{C}k_{b}T}}.$$
(4.8)

For $\dot{R} = 6000$ rpm and $N_C = 4.69$ [24], which correspond to the experimental conditions of Swahn *et al.* [5], r_{disl} variation with temperature is plotted in Fig. 4.2. Here, we take A = 0.46 eV, $\rho = 10^{16}$ m⁻², and $D = 6.2 \times 10^{-7} \exp\left(-\frac{80000}{RT}\right)$ m²s⁻¹ [9] where R is the ideal gas constant in J mol⁻¹ K⁻¹ and T is in K. Also, $\Delta \gamma_C$ is taken to be 3.5×10^{-4} as obtained with the repetitive push test in Section 3.3 (Fig. 3.36).

To summarise, the C atoms have to meet two conditions simultaneously in order to contribute to carbide coarsening; (i) $v_C > v_{disl}$, and (ii) $U < U_{\theta}$, which is only possible for those located at $r_{d\theta} < r_d < r_{disl}$ as shown in Fig. 4.3. From this condition, the



Figure 4.3: A schematic of carbon atmosphere around a dislocation. Two important limiting distances are shown to present the condition for carbon location in order to contribute to the carbide coarsening.

C migrating from dislocations to cementite can be quantified. In [68], the number of octahedral interstitial sites are given for [111] screw and [112] edge dislocations in a body centred cubic structure with respect to r_d . According to this approach, the number of octahedral interstitial sites (n_{site}) between $r_{d\theta}$ and r_{disl} is acquired as a function of r_{disl} for cementite and ϵ -carbide and shown in Fig. 4.4. Note that n_{site} is the number for one atomic plane which is normal to the corresponding dislocation.

However, these sites cannot be filled completely by C atoms, because each rolling contact cycle takes place so rapidly that C atoms do not have enough time to be segregated fully at those sites. Therefore, the possibility of the sites to be filled would be proportional to the concentration of C in the matrix. Accordingly, the number of C (n_C) which can be transported by the unit length of a dislocation can be calculated via



Figure 4.4: Number of octahedral interstitial sites per atomic plane which is normal to [111] screw and [112] edge dislocations which can be filled with carbon atoms contributing to coarsening of (a) cementite and (b) ϵ -carbide.

$$n_C = \frac{2}{6} \cdot C_{\alpha'0} \cdot n_{site} \cdot \frac{1}{s},\tag{4.9}$$

where $C_{\alpha'0}$ is the initial C mol fraction in the matrix and s is the spacing of the atomic plane normal to the dislocation. $\frac{2}{6}$ is multiplied to consider the number ratio between the lattice sites and the octahedral interstitial sites. Following Eqs. 4.8 and 4.9, n_C can be calculated for the relevant temperature and strain conditions, and it is shown in Fig. 4.5. In this case, an equal dislocation density is assumed for the screw and edge dislocations. At higher temperatures, more C atoms can be carried by dislocations due to enhanced thermal drift. Moreover, C transport is encouraged under small $\Delta\gamma_C$, i.e. less deformation, because of the slow dislocation movement resulting in larger r_{disl} (Eq. 4.8). The change is not gradual according to $\Delta\gamma_C$ due to the step-wise dependence of n_{site} on r_{disl} (Fig. 4.4). For example, for the cementite case at 100 °C, $\Delta\gamma_C = 3 \times 10^{-4}$ and 3.5×10^{-4} show the same n_C , because their r_{disl} are 1.933b and 1.790b, respectively, which coincidentally lie on the plateau for n_{site} in Fig. 4.4a. The effect of the carbide type is interesting; as expected from $U_{\theta} > U_{\epsilon}$, more C can contribute to tempering of cementite than in ϵ . In the case of ϵ , tempering cannot occur when $\Delta\gamma_C \geq 3.0 \times 10^{-4}$ or $T \leq 90$ °C.

4.1.2 Dislocation assisted diffusion

With the calculated amount of C that can be carried by dislocations, the C flux into a carbide particle can be estimated as

$$J_{disl} = \rho v_{disl} n_C. \tag{4.10}$$

Now, consider a two dimensional system where plate-like precipitate particles lie in the supersaturated matrix as shown in Fig. 4.6. When a dislocation with the C atmosphere meets a carbide particle, the C concentration profile would correspond to Fig. 4.7. According to Fick's first law,



Figure 4.5: The change in n_C according to $\Delta \gamma_C$ for different temperatures and carbide types when the initial alloy C concentration is 3.63 at%.

$$J_{disl} = -D^{disl} \frac{\partial C_V}{\partial r}.$$
(4.11)

$$\therefore \rho v_{disl} n_C = D^{disl} \frac{C_{V\alpha'0} - C_{V\alpha,eq}}{\Delta x_{disl}}, \qquad (4.12)$$

assuming that the C gradient adjacent to the dislocation is linear. Here, D^{disl} is dislocation assisted diffusivity which is defined as the diffusivity of C transported via dislocation glide. Δx_{disl} is a diffusion distance, and $C_{V\theta}$, $C_{V\alpha,eq}$, and $C_{V\alpha'0}$ are C concentration per unit volume in cementite, equilibrium ferrite, and the initial matrix, respectively. After rearrangement of the previous equation,

$$D^{disl} = \frac{\rho v_{disl} n_C \Delta x_{disl}}{C_{V\alpha'0} - C_{V\alpha,eq}}.$$
(4.13)

The necessary parameters to calculate D^{disl} are given in Table 4.1. To assess the effect of a specific parameter, this was varied while keeping the rest fixed. Adopting these parameters, D^{disl} for the same conditions in Fig. 4.5 are calculated and plotted in Fig. 4.8



Figure 4.6: A schematic diagram showing the system of interest. Plate morphology of carbide with the width of $2r_{\theta}$ and spacing of l_p in the matrix has been assumed.

in respect with $\Delta\gamma_C$. During the calculation, Δx_{disl} is regarded as the distance between the transported C and the dislocation and is approximated as b. The temperature sensitivity is remarkable; increasing from 90 °C to 100 °C results in even eight times greater D^{disl} for cementite when $\Delta\gamma_C = 3.5 \times 10^{-4}$. An interesting feature is the peaks in the plot for cementite at T = 100 °C, $\Delta\gamma_C = 3.5 \times 10^{-4}$ and at T = 90 °C, $\Delta\gamma_C = 2.5 \times 10^{-4}$, 3.5×10^{-4} . This suggests two competing mechanisms: D^{disl} is proportional to both v_{disl} and n_C (Eq. 4.13); while higher $\Delta\gamma_C$ increases v_{disl} (Eq. 4.7), it reduces n_C . It is because higher $\Delta\gamma_C$ causes lower r_{disl} (Eq. 4.8) and results in smaller n_{sites} (Fig. 4.4) which is proportional to n_C (Eq. 4.9). Comparing Figs. 4.5 and 4.8, D^{disl} follows n_C at low $\Delta\gamma_C$ regime, but deviates at high $\Delta\gamma_C$ regime as the effect of v_{disl} proportional to $\Delta\gamma_C$ becomes more obvious (Eq. 4.7).



Figure 4.7: A schematic profile of C concentration per unit volume (C_V) according to the distance from a carbide particle (r) when a dislocation carrying C meets a carbide particle with an initial radius of r_0 .
Parameter	Nomenclature	Value or expression
Operating speed	Ŕ	6000 rpm
Number of cycles per revolution	N_C	4.96
Plastic strain range per cycle	$\Delta\gamma_C$	$3.5 imes 10^{-4}$
Dislocation density	d	$10^{16}~{ m m}^{-2}$
Initial radius of the particle	r_0	10 nm
Initial volume fraction of the precipitate	V_{f0}	0.017
Lattice parameter of the martensitic matrix	$a_{lpha'}$	0.2876 nm
Lattice parameter of the cementite	$a_{ heta}$	$0.45156 \ \mathrm{nm}$
	$b_{ heta}$	$0.50837 \mathrm{~nm}$
	$c_{ heta}$	$0.67297 \mathrm{~nm}$
Lattice parameter of the ϵ -carbide	a_ϵ	0.4767 nm
	b_ϵ	0.4353 nm [73]
C concentration in the alloy	C_0	3.63 at %
Equilibrium C concentration in the matrix per unit volume	$C_{V^{lpha,eq}}$	$3.66 \times 10^{20} {\rm m}^{-3}$
Atomic weight of Fe	w_{Fe}	55.85
Atomic weight of C	m_C	12.011
Magnitude of Burgers vector	p	$rac{2}{\sqrt{3}}a_{lpha'}$
Spacing between the particles	l_p	$\frac{2r_0}{V_{f0}}$
C concentration in the alloy per unit volume	C_{V0}	$\frac{C_0}{a^3}$, /2
C concentration in cementite per unit volume	$C_{V heta}$	$\frac{\alpha'}{4}$
C concentration in $\epsilon\mathrm{carbide}$ per unit volume	$C_{V\epsilon}$	$\frac{2\sqrt{3}}{2}$
C concentration in the initial matrix per unit volume	$C_{Va'0}$	$\frac{\frac{1}{2}l_{P}C_{V0}-r_{0}C_{V\theta}}{\frac{1}{2}l_{P}-r_{0}}$

Table 4.1: Basic input parameters and calculated parameters for the carbide coarsening model. $C_{V\alpha,eq}$ is obtained from MatCalc version 5.50 (rel 1.007) with mc_fe v.2.0 thermodynamic database.



Figure 4.8: Calculated D^{disl} with respect to $\Delta \gamma_C$ for different temperatures and carbide types.

4.1.3 Hardness evolution

Carbide coarsening kinetics can now be calculated with D^{disl} . The C concentration profile adjacent to a cementite particle is schematically shown in Fig. 4.9. After the initial state which is represented in a solid line, the profile will follow the dotted line in Fig. 4.9. Relating the C flux (J) toward the cementite with the growth rate (v_{θ}) while assuming a linear gradient at the interface,

$$J = (C_{V\theta} - C_{V\alpha,eq}) \frac{dr_{\theta}}{dt} = -D^{disl} \frac{\partial C_V}{\partial r} = D^{disl} \frac{C_{V\alpha'} - C_{V\alpha,eq}}{\Delta x_{disl}},$$

$$\therefore v_{\theta} = \frac{dr_{\theta}}{dt} = D^{disl} \frac{C_{V\alpha'} - C_{V\alpha,eq}}{\Delta x_{disl} (C_{V\theta} - C_{V\alpha,eq})},$$
(4.14)

where r_{θ} is the cementite radius, t is time, and $C_{V\alpha'}$ is the C concentration in the matrix per unit volume. Considering mass conservation,



Figure 4.9: A schematic profile of the C concentration near to a cementite particle with the initial radius of r_0 . The solid line represents the initial state, while dotted ones show the profile when the cementite radius reaches $r_0 + \Delta r_{\theta}$.

$$(C_{V\theta} - C_{V\alpha'}) \Delta r_{\theta} = \frac{1}{2} \Delta x_{disl} (C_{V\alpha'} - C_{V\alpha,eq}),$$

$$\therefore v_{\theta} = D^{disl} \frac{(C_{V\alpha'} - C_{V\alpha,eq})^{2}}{2\Delta r_{\theta} (C_{V\theta} - C_{V\alpha'}) (C_{V\theta} - C_{V\alpha,eq})}.$$
(4.15)

Note that Δr_{θ} is in the denominator other than r_{θ} as in [65]. With v_{θ} and the time increment, Δt , Δr_{θ} is updated providing a new value of r_{θ} and $C_{V\alpha'}$. Vickers hardness is acquired from the relationship [64, 74]:

(Vickers hardness) =
$$1667C_{\alpha'} - 926(C_{\alpha'})^2 + 150,$$
 (4.16)

where $C_{\alpha'}$ is C concentration in the matrix in wt%. The parameters given in Table 4.1 are adopted for the calculation. The initial value of Δx_{disl} is assumed to be b.

The effect of $\Delta \gamma_C$ and T can be readily understood with the results shown in Fig. 4.10. Since the reaction is diffusion controlled, D^{disl} dictates the kinetics. At 90 °C, hardness plots for $\Delta \gamma_C = 2.0 \times 10^{-4}$, 2.5×10^{-4} , and 3.0×10^{-4} overlap, which is caused by similar D^{disl} (Fig. 4.8). This also applies to two overlapping cases at 100 °C: for $\Delta \gamma_C = 2.5 \times 10^{-4}$ and 3.5×10^{-4} , and for $\Delta \gamma_C = 3.0 \times 10^{-4}$ and 4.0×10^{-4} . Therefore, dislocation assisted tempering kinetics does not follow a monotonic relationship with $\Delta \gamma_C$, while increasing T always results in faster kinetics. In other words, DER formation kinetics can be assessed by D^{disl} which depends on the bearing operating conditions and the degree of deformation. Another example is the influence of different carbide types; since ϵ -carbide has low D^{disl} at 100 °C (Fig. 4.8), tempering is slower than for cementite (Fig. 4.10b).

With the same D^{disl} , the initial microstructure strongly influences the coarsening kinetics. Effects of the initial precipitate radius (r_0) and volume fraction (V_{f0}) are shown in Fig. 4.11a and b, respectively. It is clear that the tempering becomes slower with increasing r_0 . However, the kinetics is less sensitive to r_0 than T; coarsening can be more effectively retarded by lowering T by 10 °C than increasing r_0 by a factor of two. When V_{f0} is increased with constant r_0 , C is depleted in the matrix resulting in a lower initial hardness (Fig. 4.11b). Since hardness reduction rate is not so sensitive to V_{f0} , its effect on initial hardness is rather significant; alloys with higher V_{f0} always show lower hardness at any N.

The evolution of the particle radius (r_{θ}) and the volume fraction (V_f) of cementite for the basic conditions (Table 4.1) are plotted in Fig. 4.12a and b, respectively. Both show sigmoidal evolutions.

4.1.4 Application to experimental results

By changing some input parameters, the model can be applied to predict the hardness reduction associated to DER formation during bearing operation. Two experimental data sets are compared with the model results, and another data set is introduced to show the special case when the model is not applicable.



Figure 4.10: Calculated hardness evolution during dislocation assisted tempering at (a) 90 °C and (b) 100 °C. All data are calculated in the case of cementite unless indicated.



Figure 4.11: Calculated hardness evolution during dislocation assisted tempering of cementite for different initial (a) radius (r_0) and (b) volume fraction.

At 100°C



Figure 4.12: Calculation results during dislocation assisted tempering of cementite for the (a) particle radius (r_{θ}) , and (b) volume fraction of the cementite at 100 °C.

Table 4.2: Heat treatment of samples for the full endurance bearing tes

		1	0
	Austenitisation	Oil quench	Tempering
Swahn1 $[5]$	860 °C, 20 min	no information	160 °C
SKF1	860 °C, 20 min	$60 ^{\circ}\mathrm{C}$ followed by cold water rinse	160 °C, 90 min
SKF2	860 °C, 20 min	$60~^\circ\mathrm{C}$ followed by cold water rinse	220 °C, 4 h

4.1.4.1 Experimental

Three data sets introduced here are (i) Swahn1 is from the literature [5], (ii) SKF1 and (iii) SKF2 from the samples supplied by SKF. All were from 6309 deep groove ball bearings operated in full endurance bearing tests. The composition of all samples was 100Cr6 (Table 2.1) with the heat treatment procedure shown in Table 4.2 to produce martensitic structures. The test conditions are given in Table 4.3.

 \dot{R} and N_C are the same as in Table 4.1. $\Delta \gamma_C = 3.5 \times 10^{-4}$ under $p_0 = 3.4$ GPa is deemed reasonable according to the repetitive push test results (Fig. 3.36). Note that dislocation assisted tempering cannot occur for ϵ -carbide at such $\Delta \gamma_C$ (Fig. 4.8). For the

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	p_0 / GPa	$T / ^{\circ}C$	\dot{R} / RPM	N / cycles
Swahn1 [5]	3.3 or 3.7	50-55 (lubricant)	6000	$10^6, 10^7, 10^8, 10^9$
SKF1	3.2	$83 \ (outer \ ring)$	6000	$1.29 \times 10^5, 1.29 \times 10^6,$
				$1.29 \times 10^7, 1.29 \times 10^8$
SKF2	3.3	25 (lubricant),	6000	$5.96 \times 10^8, 1.49 \times 10^9,$
		73 (outer ring)		1.98×10^9

Table 4.3: Conditions of the full endurance bearing tests. The temperature of the lubricant was measured on inlet.

Table 4.4: Precipitate sizes [15] and estimated equivalent radii assuming cylindrical morphology after tempering for 0.25–4 h at 160 °C.

I IIIIC / II DIMINGUOL / IIIII DOILGUI / IIIII DOULVAI	ent radius / nm
0.25 2 2	1.1
1 5 10	3.6
2 10 70	10.9
4 10 72	11.1

carbide particle size, information for a similar heat treatment reported in the literature [15] is adopted; the equivalent radii have been estimated assuming cylindrical carbide particle (Table 4.4). Since SKF1 was tempered for 90 min, r_0 is given as 7 nm which was also used for Swahn1 due to no information was reported on the tempering time. Because SKF2 is tempered at a higher temperature, r_0 is taken to be 15 nm. Since the initial hardness depends on V_{f0} via $C_{\alpha'0}$ (Eq. 4.16), V_{f0} was set to match the calculated hardness with the measured one; these values are given in Table 4.5.

Hardness data of Swahn1 were acquired directly from [5]. The data of SKF1 and SKF2 were obtained from measured hardness profiles. For the measurements, microindentation hardness tests were done on axial sections of the inner rings (Fig. 2.2b). The samples were prepared in the same way as in Section 3.2.1.2 without etching. Due to the grooves

Table 4.5: Initial volume fractions of carbide estimated from the initial Vickers hardness.

	Swahn1	SKF1	SKF2
Initial Vickers hardness	$870 \pm 20 \ [5]$	813 ± 10	716 ± 9
V_{f0}	0.0135	0.0335	0.0545



Figure 4.13: Microindentations made for measuring hardness for RCF-tested bearings.

of the inner ring, the contact area between balls and the inner ring are larger than the one shown in Fig. 3.15a. Therefore, 4×5 indentations with 200 μ m spacing were made as shown in Fig. 4.13. Due to the limitation in indentation spacing (> 2.5d) [52], d should be smaller than 80 μ m. Therefore, L = 1 kg was chosen to meet the requirement (Fig. 3.17). Three measurement sets were made for each test condition. If the standard deviation was larger than 15, the average was acquired only from the two similar values. This higher standard deviation may be originated from inhomogeneous microstructure containing white-etching bands (WEBs). From the profile, hardness at the depth showing the greatest change was obtained and used for comparison with predicted values. For the hardness of the unaffected area, the region at the bottom left of the sections was mesured (Fig. 4.13). Five indentations were made for each section: i.e. fifteen indentations for each test condition.

The same methods and equipments as in Section 3.2.1.2 were used for microscopy. Axial sections were etched after the hardness measurement for OM and SEM. Also, circumferential sections were observed. For TEM, the samples were prepared from the tangential section. Grinding the specimen was done more carefully to make sure that the thin film lies within the DER; a plate with the tangential section was cut and 150 μ m was ground off from the surface. Then, it was ground again from the other side resulting in a 80–90 μ m-thick film.

4.1.4.2 Results

Hardness profiles are presented in Fig. 4.14 for SKF1 and SKF2. SKF1 does not show large hardness change due to the relatively small N (< 10⁹); however, the hardness evolution trend is obvious. The hardness increased between $N = 1.29 \times 10^5$ and 1.29×10^6 cycles and then decreased. On the other hand, all samples showed higher hardness than that of the unaffected area in SKF2. The hardness was similar for all cases observed, which may be due to a small difference in N. Hardness at depths of 250 μ m and 150 μ m were taken for SKF1 and SKF2, respectively for further comparison with the calculation results.

In Fig. 4.15, the calculated hardness evolution is shown with experimental results. The experimental results of Swahn1 can be best predicted with the dislocation assisted tempering at 100 °C. From Table 4.3, it is seen that the temperature difference between the lubricant inlet and the outer ring is ~ 50 °C. Since the temperature of the inner ring during the tests would be similar to that of the outer ring, operating temperature of Swahn1 may have been reached ~ 100 °C, which is consistent with the results. Despite the limited number of data points, SKF1 seems to be well predicted with lower temperature which matches the operating temperature (83 °C) as in Table 4.3. However, the model cannot predict the experimental data of SKF2.

The microstructural changes in SKF1 are shown in Fig. 4.16. DER formed at a depth of 100–350 μ m under the surface between $N = 1.29 \times 10^6$ and 1.29×10^7 cycles, which corresponds to the period at which hardness reduction is first observed after its increase (Fig 4.14a). The boundary between DER and unaffected region near to the surface is



Figure 4.14: Hardness profiles of (a) SKF1 and (b) SKF2. Two grey horizontal lines in each plot represent the hardness range of the unaffected region.



Figure 4.15: Calculated hardness change with the experimental data of (a) Swahn1 [5], (b) SKF1, and (c) SKF2.

diffuse. Secondary electron images in Fig. 4.17a show that the DER is composed of light contrast patches. While these patches were scattered at the boundary, their frequency increased as the centre of the DER is reached. The light contrast comes from a rougher surface in the patches. The rougher surface after etching may be due to the deformation of the martensitic matrix or coarsening of temper carbides. TEM bright field images of the tangential sections in the unaffected area and the DER are compared in Fig. 4.18. Some martensitic plate boundaries became ambiguous by the formation of a polygonal cell structure within the plate as well as a entangled dislocation structure. A selected area diffraction pattern (Fig. 4.19c) is produced from the 500 nm region encircled in Fig. 4.19a showing the cell orientations are not completely random. The cell size can be obtained from the dark field image formed with small portion of the arc in Fig. 4.19c; it is in the range of tens of nanometres (Fig. 4.19b). Since the temper carbides were very small (assumed as 7 nm for the calculation), it was difficult to identify the changes in carbides from the deformed structures with small cell structures. Moreover, the model results for $N = 1.29 \times 10^8$ cycles, $r_{\theta} \sim 10$ nm and $V_f \sim 0.05$ show a small change in radius considering the initial state $(r_0 = 7 \text{ nm and } V_{f0} = 0.0335)$ (Fig. 4.20).

The discrepancy between the calculation and experimental results for SKF2 can be explained by its microstructure (Fig. 4.21). No DER was observed in SKF2; instead, 30° WEBs appeared. These bands were clearly recognised as shown in Fig. 4.22; they were parallel to the surface in the axial section and inclined $\sim 20-30^{\circ}$ in the circumferential section, which is consistent with previous studies on WEBs (Fig. 2.12, [5, 21, 23, 25, 26, 28, 29]). When these bands were observed closely employing SEM, they represented two features: (i) smoothly etched thin areas enveloping dented plates (marked with arrows in Fig. 4.23a), and (ii) dented relatively thick areas (marked with dark arrows in Fig. 4.23b) within which very fine particles (marked with yellow arrows in Fig. 4.23b) are observed. Based on the observation and the literature [5, 25, 32], it is conjectured that the first feature corresponds to lenticular carbides, while the second represents cellular structures. It is interesting that the region containing the WEBs show higher hardness



Figure 4.16: Optical micrographs of the axial section of SKF1 operated for (a) $N = 1.29 \times 10^5$ cycles, (b) $N = 1.29 \times 10^6$ cycles, (c) $N = 1.29 \times 10^7$ cycles, and (d) $N = 1.29 \times 10^8$ cycles.



Figure 4.17: Secondary electron micrographs of the axial section of SKF1 operated for $N = 1.29 \times 10^8$ cycles showing the areas (a) near to the surface, (b) far from the surface (unaffected), and (c) within the DER.



Figure 4.18: Transmission electron bright field images of SKF1 operated for $N = 1.29 \times 10^8$ cycles showing the areas (a) far from the surface (unaffected), and (b) within the DER.



Figure 4.19: Transmission electron micrographs of SKF1 operated for $N = 1.29 \times 10^8$ cycles. (a) A bright field image marked with the location of the selected aperture for the diffraction pattern in (c). (b) A dark field image obtained with one of $(110)_{\alpha}$ reflections marked in the diffraction pattern. (c) The diffraction typical of the beam direction parallel to $[111]_{\alpha}$.



Figure 4.20: Calculation results for the (a) particle radius (r_{θ}) , and (b) volume fraction (V_f) of the cementite of SKF1.

as in white-etching areas, which may indicate carbide dissolution rather than coarsening.

4.2 Discussion

4.2.1 Controlling the kinetics of dark-etching region formation

Based on the study in Section 4.1.3, two ways to delay DER formation would be reducing D^{disl} or changing the microstructure.

The factors affecting D^{disl} can be examined by rearranging Eq. 4.13 using Eqs. 4.7 and 4.9;

$$D^{disl} = \frac{\rho \frac{RN_C \Delta \gamma_C}{60\rho b} \frac{2}{6} C_{\alpha'0} \frac{n_{site}}{s}}{C_{V\alpha'0} - C_{V\alpha,eq}} = \frac{\dot{R}N_C}{180bs} \cdot \frac{C_{\alpha'0}}{C_{V\alpha'0} - C_{V\alpha,eq}} \cdot \Delta \gamma_C \cdot n_{site}$$
(4.17)

Expressing the diffusion coefficient in terms of $m^2 \text{ cycle}^{-1}$ rather than $m^2 \text{ s}^{-1}$, the equation is rearranged as,

$$\frac{D^{disl}}{\dot{R}N_C} = \frac{1}{180bs} \cdot \frac{C_{\alpha'0}}{C_{V\alpha'0} - C_{V\alpha,eq}} \cdot \Delta\gamma_C \cdot n_{site}.$$
(4.18)

The first term on the right side $(1/_{180bs})$ is a constant. The second term is approximately constant since $C_{V\alpha'0} \gg C_{V\alpha',eq}$ and $C_{\alpha'0}$ is almost proportional to the $C_{V\alpha'0}$ at the initial



Figure 4.21: Optical micrographs of the axial section of SKF2 operated for $N = 1.49 \times 10^9$ cycles. The dark contrast shown at the depth of 400–800 μ m is not a DER but a segregation band which appears throughout the specimen.



Figure 4.22: Optical micrographs of (a) the axial section and (b) the circumferential section of SKF2 operated for $N = 1.49 \times 10^9$ cycles. Some WEBs are marked with yellow arrows.



Figure 4.23: Secondary electron micrographs of the axial section of SKF2 showing two features of the 30° WEBs, some of which are indicated with the arrows.



Figure 4.24: The relationships among parameters which can influence the kinetics of DER formation.

Therefore, dislocation assisted diffusivity can be controlled by $\Delta \gamma_C$ and n_{site} . state. n_{site} is determined by the limiting distance within which C atoms are more stable with dislocation than the carbide $(r_{d\theta} \text{ or } r_{d\epsilon})$ and r_{disl} (Section 4.1.1). The limiting distance can be controlled by changing carbide types in the alloy. In Eq. 4.8, factors affecting r_{disl} are T, $\Delta \gamma_C$, and RN_C . Although T is in the denominator of Eq. 4.8, its influence is greater for D due to the exponential dependence. The dependence of the kinetics of dislocation assisted tempering on above factors is summarised in Fig. 4.24a; note the marked boxes which denote that both enhancing and suppressing DER formation can be achieved by changing $\Delta \gamma_C$. This implies two competitive mechanisms by $\Delta \gamma_C$ which has been explained in Section 4.1.2. As a result, the complicated behaviours between D^{disl} and $\Delta \gamma_C$ shown in Fig. 4.8 is observed; however, the overall trend is that D^{disl} decreases with $\Delta \gamma_C$. This suggests that the direct influence of $\Delta \gamma_C$ on D^{disl} via Eq. 4.18 is less than its indirect effect via n_C . Hence, DER formation can be generally delayed with increasing $\Delta \gamma_C$. $\Delta \gamma_C$ depends on the test conditions and the bearing material behaviour. It can be increased with contact pressure as shown by the repetitive push test (Fig. 3.36), or it can be controlled with the ductility of the material.

The second way to delay DER formation is tailoring the microstructure via heat-

treatment by either increasing r_0 or reducing V_{f0} (Fig. 4.24b, Table 4.1). However, r_0 and V_{f0} cannot be controlled independently; both r_0 and V_{f0} generally increase during the tempering. Moreover, heat-treatment would also affect the carbide types; longer tempering results in transition from ϵ to cementite [15]. Therefore, intermediate tempering time is necessary to prevent early DER formation. Another option is to reduce the number of possible nucleation sites of temper carbides during tempering, which results in greater r_0/V_{f0} . This can be attained by reducing the crystal defects in the matrix, which are reported to be the carbide nucleation sites [15]; one way would be quenching to higher temperature after partial austenitisation. Changing the microstructure should be carefully designed since it may also influence $\Delta \gamma_C$ as well as carbide types, which may result in lower hardness.

To sum up, the kinetics of DER formation can be controlled through a choice of operating conditions and by tailoring microstructure. Carbide types, temperature, and speed are directly related to D^{disl} , while the initial microstructure varies r_0 and V_{f0} . $\Delta \gamma_C$ controls D^{disl} in a complex way; it may both increase and decrease D^{disl} depending on its range and temperature. $\Delta \gamma_C$ can be varied via changing the contact pressure or ductility of the material.

4.2.2 The limit in the model application

The special case of SKF2 shows that the model is not always applicable. Although it is generally believed that DERs precedes WEBs (Table 2.3, Fig. 2.11), the WEBs appeared without any indication of the DER in SKF2. The same phenomenon has been reported by Lund [26] for HRC 59 (~HV 673 [75]) stabilised alloy. Considering that the initial hardness of SKF2 was HV 716 \pm 9 (Table 4.5), it seems that DER does not appear for the material with relatively low hardness as suggested by Lund [26]. Another reason may be smaller amount of retained austenite due to longer tempering; this needs to be further investigated.

A plausible explanation comes from the change in the material behaviour during the

deformation. In the model, $\Delta \gamma_C$ is assumed to be constant throughout the calculation. However, $\Delta \gamma_C$ will increase for constant contact pressure as the material softens, and D^{disl} generally decreases (Fig. 4.8), limiting the C transport by dislocation for DER formation. Therefore, it is essential to estimate $\Delta \gamma_C$ of the materials with different hardness to identify the limit in dislocation assisted tempering.

It is not yet clear if the formation of WEB and DER is related. Since the WEB shows similar characteristics with the WEA (cellular structure, carbide dissolution, C supersaturation, and high hardness [34, 36]), it may form due to the stress concentration adjacent to "certain defects". However, the nature of such defects are unlikely inclusions as in the case of the WEA, since their morphologies are completely different; elongated bands versus the butterfly-shape. Moreover, no evidence is yet available for WEBs forming only in the softened matrix.

4.2.3 Dislocation assisted diffusivity vs. thermal diffusivity

Fig. 4.8 is replotted in logarithmic scale for D^{disl} in Fig. 4.25 to be compared with thermal diffusivity, $D = 6.2 \times 10^{-7} \exp\left(-\frac{80000}{RT}\right) \text{ m}^2 \text{s}^{-1}$ [9]. It is seen that the order of D^{disl} shown in Fig. 4.8 is similar to D at the relatively low temperature (20–40 °C) and D at 80–100 °C is larger than D^{disl} . However, hardness reduction is not detected other than in the region with maximum shear stress throughout bearing operation; this implies that supersaturated C in an unaffected region are segregated at defects such as martensite plate boundaries and pre-existing dislocations [76], and its thermal diffusion is prevented. Hence, it is worth emphasising that C diffusion assisted by dislocations does not mean that the thermal diffusion of C are enhanced during deformation. It is only via dislocation gliding that C can be transported for carbide coarsening since its diffusion is limited without deformation.



Figure 4.25: Comparison between dislocation assisted diffusivity (markers) and thermal diffusivity (horizontal lines) of C.

4.2.4 Interface reaction controlled growth

In the model presented in this chapter, it is assumed that carbide growth is governed by diffusion. In order to appraise this assumption, the velocity of interface-controlled growth can be estimated as:

$$v_{\theta,int} = \frac{M_{\alpha\theta} \Delta G_{m\theta}}{V_m},\tag{4.19}$$

where $M_{\alpha\theta}$ is the ferrite/cementite interface mobility, $\Delta G_{m\theta}$ is the driving force for cementite precipitation, and V_m is the molar volume. $\Delta G_{m\theta}$ is calculated by MatCalc version 5.50 and taken as 14.6 kJ/mol and $M_{\alpha\theta}$ is taken to be from 9×10^{-16} to 5×10^{-15} m⁴J⁻¹s⁻¹ [77]. Note that this $M_{\alpha\theta}$ value is appropriate for pearlite growth, which occurs at 677– 717 °C. Here, we take this value as the upper limit of the mobility. The resultant $v_{\theta,int}$ is 1.85–10.3 μ m/s, which is far larger than v_{θ} (< 10^{-6} μ m/s) in Equation 4.14. Hence, although the C transfer across the interface is required, the assumption of diffusion controlled growth is still valid.

4.3 Summary

DER is one of the principal forms of microstructural damage in bearing operation. It manifests from its different etching behaviour as well as hardness with respect to the unaffected region. These are attributed to carbide coarsening causing the matrix to gradually become depleted in C. Coarsening is of a different nature from thermally-assisted tempering since it is driven by plastic deformation, and not interfacial energy reduction. Therefore, dislocation assisted tempering is suggested: by creating a C atmosphere around a gliding dislocation, which can act as a mediator which transports C atoms from the matrix to the carbide, carbide coarsening may occur.

The amount of C that can be carried by dislocations is calculated by considering its dependence on the binding energy of C in the carbide and with a moving dislocation during the deformation. Then, dislocation assisted coarsening was calculated by applying Fick's first law to the C concentration profile at the time when a dislocation approaches a carbide. From this, C depletion in the matrix can be predicted and the resulting hardness decrease is prescribed.

The factors influencing the tempering kinetics during bearing operation are studied, and it is suggested that DER formation can be postponed by choosing the test conditions and microstructure of the material. Lower operation temperature, higher speed, large plastic strain of the material, more ϵ , larger carbide particles, and small carbide volume fraction will decelerate DER formation. Moreover, the calculation results were compared with available experimental data and the limit for model application is discussed.

Chapter 5

Stress concentration and the formation of white-etching areas

Contrary to dark-etching regions (DERs), which gradually form as a function of depth under the contact surface, white-etching areas (WEAs) are localised adjacent to cracks (Fig. 2.13). Therefore, WEA formation is proposed to result from severe deformation caused by stress raisers [36]. According to Chapter 4, such concentrated stress can cause large strain, i.e. fast dislocation glide which prevents C from being transported. Hence, DER formation would be prohibited in the surroundings of stress raisers; instead, a nanocrystalline structure forms and carbide particles dissolve [34, 78].

In this chapter, stress concentration nearby a crack is quantified employing the finite element method (FEM) to identify the conditions for WEAs formation. The microstructure and atomic distribution within a WEA is studied with advanced techniques, including transmission electron microscopy (TEM) and atom probe tomography (APT).

5.1 Stress concentration at a crack

The microstructure of a bearing steel may display defects which can act as stress raisers. In most cases, these may be the interfaces between a inclusion and the matrix or the cracks formed during cyclic loading with their typical dimension $\sim 1 \ \mu$ m. As a result, WEAs are



Figure 5.1: A spring (1) representing an element in FEM with two nodes 1, 2 on its sides.

observed with the inclusions and microcracks [22, 36, 57]. In this chapter, cracks formed with different sizes and orientations are introduced to an FEM model to study the effects on stress concentration. This follows earlier work where FEM was employed to obtain the stress distribution around the different types of inclusions [79, 80].

5.1.1 Finite element method

5.1.1.1 Spring analogy

FEM requires space discretisation, which is a process of dividing a body into a finite number of elements. The pattern formed by the elements is called as a mesh, and a point at which one element is connected to another is defined as a node. The whole body can be described as a system with a number of springs representing the elements.

One element (spring) with two nodes on each side is shown in Fig. 5.1. The applied forces (F_1, F_2) and displacements (u_1, u_2) at the respective nodes are called as nodal forces and nodal displacements. Element (1) is represented by its stiffness $k^{(1)}$, which is equivalent to a spring constant. Considering the force equilibrium of the element (1); i.e. $F_1 + F_2 = 0$,

$$F_1 = k^{(1)}(u_1 - u_2),$$

$$F_2 = k^{(1)}(u_2 - u_1).$$
(5.1)

$$\xrightarrow{\rightarrow u_1} F_1 \xrightarrow{k^{(1)}} F_2 \xrightarrow{k^{(2)}} F_3 \xrightarrow{k^{(3)}} F_4$$

$$\xrightarrow{\rightarrow u_4} F_4$$

Figure 5.2: A FEM system with three linearly connected springs.

Writing them in matrix form,

$$\begin{bmatrix} F_1 \\ F_2 \end{bmatrix} = \begin{bmatrix} k^{(1)} & -k^{(1)} \\ -k^{(1)} & k^{(1)} \end{bmatrix} \begin{bmatrix} u_1 \\ u_2 \end{bmatrix}.$$
 (5.2)

When the expression is extended by additional springs, for example three (Fig. 5.2), the force-displacement equation becomes [81]

$$\begin{bmatrix} F_1 \\ F_2 \\ F_3 \\ F_4 \end{bmatrix} = \begin{bmatrix} k^{(1)} & -k^{(1)} & 0 & 0 \\ -k^{(1)} & k^{(1)} + k^{(2)} & -k^{(2)} & 0 \\ 0 & -k^{(2)} & k^{(2)} + k^{(3)} & -k^{(3)} \\ 0 & 0 & -k^{(3)} & k^{(3)} \end{bmatrix} \begin{bmatrix} u_1 \\ u_2 \\ u_3 \\ u_4 \end{bmatrix}.$$
 (5.3)

It is seen that the matrix consisting of the stiffness of all elements is the combination of the stiffness matrix for individual elements;



Figure 5.3: A simplified diagram showing how the system variables are related in FEM. This is a powerful property of the stiffness matrix in FEM that allows a separate stiffness analysis per element, which can be combined into the global stiffness matrix, [K].

5.1.1.2 Stiffness matrix

As shown in Eqs. 5.2 and 5.3, the stiffness matrix, [K], relates the nodal displacements, [u], to nodal forces, [F]; i.e. [F] = [K][u]. In the spring analogy, [K] is introduced to be the equivalent to the spring constant, which would depend on the material response to deformation. However, [K] is not obtained in a straightforward manner since it is determined not only by stress-strain behaviour of the material but also by the discretisation pattern. Once [K] is known, unknown components of [u] or [F] can be calculated. [K] is calculated through three relationships between (i) [u] and the strain matrix $[\epsilon]$, (ii) $[\epsilon]$ and stress matrix $[\sigma]$, and (iii) $[\sigma]$ and [F] as shown in Fig. 5.3. [u] is related to $[\epsilon]$ through the nodal coordinates which are determined by the discretisation pattern including the element types and the nodal positions. The $[\epsilon]$ - $[\sigma]$ relationship comes directly from the material property. $[\sigma]$ and [F] are related by the principle of virtual work or minimisation of the total potential energy. During such process, $[\sigma]$ is integrated throughout the whole elemental volume, which would be affected by the discretisation pattern again; here, the positions of the integration points play a role as well. The important point is that the calculation should always give the same results with the appropriate discretisation, although the discretisation pattern affects [K].

5.1.2 Calculation conditions

5.1.2.1 General conditions

All FEM calculations were done employing ABAQUS CAE version 6.11.1. The system dimension and loading conditions are shown in Fig. 5.4. The system consists of a 1×1 mm²-two dimensional area where a constant pressure is applied on the top central part of its surface. The term 'a' is adopted to indicate the half-length of this region in analogy to Hertzian elastic contact (Fig. 2.3a). $p_0 = 3$ GPa was applied, which is a pressure similar to the test conditions given in Table 2.3. Note that the origin is at the centre of the top surface; x-axis is horizontal and z increases with depth. The bottom surface is always pinned; i.e. its nodal displacement is zero.

For the material behaviour, both elastic and plastic properties are given. The elastic modulus and the Poisson ratio were taken to be 210 GPa and 0.3, respectively (Section 3.2); the stress consistent with 0.002 strain was referred to as a yield strength for this model. For the plastic behaviour, the results from the compression test of a 100Cr6 steel which was partially austenitised at 840 °C and tempered at 149 °C are taken from Stickels [82] and shown in Fig. 5.5. Following the given trend line, the data were extrapolated up to a strain of 2, and input into the program.

5.1.2.2 Preliminary calculation

The aim of the preliminary calculation was to optimise a and choose the best pinning condition for the sides of the body, which resembles bearing loading. The mesh contained $10 \times 10 \ \mu \text{m}^2$ square CPS4R (continuum, plane stress, 4-node, reduced integration) elements. a was varied by 50, 100, 200 μ m and sides marked by empty triangles in Fig. 5.4 are either pinned or unconstrained.

The calculation results were investigated in terms of normal stresses (σ_x , σ_z), at x = 0according to z as shown by the dotted line in Fig. 5.4. Note that $\tau_{xz} = 0$ along the line and the principal shear stress (τ_1) can be obtained from Eq. 2.10. The corresponding results



Figure 5.4: A system investigated by FEM in this study. The red surface is where constant pressure of 3 GPa is applied. Pinned sides are marked with triangles. Filled triangles indicate the side which is always pinned, while empty triangles point the sides which is selectively pinned in the preliminary calculation.

are shown in Fig. 5.6 with the stress components obtained from Hertizian contact theory for comparison. All conditions show similar stress evolution compared with Hertzian contact theory. It is clear that the stress distributions with pinned sides (Fig. 5.6b,d,f) coincide better with Hertzian contact theory than those with unconstrained sides (Fig. 5.6a,c,e). This would be because limited displacement of the pinned sides can better represent the Hertzian stress (Fig. 2.3b), which vanishes at the edge of the contact area. The discrepancy in σ_x becomes remarkable with increasing a. While the $a = 50 \ \mu m$ case shows a monotonic decrease of $|\sigma_x|$ with depth (Fig. 5.6b), it decreases and then moderately increases for $a \ge 100 \ \mu m$ (Fig. 5.6d,f). Therefore, $a = 50 \ \mu m$ with pinned sides was chosen for simulating bearing operation, because the resulting stress distribution takes after what is predicted by the Hertzian theory.

The crack was introduced to the location showing the maximum τ_{xz} , as τ_1 can be reduced to maximum τ_{xz} when the rolling contact fatigue damage forms (Section 2.4). In the preliminary calculation, the maximum τ_{xz} equal to $0.28p_0$ resulted at (x, z) = $(\pm a, 0.4a)$, which are not far from those in Table 2.2; this suggests that the simulation condition is plausible although the loading condition does not exactly correspond to that



Figure 5.5: The material plastic property obtained by compression tests with the 100Cr6 steel, which was partially austenitised at 840 °C and tempered at 149 °C. Both experimental data and the trend lines were adopted from Stickels [82].

Table 5.1: The magnitude of the τ_{xz} at $(x, z) = (\pm a, 0.4a)$ according to the element size.

Element size	$10 \times 10 \ \mu m^2$	$5 \times 5 \ \mu m^2$	$2 \times 2 \ \mu m^2$	$1 \times 1 \ \mu m^2$
	$0.28p_0$	$0.30p_0$	$0.30p_{0}$	$0.30p_{0}$

of Hertzian contact.

Additional calculations were carried out with smaller elements to understand the effect of mesh on the stress distribution. τ_{xz} values at $(x, z) = (\pm a, 0.4a)$ for different element size are given in Table 5.1. It is seen that the difference in τ_{xz} was not significant; therefore, the crack was introduced at $(x, z) = (\pm a, 0.4a)$ for the calculation hereafter.

5.1.2.3 Stress concentration around a crack

A crack is introduced in the subsurface as shown in Fig. 5.7a. The centre of the crack was located at the one of the maximum τ_{xz} points, (x, z) = (a, 0.4a). The orientation of the crack (θ_{crack}) was varied from 0° to 90° with respect to the x-axis in 10° steps and the sizes of the crack (d_{crack}) were 1, 0.5 and 0.25 μ m. The crack is assigned as a seam in ABAQUS, which introduces independent double nodes on the line. However, the interaction between these nodes was not defined; i.e. no sliding or contact between the crack surface is assumed, and overlapping of the nodes is allowed. Therefore, this condition assumes the initial stage when crack has just nucleated, and deformation along the crack was not taken into account.

The mesh is refined adjacent to the crack by providing different seed spacings. The seed spacing was 10 μ m for the sides in solid lines in Fig. 5.7b and 5 μ m for the surface in dotted lines. The surface region marked in red above the crack was given 1 μ m-spacings. 100 nm-spaced mesh seeds are given along the crack and also on four lines are created adjacent to it, which are shown in Fig. 5.7c. Therefore, ten, five, and three elements lie on 1, 0.5, 0.25 μ m cracks, respectively. An example of the resulting mesh for 1 μ m, 0° crack is shown in Fig. 5.8. The element was CPS4R, which was the same element as in the preliminary calculation and no significant level of element distortion was reported.

After calculation, τ_{xz} values were obtained at the integration point of each element.



Figure 5.6: The normal stresses (σ_x, σ_z) obtained from the preliminary calculation. Lines are the stress state calculated by Hertzian elastic theory and markers are calculated nodal stresses data from FEM for (a,b) $a = 50 \ \mu m$, (c,d) $a = 100 \ \mu m$, and (e,f) $a = 200 \ \mu m$. (a,c,e) were calculated with the unconstrained and (b,d,f) were with the pinned condition for the sides marked with empty triangles in Fig. 5.4.



Figure 5.7: (a) Configurations of the introduced cracks and seeded edges (b) on the sides of the body and (c) near the crack for the mesh construction.


Figure 5.8: An mesh construction example for a crack with $d_{crack} = 1 \ \mu m$, $\theta_{crack} = 0^{\circ}$. (a) shows the whole body, and (b,c) are magnified images of the circled region in (a,b), respectively. The crack is marked by a thick red line in (c).



Figure 5.9: Schematics showing (a) the nodes and integration points adjacent to the 500 nm crack, (b) paths and (c) elements where the data were obtained.

The elements and nodes near the crack are schematically shown in Fig. 5.9a. For CPS4R elements which are almost square adjacent to the crack, the integration points lie almost at the midpoints of the elements. The data were obtained from the elements of the lower and upper layers along the crack extension line. The origin was set to be at the left end of the crack and four sets of data will be presented for each condition following Path 1 to Path 4 in Fig. 5.9b. The data obtaining points were the integration points, which were $x_{crack} = \pm 0.5, \pm 1.5, \pm 2.5 \,\mu\text{m}$ and so on. However, the data directly above and below the crack, e.g. at $x_{crack} = 0.05, 0.15, 0.25, 0.35, 0.45 \,\mu\text{m}$ for the 500 nm crack, were not plotted because they are not meaningful without the interaction properties as mentioned before. The data for each element next to the crack, which are marked as i–iv in Fig. 5.9c will also be presented.

5.1.3 Results

The contour maps of τ_{xz} near the crack are shown in Fig. 5.10. For all cracks, there are two pairs of stress concentration regions at the end of the crack; they will be called "wings" here. One pair of wings are parallel to the surface while the other are perpendicular and the size is similar for the wings in the same direction. When the crack is tilted, the size of each pair changes together as schematically drawn in Fig. 5.11. For example, when $\theta_{crack} = 0^{\circ}$, the perpendicular wings are larger than the than the horizontal wings. When $\theta_{crack} = 40^{\circ}-60^{\circ}$, all wings assume a similar size, while the different sized wings occur again as $\theta_{crack} = 90^{\circ}$ where the horizontal wings become larger than the perpendicular wings. Moreover, it is obvious that the stress concentration is higher for larger cracks.

The one-dimensional profiles of τ_{xz} along the paths in Fig. 5.9b are given in Fig. 5.12. The plot in Fig. 5.12a shows the case for different crack sizes and plots in Fig. 5.12b–e are for 1 μ m cracks with different orientation and paths. Despite numerical fluctuations, stress profiles for all conditions showed a similar trend in that the stress is mostly concentrated at the crack tip, and their concentration reduces and smoothens out away from the crack, which is consistent with the literature [42]. Path 1 and 4 as well as Path 2 and 3 share very similar stress profiles, which is observed for all conditions. Moreover, all cases share the feature that the concentration is more pronounced for larger d_{crack} (Fig. 5.12a).

Since the evolution of stresses according to θ_{crack} is not monotonic, stresses at the four elements at the crack tip (Fig. 5.9c) are shown in Fig. 5.13a for $d_{crack} = 1 \ \mu$ m. The values were shown relative to τ_{xz0} , which is defined to be τ_{xz} in absence of the crack and obtained from the preliminary calculation (Table 5.1). Two element pairs showed similar trend; Elements i and iv as one pair and Elements ii and iii as another pair. Considering the stress state during the rolling contact where the ball or roller rolls over the inner ring, the applied pressure moves relative to the crack and Elements i and iii would experience the same stress state as Elements ii and iv, respectively. Plus, considering the similar stress state for Path 1, 4 and Path 2, 3 (Fig. 5.12), the stress state of all Elements i–iv during RCF would be very similar and the strongest stress concentration among the elements





Figure 5.10: Contour maps of τ_{xz} for different crack sizes (a_{crack}) and orientations (θ_{crack}) . (a) $\theta_{crack} = 0^{\circ}-40^{\circ}$. (b) $\theta_{crack} = 50^{\circ}-90^{\circ}$. A map on the upper right hand corner corresponds to the stress state at the same location without a crack.



Figure 5.11: Schematics of stress concentration regions ("wings") for different crack orientation.

would be the most important. Hence, larger value was selected between τ_{xz} of Elements i and ii, and it is plotted with respect to θ_{crack} in Fig. 5.13b. It is seen that τ_{xz} does not depend significantly on θ_{crack} and always in the range of 1.3–1.8.

In summary, the effect of crack size and orientation is quantified employing FEM. The preliminary calculation without a crack suggested that the stress state originated from the uniform pressure applied to the finite area was similar to that from Hertzian contact. In the final calculation, a crack was introduced and the shear stress at the crack tip was analysed. The size of the stress concentrated region was controlled by the crack size and orientation. However, the factor of the shear stress (τ_{xz}) concentration was always in the range of 1.3–1.8 and did not show a significant dependence on the crack orientation.

5.2 Characterisation of carbide dissolution

5.2.1 White-etching area and the similar phenomena

White-etching areas (WEAs) are reported to form around the cracks or inclusions [11, 34]. Since the crack is a stress raiser and the inclusion/matrix interface can develop into a crack, it is likely that WEAs are the result of higher degree of deformation close to the crack. As mentioned in Section 2.3.3, the microstructure inside the WEAs is very different from that of the matrix; a nanocrystalline structure forms and carbide particles dissolve [34, 78, 83, 84]. Regions showing the similar etching behaviour are also observed



Figure 5.12: One dimensional evolution of the normal stress τ_{xz} along the paths shown in the upper right corner. (a) is for $\theta_{crack} = 0^{\circ}$ with different crack sizes ($d_{crack} = 1, 0.5, 0.25 \ \mu$ m) and (b–e) are for $d_{crack} = 1 \ \mu$ m with different crack orientations ($\theta_{crack} = 0, 40, 90^{\circ}$).



Figure 5.13: Evolution of τ_{xz} with respect to the crack orientation (θ_{crack}) (a) for all elements at the tip of the 1 μ m crack as shown in the top and (b) considering rolling contact cycles.

in pearlitic railway steels which suffer from both rolling and sliding cycles [85–88]. The damage is manifested as the white-etching layer (WEL) right below the surface, the thickness of which is tens of μ m.

Interestingly, these microstructural changes are not only limited to WEAs and WELs, but also observed during severe plastic deformation (SPD). In this case, they do not show the localised region with different etching behaviour, but the alteration takes place throughout the material. Reported examples of SPD where either nanocrystalline structure or carbide dissolution is observed include drawing of pearlitic steels or martensitic steels, mechanical milling (MM), high pressure torsion (HPT), ball drop test, and surface mechanical attrition treatment (SMAT). A summary of the references is shown in Table 5.2, according to their observed microstructures. Here, many microstructural features are interconnected; because of carbide deformation, carbide particles dissolve resulting in nonstoichiometric cementite and a C-rich matrix. Fine carbide particles are sometimes seen; however, it is not clear whether they are caused by incomplete dissolution or reprecipitation. An interesting feature in the distribution of alloying elements is that both homogeneous and heterogeneous distributions are observed. Although it may be ironic to observe such contrary phenomena in the same deformation process, it suggests that the alloying element distribution may depend on the degree and rate of deformation. It is also seen that the microstructural features were mostly observed by TEM and the atomic distribution of alloying elements was studied with APT.

Since the distribution of alloying elements in WEAs has never been investigated, APT is employed here. Moreover, TEM is adopted to find whether carbides are completely dissolved [37], or small carbide particles are present in the WEA [78, 89].

Table 5.2: Microstructural chang techniques are presented in paren high pressure torsion, SMAT: surf microscopy, SEM: scanning electr analysis, APFIM: atom probe field	es observed in rolli thesis. (WEL: wh face mechanical att on microscopy, FI ion microscopy)	ng contact fatigue (RCF) and severe plastic deformation (SPD). Employed ite-etching layer, WEA: white-etching area, MM: mechanical milling, HPT: rition treatment, TEM: transmission electron microscopy, SIM: scanning ion M: field ion microscopy, XRD: x-ray diffractometry, TMA: thermomagnetic
Microstructural observation	Deformation	References (techniques)
Nanocrystalline structure	RCF(WEL)	[85](TEM), [86](TEM, SIM)
	RCF(WEA)	[83](TEM), [84](TEM), [78](TEM), [37](TEM), [89](TEM, SEM)
	Cold drawing	[90](TEM), [91](FIM), [92](TEM)
	MM	[93](TEM), [94](TEM), [95](TEM)
	HPT	[96](FIM), [97](TEM)
	Ball drop test	[98](TEM)
	SMAT	[99](TEM)
Carbide dissolution	RCF(WEL)	[85](XRD), [86](TEM), [87](TEM), [37](TEM), [89](TEM)
	RCF(WEA)	[83](TEM), [78](TEM)
	Cold drawing	[100](TMA, Mössbauer), [101](Mössbauer), [90](TEM), [102](TEM), [103](TEM),
		[104](APT), [92](APT)
	MM	[93](TEM)
	HPT	[96](APT), [105](TEM), [97](TEM, magnetic saturation, XRD)
	Ball drop test	[98](TEM)
	SMAT	[99](TEM)
Carbide	RCF(WEL)	[85](TEM), [87](TEM)
${ m fragmentation/elongation}$	RCF(WEA)	[83](TEM), [37](TEM)
	Cold drawing	[106](TEM), [107](FIM), [90](TEM), [104](TEM), [108](TEM)
	HPT	[109](SEM)

	SMAT	[99](TEM)
Fine carbide particles	RCF(WEL)	[86](APT)
	RCF(WEA)	[78] (TEM)
	Cold drawing	[102](TEM), [104](TEM)
Nonstoichiometric cementite	RCF(WEL)	[86](APT)
	Cold drawing	[110](APT), [106](APT), [111](APT), [102](EELS), [104](APT), [108](TEM),
		[92](APT)
	HPT	[96](APT)
Higher C content in matrix than	RCF(WEL)	[85](APT), [86](APT)
its equilibrium content	Cold drawing	[110](APT), [106](TEM), [91](APFIM), [104](APT), [112](Synchrotron XRD)
	MM	[93](APT), [95](APT)
Uniform C distribution	RCF(WEL)	[85](APT)
	Cold drawing	[103](APT/FIM)
C segregation to	RCF(WEL)	[86](APT)
crystalline defects	Cold drawing	[113](APFIM), [106](TEM), [107](FIM), [91](APFIM), [92](APT)
	MM	[93](APT), [95](APT)
	HPT	[96](APT)
Uniform substitutional alloying	RCF(WEL)	[86](APT)
element distribution	Cold drawing	[110](APT), [114](APT)
Substitutional alloying element	RCF(WEL)	[85](APT)
segregation to crystalline defects	MM	[95](APT)
High hardness	RCF(WEL)	[86]
	RCF(WEA)	[89]
	Cold drawing	[90], [108]
	MM	[94]
	Ball drop test	[98]

5.2.2 Experimental

5.2.2.1 Atom probe tomography

APT is a powerful tool to study atomic distribution in a localised area. It has been employed in a number of studies as shown in Table 5.2. The sample is needle-shaped, the tip radius of which is about 50–100 nm [115]. High voltage pulse cycles are applied with a nano-second duration at cryogenic temperatures. As a result, the surface atoms at the tip form an atmosphere of ions. Among these ions, only those that pass the probe aperture are analysed in the mass spectrometer. The basic principle of analysis starts with the energy conservation principle, in which the potential energy of ion at the surface is equal to the kinetic energy of ion when it is evaporated [115, 116];

$$n_{ion}eV_{APT} = \frac{1}{2}m_{ion}v^2.$$
 (5.5)

$$\therefore \ \frac{m_{ion}}{n_{ion}} = 2eV_{APT} \left(\frac{d_{ion}}{t_{ion}}\right)^2, \tag{5.6}$$

where n_{ion} , m_{ion} , v, d_{ion} , t_{ion} are the charge, mass, velocity, flight distance, and flight time of the ion, respectively, e is the elementary charge of an electron, and V_{APT} is the applied voltage. Hence, once d_{ion} , t_{ion} , and V_{APT} are known, the mass-to-charge ratio (m_{ion}/n_{ion}) can be acquired and is accumulated during the measurement. From this, each atom detected from a certain location is chemically determined.

When the system contains C, C ions usually form compounds [117]. Since only the mass-to-charge ratio is recorded in APT, some of these species may not be identified correctly. Considering the experimentally determined abundance of these species in a Fe-C system, it is found that approximately 10% of total C content is underestimated in the general case where C_4^{++} is regarded as C_2^+ [117]. On the other hand, Fe can also be underestimated due to the pile-up effect where multiple ions arrive at the detector so closely that they cannot be recorded separately [118]. Moreover, some alloying elements can be either overestimated or underestimated due to its evaporability when the measurement

	С	Cr	Mn	Si	Mo	Ni	Cu
wt%	0.92 - 1.02	1.65 - 1.95	0.25 - 0.40	0.25 - 0.40	0.15 - 0.25	0.25(max.)	$0.30(\max.)$
$\mathrm{at}\%$	4.12 - 4.56	1.70 - 2.01	0.24 - 0.39	0.48 – 0.76	0.08 - 0.14	$0.23(\max.)$	$0.25(\max.)$

Table 5.4: Composition range of the sample studied for the white-etching area. Fe content is balanced accordingly.

takes place above 65 K [119]. However, these errors do not significantly influence the APT results and solute redistribution has been successfully investigated.

5.2.2.2 Sample preparation

An RCF-tested sample has been supplied by SKF. Its composition range is shown in Table 5.4. Comparing it to Table 2.1, the sample composition is within the limits of 100CrMo7, except for a slightly higher Si content. The sample was partially austenitised at 830–870 °C and quenched in a salt bath, followed by tempering at 230–250 °C. The microstructure after heat treatment was tempered martensite containing both residual cementite and temper carbides, as shown in Fig. 5.14. The austenite amount was less than 1 vol% as confirmed by XRD. The sample preparation and experimental methods for studying the initial microstructure are the same as described in Section 3.2.1.2.

During RCF-testing, a WEA appeared adjacent to the crack at ~200 μ m under the surface, as shown in Fig. 5.15a. A TEM sample and two APT samples were extracted from this area employing the lift-out method by focused ion beam (FIB) [120, 121]. FEI Helios NanoLab was employed for preparing TEM sample. Firstly, a Pt protective later was deposited on the region of interest and it was roughly cut with a 2–15 nA beam current. Then, an ~20 × 5.5 μ m² area was cut out and finally thinned with the beam current of 100–1000 pA. A Philips CM30 with a 300 keV-electron beam was adopted to study the microstructure. APT samples were extracted with a Zeiss NVision dual column FIB. An ~20 × 3 μ m² protective Pt strap was deposited for each sample region and a bar-shaped volume was isolated and mounted on a Si microtip post. Each section was finally sharpened to the tip radius <50 nm with the Ga ion-beam. In order to prevent



Figure 5.14: (a) A secondary electron micrograph and (b) a bright field transmission electron micrograph of the sample microstructure after the heat treatment.

Ga damage, the final milling was done with a low beam energy (2 keV). The prepared samples were investigated in a Local Electrode Atom Probe (LEAP) 3000HR and the data were reconstructed and analysed using IVAS software (version 3.6.0) [122].

Since Ga implantation is unavoidable, Ga damage was assessed in both APT samples. Only a trace amount of Ga was detected in both regions, except at the tip of Region 1 shown in Fig. 5.16; this region is excluded from the investigation.

5.2.3 Results

5.2.3.1 Microstructures

As its name implies, a WEA can be detected by a lighter colour in contrast with the matrix by OM (Fig. 5.15a,b). However, this contrast could not be identified with SEM; instead, the WEA showed a smooth topology which might be caused by smearing out of residual carbide particles (Fig. 5.15c). The direction of such flow-like morphology was parallel to the crack in contact with the WEA. Although small cavities were observed, this feature also appeared in the matrix. Moreover, cavities were not observed in TEM samples, which suggests that the cavities detected by SEM were caused not from RCF



Figure 5.15: (a) An optical micrograph showing the location of the white-etching area of interest. The marked regions in (a,b) are magnified in (b,c), respectively. (c) is a secondary electron micrograph showing the region where TEM and APT samples are obtained. (b, c) are adopted from [123].



Figure 5.16: The reconstructed volume of Region 1. 1.5 at%C isoconcentration surfaces are shown in dark brown, and Ga ions are shown with blue dots.

but from sample preparation.

The TEM sample was selected to contain three different regions; the matrix outside the WEA, the WEA, and the crack around which the WEA appears (Fig. 5.17). Therefore, the WEA/matrix interface as well as the region near the crack could be investigated, and they are shown in Fig. 5.18. All three regions showed a clear nanocrystalline structure; it is assumed that the structure boundaries corresponded to dislocation tangles and the structure is referred to as dislocation cells from now on. Although the cells appeared in different sizes, they were randomly distributed throughout the sample regardless of the position within the sample. This is different from what is observed in butterflies where the cells become coarser away from the inclusion [78, 89]. A diffraction pattern and images of the cells are shown in Fig. 5.19. The pattern consisted of a series of rings which corresponded to the lattice spacing of ferrite implying the random orientation of a number of cells within the selected area. The cells were smaller than 50 nm as seen in the dark field image.

The sharp WEA/matrix interface was observed as shown in Figs. 5.15 and 5.20. The



Figure 5.17: (a) An optical micrograph of the TEM sample prepared by the FIB. A crack located in the sample edge is seen. (b) A schematic of the sample with different regions of interest.



Figure 5.18: (a,b,c) Corresponding bright field transmission electron micrographs of the regions (i, ii, iii) in Fig. 5.17b.



Figure 5.19: The bright and dark field images, as well as the diffraction pattern of the region in Fig. 5.18b. The positions of the selected area aperture for the diffraction pattern and the objective aperture for the dark field imaging are marked in yellow. A portion of the $(110)_{\alpha}$ reflection rings was chosen for the dark field imaging.

matrix is typical tempered martensite similar to the initial structure in Fig. 5.14b. Note that the matrix has also experienced some degree of deformation.

One decaying particle of residual carbide was detected at the matrix/WEA interface (Fig. 5.21) and was tilted to the $[111]_{\theta}$ zone. When a $(110)_{\theta}$ reflection was chosen, the dark field revealed not only the residual carbide but also small scattered particles in the WEA. Also, Fig. 5.22 shows another dark field image of the scattered particles. In the diffraction pattern displaying rings of the ferrite lattice, the reflection inside the $(110)_{\alpha}$ is selected to create the dark field image. Since both reflections chosen above correspond to the plane with its spacing larger than $(110)_{\alpha}$, it is most likely that these particles are carbide. The size of the cells and carbide particles can be well compared in Fig. 5.23; the cells (< 50 nm) are usually larger than carbide particles (< 10 nm).

One interesting feature has been revealed within the WEA. In Fig. 5.24, two 200 nm sized-features are observed. When the selected area aperture was placed in the marked area, diffraction pattern in $\langle 111 \rangle_{\alpha}$ zone stressed out with a dim ferrite diffraction ring pattern. When one of $\{110\}_{\alpha}$ reflections was selected, these features gave the contrast to the dark field image, which suggests that these features are ferrite grains in the $\langle 111 \rangle_{\alpha}$ zone.



Figure 5.20: The region containing both the matrix and the white-etching area. The boundary between them is indicated with arrows.



Figure 5.21: The bright and dark field images of the matrix/white-etching area boundary region with the decaying cementite, which was selected (in a yellow circle) to create the diffraction pattern shown. The selected $(110)_{\theta}$ reflection to create the dark field image is marked in the pattern [123].



Figure 5.22: The bright and dark field images, as well as the corresponding diffraction pattern marked in the bright field image. The selected reflection to create the dark field image is marked in the pattern and was within the $(110)_{\alpha}$ reflection ring.



Figure 5.23: The dark field images of (a) the nanocrystalline cells and (b) the carbide particles [123].



Figure 5.24: The bright and dark field images, as well as the diffraction pattern in one region inside the white-etching area. The positions of the selected area aperture for the diffraction pattern and the objective aperture for the dark field imaging are marked in yellow. A $(110)_{\alpha}$ reflection was chosen for the dark field imaging.

Therefore, they may be either the intermediate state transforming into the nanocrystalline structure or Ga-damaged regions.

5.2.3.2 Distribution of alloying elements

The approximate volumes investigated by APT were $80 \times 80 \times 120 \text{ nm}^3$ and $80 \times 80 \times 180 \text{ nm}^3$ (Fig. 5.25a,b), and the total number of detected ions by APT was 1.98×10^6 and 3.74×10^7 for Region 1 and Region 2, respectively. The compositions obtained from the number of detected ions are given in Table 5.5. Comparing them with the alloy composition shown in Table 5.4, the content of substitutional alloying elements lies in the range while that of C is significantly lower; this is attributed to the small scattered carbide particles (Figs. 5.21, 5.22, and 5.23b) were not sufficiently covered in the APT sample; only two C clusters have been detected in Region 1. Therefore, the low C content shown in Table 5.4 seems to represent the C content in the nanocrystalline matrix without carbide particles.



Figure 5.25: (a,b) Overall cell structures. (c,d) Atomic distributions in 10 nm-thick slices perpendicular to the in-depth direction of the specimen, the position of which is shown by arrows in (a,b). (e,f) Small polygonal cells enveloped by C in (a,c,e) Region 1 and (b,d,f) Region 2 studied by the APT. Red and black points represent Si and C atoms, respectively [123].

Table 5.5: Detected atomic percent in Region 1 and Region 2. Only major elements has been considered and data were background-corrected. The error ranges from the mass spectrometer were less than 0.001 at% for all elements except Cr, which range was 0.003 at%.

Region	C / at%	Cr / at $\%$	Mn / at%	Si / at $\%$	Mo / at%	Ni / at%	Cu / at%
1	0.319	1.747	0.347	0.562	0.089	0.125	0.138
2	0.250	1.972	0.343	0.529	0.081	0.126	0.043

Since Si and C showed the strongest segregation among the alloying elements, their spatial distribution is shown in Fig. 5.25a,b. Their segregation can be visualised by 10 nm slices in Fig. 5.25c,d taken from the needles. The overall morphologies of both Region 1 and Region 2 are similar; they are composed of nanocrystalline cells, which boundaries are rich in Si or C. These cell structures showed two types of morphologies; (i) small (< 50nm) polygonal cells which were enveloped only by C (Fig. 5.25e,f), and (ii) large cells of length over 100 nm as manifested by Si and C segregation; the large cells were only partially covered by Region 1, and their morphology can be better appreciated in Region 2. The size of the small cells agrees with what is observed by TEM in Figs. 5.19 and 5.23a. The large cells were sometimes elongated and twisted. Since this morphology is not observed by TEM, it suggests that Si and C do not segregate to all boundaries of the cells observed by TEM. In other words, each cell appearing with Si and C segregation from the APT study is the group of several cells shown by TEM. The degree of segregation at the cell boundaries usually reaches up to 2.0-3.0 at% for both Si and C as shown in Figs. 5.26 and 5.27. Note that the region in Fig. 5.27 showing ~ 6 at%C may be the part of a C cluster.

C segregation is not only limited to the cell boundaries but also observed in the form of clusters within the cell. As shown in Fig. 5.28, the size of the cluster is \sim 7 nm which is consistent with the scattered carbide from TEM observation (Figs. 5.21, 5.22, and 5.23b). Moreover, C content reaches only up to \sim 9 at% which is lower than cementite (25 at%) and ϵ (29 at%); such off-stoichiometry has been reported frequently in the literature (Table 5.2). Moreover, precipitation of the nonstoichiometric carbide has not



Figure 5.26: (a,b) C atom and isoconcentration (1.7 at%) surface constructions across a cell boundary in Region 1. (c) One dimensional concentration profile across the cell boundary based on the number of atoms detected within the cylinder in (a,b), which has a 3 nm diameter [123].



Figure 5.27: (a,b) Si and C atom and isoconcentration (1.7 at%C, 1.5 at%Si) surface constructions along the cell boundaries in Region 2. (c) One dimensional concentration profiles based on the number of atoms detected within the cylinders in (a,b), which have a 5 nm diameter [123].



Figure 5.28: (a,b) C atom and isoconcentration (1.7 at%) surface constructions of one cell in Region 1 showing two C clusters. (c) One dimensional concentration profile based on the number of atoms detected within the cylinder in (a,b), which has 3 nm diameter [123].

been observed. Hence, it is suggested that these small carbide particles are the remaining parts of carbide dissolution considering that nonstoichiometry may be caused by vacancy formation during the process [96]. However, the nature of the small carbide particles needs further clarification.

Another interesting feature is that C and Si seem to avoid each other. This is obvious in Figs. 5.26c and 5.27c2 where C and Si peaks do not overlap. In the case when Si peaks do not appear, Si concentration is suppressed in the region rich in C (Figs. 5.27c1 and 5.28c). This behaviour may be the result of Si increasing C chemical activity [124]. Hence, the interaction between Si and C would play an important role in C diffusion and affect the kinetics of WEA formation.

In summary, the WEA adjacent to the crack contained a homogeneous structure with

fine cells (< 50 nm) and scattered carbide particles (< 10 nm). Although the terminology 'cell' used here needs a clearer definition, Si and C atoms are segregated to the part of the cell boundaries. Additional C segregation occurs to form C clusters, the size of which corresponds to the scattered carbide particles observed by the TEM. These clusters in the cells contained less C (< 25 at%) than normal carbides. Moreover, Si and C seem to avoid each other, which may influence the kinetics of nanocrystalline structure formation and carbide dissolution.

5.3 Discussion on white-etching area formation

Based on the FEM calculation and experimental studies on the WEA, its formation is discussed in this section. According to the FEM results, the size of the stress concentrated area depends on the crack orientation while the degree of the concentration at the crack tip does not vary significantly. This is consistent with WEAs formed adjacent to the crack which does not depend on the crack orientation (Fig. 2.13). Besides, the directionality of WEAs in butterflies [89] may be explained by that the butterflies may be formed in the region showing large gradient of stress concentration in the contour map (Fig. 5.10). However, it is suggested that the whole rolling process involving the pressure moving over the body should be considered as in [61], or the effect of inclusions [34] needs to be taken into account for the complete explanation.

The discrete WEA/matrix interface suggests that the phenomenon responsible for the WEA formation is very sensitive to the changes in the degree of deformation caused by the presence of the crack. The responsible phenomena for the WEA formation would be suddenly activated when the critical deformation level is reached. Therefore, it is suggested that there exists a threshold stress level beyond which carbide dissolution and nanocrystalline structure formation suddenly take place as will be considered in Chapter 6.

The mechanism of the WEA formation can be suggested based on these studies and [96] which introduced a mechanism for nanocrystalline structure formation during SPD. When a crack is present in the material, the stress level is raised adjacent to the crack as calculated by FEM and these regions are deformed more severely. Moreover, more cracks can be generated by stress concentrators, which synergistically further increases the stress concentrated area. As a result, dislocations can start to deform carbides by their accumulation at the matrix/carbide interface and possibly by carbide shearing. As carbides deform, they become unstable and start to dissolve, suggesting their free energy increase during the process. With the APT results showing C segregation to cell boundaries, it is suggested that C diffuses away from the carbides by pipe diffusion along the dislocations which entangles during deformation to form the cell boundaries. This C diffusion would be affected by Si which also segregates to the cell boundaries and increases C activity. Moreover, when C atoms are located in the dislocation strain field, the dislocation cell growth is discouraged by the pinning effect of C, resulting in a very fine cell structure. This may also facilitate the dislocation accumulation at the cell boundary leading to high angle cell boundaries.

5.4 Summary

In this chapter, phenomena regarding the WEA are investigated with FEM calculation and advanced experimental techniques. The stress was found to be concentrated at the crack tip with the applied stress of 3 GPa. The degree of stress concentration did not depend much on the crack orientation although the size of the stress concentrated region varied for different conditions. The shear stress (τ_{xz}) increases by a factor of ~1.8 when a 1 μ m crack is present. Within the WEA, a homogeneous nanocrystalline cell structure and small scattered carbide particles were observed; the cell size was less than 50 nm and the scattered particles were smaller than 10 nm. The scattered carbides contained less than 25 at%C. Some of the cell boundaries were decorated with both Si and C atoms while some cells were only enveloped by C atoms. The interesting feature that Si and C atoms avoid each other is suggested to affect the kinetics of WEA formation which involves C diffusion through the cell boundaries.

Chapter 6

Dissolution of carbide particles

One of the interesting features appearing during rolling contact fatigue (RCF) is that the damage manifested by completely opposite microstructure evolution forms simultaneously. An adequate degree of deformation encourages carbide coarsening to form dark-etching areas (DERs) via gliding of dislocations transporting C atoms, as introduced in Chapter 4. However, when deformation gets more severe by stress raisers within the material, C cannot keep up with dislocation glide, and carbide particles are observed to dissolve and generate white-etching areas (WEAs).

In this chapter, carbide dissolution in WEAs is studied. Firstly, the driving force of the carbide dissolution is discussed. With two different schemes, the accumulated driving force during RCF is estimated and used to calculate the reduced amount of carbide at equilibrium. This represents the carbide dissolution assuming that the equilibrium is reached immediately after a driving force is provided to the system. However, since the dissolution requires not only sufficient driving force but also C diffusion, diffusion controlled kinetics is investigated as well.

6.1 Driving force

The driving force for carbide dissolution in the WEA is very different from that of carbide coarsening occurring in the DER. In the case of the DER, the driving force is inherent to

the initial structure with underdeveloped carbides and the matrix supersaturated with C, which has not reached equilibrium. Therefore, the carbide fraction is ready to increase once C diffusion from the matrix is possible. However, C diffusion can only be achieved through deformation at low temperature. During bearing operation, appropriate amount of deformation is provided so that dislocations can glide with a C atmosphere to promote carbide coarsening and DER generation.

On the other hand, carbide dissolution differs from carbide coarsening in that its driving force is not present in the initial structure. Additional energy can be provided by deformation and accumulated in the microstructure over a number of stress cycles making carbides unstable. In other words, the additional energy given by deformation would increase the Gibbs free energy of carbides as shown in Fig. 6.1. By adding the energy, G_{diss} , the Gibbs free energy of the precipitate increases from $G^{k,i}$ to $G^{k,f}$. As a result, the precipitate phase fraction is reduced, and C content in the matrix is enriched from $X_C^{\alpha,i}$ to $X_C^{\alpha,f}$. Therefore, we define this additional energy, G_{diss} , as the driving force for the carbide dissolution. Note that G_{diss} is only added to the carbide energy although both matrix and carbide particles equally experience deformation. This can be explained by the microstructure in [99] where a number of dislocations were observed to accumulate at the matrix/carbide interface and the carbide elongation is caused by the deformation. Moreover, the ratio of interface area to its volume is much less for the matrix than the carbide in the system; thus, the interfacial area would be a smaller portion of the matrix compared to the carbide. Therefore, a higher degree of deformation is felt by carbide particles rather than the matrix.

 G_{diss} can be determined quantitatively with (i) macroscopic and (ii) microscopic approaches. The macroscopic approach employs the plastic deformation behaviour of the material which is obtained from the results of the repetitive push test (Section 3.3) and the stress concentration calculation (Section 5.1). The energy per stress cycle during the bearing operation in the unit volume of the material is the area within its stress-strain hysteresis loop (Fig. 2.16). However, taking only half of \overline{AB} is the strain responsible for



Figure 6.1: A schematic Gibbs free energy (G) diagram with respect to C mol fraction (X_C) . G^{α} is the Gibbs free energy of the matrix, and $G^{k,i}$ and $G^{k,f}$ is that of the precipitate before and after adding the driving force for its dissolution (G_{diss}) . X_C^k is the C mol fraction in the precipitate, and $X_C^{\alpha,i}$ and $X_C^{\alpha,f}$ are that in the matrix before and after adding G_{diss} [125].

plastic deformation as mentioned in Section 2.4, the accumulated plastic energy per cycle U_C can be estimated as

$$U_C = 2\tau_a \Delta \gamma_C,\tag{6.1}$$

where τ_a is plastic shear stress amplitude experienced by the material defined in Fig. 2.16, and $\Delta \gamma_C$ is the plastic shear strain range per stress cycle. Following Section 2.4, τ_a would be equal to the magnitude of the maximum τ_{xz} which is 0.25 p_0 (Table 2.2). Therefore,

$$U_C = 0.5 p_0 \Delta \gamma_C. \tag{6.2}$$

To relate U_C to G_{diss} , α_{θ} is introduced to be the fraction of U_C that can be additionally stored at the carbide/matrix interface relatively to that in the matrix. Also, since the region adjacent to a crack is that of interest, a stress concentration factor $(\alpha_{\tau_{xz}})$ is introduced and defined as the ratio between τ_{xz} and τ_{xz0} . The stress concentration will also introduce higher strain, which is considered by multiplying α_{γ} . Then,

$$G_{diss} = \alpha_{\theta} \alpha_{\tau_{xz}} \alpha_{\gamma} U_C V_m N = 0.5 \alpha_{\theta} \alpha_{\tau_{xz}} \alpha_{\gamma} p_0 \Delta \gamma_C V_m N, \qquad (6.3)$$

where V_m is molar volume and N is the number of cycles.

In the microscopic approach, the increase of the matrix/carbide interfacial area due to carbide deformation is estimated in the system shown in Fig. 6.2. It is assumed that the strain can be accumulated via continuous deformation of carbide, which is based on the heavy deformation of residual carbide observed in Fig. 6.3; temper carbide particles are regarded to show the same type of behaviour. In the model, a carbide particle is assumed to be take a cubic shape, the side length of which being $2r_{\theta}$ with one side pair is strained by deformation. The corresponding increase in the interfacial area after N $(\Delta A_{\alpha\theta})$ is given as

$$\Delta A_{\alpha\theta} = 2 \times (2r_{\theta})^2 \left(\sqrt{1 + (\Delta \gamma_N)^2} - 1 \right), \tag{6.4}$$



Figure 6.2: A schematic system showing the carbide deformation after N.

where $\Delta \gamma_N$ is the accumulated strain over N, which can be expressed as $\Delta \gamma_N = \alpha_\gamma \Delta \gamma_C N$. This provides the additional Gibbs free energy by capillary to be expressed as,

$$G_{\Delta A_{\alpha\theta}} = \frac{V_m}{(2r_\theta)^3} \Delta A_{\alpha\theta} \gamma_{\alpha\theta} N = \frac{\gamma_{\alpha\theta} V_m}{r_\theta} \left(\sqrt{1 + (\alpha_\gamma \Delta \gamma_C N)^2} - 1 \right).$$
(6.5)

The capillary effect at N = 0 due to small carbide size has to be added to provide the driving force for carbide dissolution. Therefore,

$$G_{diss} = \frac{V_m}{(2r_\theta)^3} 6(2r_\theta)^2 \gamma_{\alpha\theta} + G_{\Delta A_{\alpha\theta}} = \frac{\gamma_{\alpha\theta} V_m}{r_\theta} \left(\sqrt{1 + (\alpha_\gamma \Delta \gamma_C N)^2} + 2 \right).$$
(6.6)

6.2 Carbide dissolution – equilibrium calculation

6.2.1 Calculation conditions

With G_{diss} calculated by Eqs. 6.3 and 6.6, the carbide Gibbs free energy is increased and the equilibrium is re-evaluated to obtain an updated fraction of carbide depending on N. In this way, carbide dissolution can be studied under the assumption that carbide dissolves immediately when it feels G_{diss} ; i.e. the time for the carbide to reach equilibrium is assumed to be negligible.

In Eq. 6.3, we take $\alpha_{\tau_{xz}} = 1.5$ according to Fig. 5.13b, p_0 and $\Delta \gamma_C$ are given as 3 GPa and 3.5×10^{-4} , respectively according to Fig. 3.36. From Fig. 3.36, it is seen that the



Figure 6.3: (a) An optical micrograph of the white-etching area studied in Section 5.2. (b) A secondary electron micrograph showing heavy carbide deformation observed in the region marked in (a).

dependence of $\Delta \gamma_C$ on the stress becomes weaker with larger stress. Therefore, α_{γ} would be smaller than $\alpha_{\tau_{xz}}$ and here, we take $\alpha_{\gamma} = 1.25$. V_m is calculated with the cementite lattice information given in Table 4.1 and α_{θ} is assumed to be 0.1 implying that 10% more deformation is stored at the carbide/matrix interface than in the matrix. In Eq. 6.6, V_m , α_{γ} , and $\Delta \gamma_C$ are taken the same as in Eq. 6.3 and $\gamma_{\alpha\theta}$ was approximated to be 0.391 J/m² [15], which was obtained by comparing the experimental work with the simulation results on the precipitation kinetics.

The equilibrium was calculated at 50 °C assuming paraequilibrium by MatCalc version 5.50 (release 1.007) with the mc_fe version 2.000 thermodynamic database. Since the database of transition carbides is unavailable, that of cementite was used.

Four popular bearing steel compositions (Table 2.1) were adopted and only temper carbides were considered during calculations. Since martensitic bearing steels always contain residual carbides, the matrix composition for precipitation of temper carbide is different from the alloy composition. To exclude the residual cementite which constitutes approximately 4 vol% [126], the temperature of austenite+cementite phase field showing 4 vol% cementite was determined for each alloy and the corresponding austenite composition
Table 6.1: The alloy compositions (all in wt%) after excluding 4 vol% residual cementite, which is the austenite composition at the given temperature where 4 vol% cementite exists [125]. Fe content is balanced accordingly.

	0.					
	Temperature / °C	С	Cr	Mn	Si	Mo
100Cr6	809.0	0.80	1.12	0.34	-	-
100CrMo7-3	809.0	0.75	1.35	0.68	-	0.32
100CrMnMoSi8-4-6	820.5	0.75	1.50	0.97	0.52	0.50
$100 \mathrm{CrMo7}$	802.5	0.75	1.30	0.34	-	0.18

Table 6.2: Precipitation driving force $(\Delta G_{m\theta})$ obtained by MatCalc [125]. 100Cr6 100CrMo7-3 100CrMnMoSi8-4-6 100CrMo7

	100Cr6	100CrM07-3	100CrMn10518-4-6	IUUCrivioi
$\Delta G_{m\theta} / \text{kJ mol}^{-1}$	-14.62	-14.12	-13.90	-14.30

at that temperature was taken for the calculations as shown in Table 6.1.

6.2.2 Results

Calculation results for the different alloy compositions are shown in Fig. 6.4. Note that the abscissa scale is different for each plot although the dissolution order of the alloys is the same: 100CrMnMoSi8-4-6, 100CrMo7-3, 100CrMo7, and 100Cr6. Faster dissolution is predicted for the macroscopic model than the microscopic model. From the experiments [35], dissolution was observed at $\sim 7 \times 10^4$ cycles with $p_0 = 3.53$ GPa which lies between what is predicted here by the two models. The discrepancy may come from the value of α_{θ} , α_{γ} in Eqs. 6.3 and 6.6, or from ignoring the time necessary for C to diffuse out of the carbide. It is ambiguous at this stage to determine which is a better approach. However, the relative effects for the different factors can be well explained. The influence of the composition is likely to come from the different carbide stability which can be evaluated by the precipitation driving force ($\Delta G_{m\theta}$) given in Table 6.2. Carbides with large $|\Delta G_{m\theta}|$ are more stable and less susceptible to dissolution. However, note that these results are based on the assumption of the same $\Delta \gamma_C$ for all compositions.

The influence of $\Delta \gamma_C$ as well as r_{θ} can be directly assessed from Eqs. 6.3 and 6.6. In both equations, the N necessary to reach the certain G_{diss} is smaller for larger $\Delta \gamma_C$ which



Figure 6.4: Calculated equilibrium weight fraction of cementite with respect to the number of cycles (N) with G_{diss} by (a) macroscopic (Eq. 6.3) and (b) microscopic (Eq. 6.6) aspects.

can be varied not only by the alloy composition but also by the heat treatment. Also, smaller r_{θ} would show the same effect via Eq. 6.6.

6.2.3 Abrupt dissolution

Although G_{diss} evolves gradually with respect to N in both approaches, the equilibrium carbide fraction seems stable over some N and suddenly drops (Fig. 6.4). G_{diss} at the moment is similar to $|\Delta G_{m\theta}|$ in Table 6.2. It is suggested that although G_{diss} steadily accumulates throughout deformation, it is only when G_{diss} exceeds $\sim |\Delta G_{m\theta}|$ that carbides suddenly dissolve. This is consistent with the sudden detection of the WEA during the bearing operation and the sharp WEA/matrix boundary shown in Figs. 5.15 and 5.20. Despite the gradual concentration of the stress adjacent to the crack (Fig. 5.10), the boundary is set by $|\Delta G_{m\theta}|$ determining whether the WEA is formed.

Abrupt dissolution takes place because the Gibbs free energy of ferrite is nearly proportional to C content when this is larger than $\sim 10^{-10}$ at% (Fig. 6.5). With the tangent construction, cementite fraction is given by the lever rule and C content in the matrix is determined by the point where the tangent line meets ferrite Gibbs free energy curve. In Fig. 6.5b, it is seen clearly that G_{diss} would not show much effect on both cementite fraction and matrix C content until the Gibbs free energy of cementite gets near to that of the ferrite energy curve, which is the point when G_{diss} becomes similar to $|\Delta G_{m\theta}|$. Once it reaches near to $|\Delta G_{m\theta}|$, the carbide amount is expected to decrease dramatically.

6.3 Carbide dissolution – diffusion controlled kinetics calculation

The calculation conducted in the previous section assumes that the system proceeds to the equilibrium state immediately when G_{diss} is provided. However, carbide dissolution also demands C diffusion away from the carbides. If the diffusion takes longer time than that for G_{diss} accumulation, carbide dissolution will be controlled by diffusion. The aim of the



Figure 6.5: (a) Gibbs energy curves of ferrite and cementite with respect to C content in at%. Only the increasing part of the ferrite Gibbs free energy is shown on the plot, since its minimum point lies below 10^{-10} at%. Note that cementite is a stoichiometric compound whose C content is 25 at%, thus showing a very narrow curve. (b) Tangent constructions (dash-dotted lines) for finding the equilibrium. Overall C content is also shown with the dotted line.

calculation in this section is to study how this process is influenced by alloy compositions and microstructure by MatCalc, which is well known for its competence in precipitation kinetics calculations [127–129].

6.3.1 MatCalc

The precipitation kinetics calculation in MatCalc is based on numerical Kampmann-Wagner model [130]. Precipitate particles with identical size and composition belong to a class, and kinetics are assessed separately by the class during the calculation. The precipitation simulation process is illustrated in Fig. 6.6. For each precipitate phase, the nucleation rate is assessed and new precipitate classes may be introduced for every time increment. For existing classes, growth of the precipitate particles are evaluated and updated. The precipitate class showing smaller particle size than the minimum nucleus radius (taken to be 0.35 nm in this study) is removed. This procedure is repeated during the simulation.

The nucleation rate $(J_{nucl}(t))$ is expressed according to the classical nucleation theory



Figure 6.6: Time integration of the evolution equations as implemented in MatCalc (adopted from [130]).

as [130, 131],

$$J_{nucl}(t) = Z\beta^* N_0 \exp\left(-\frac{G_c}{k_b T}\right) \exp\left(-\frac{\tau_{inc}}{t}\right),\tag{6.7}$$

where N_0 is the total number of the possible nucleation sites, G_c is the energy barrier against nucleation, k_b is the Boltzmann constant, T is temperature, and t is time. Z is the Zeldovich factor incorporating the nucleus instability due to thermal vibrations and it is given by

$$Z = \sqrt{-\frac{1}{2\pi k_b T}} \frac{\partial^2 \Delta G_{nucl}}{\partial n_{nucl}^2},\tag{6.8}$$

where ΔG_{nucl} is the Gibbs free energy for nucleation, and n_{nucl} is the number of atoms in the nucleus. β^* is the atomic attachment rate and for a multi-component system, it is expressed as [130],

$$\beta^* = \frac{4\pi r_c}{d_{latt}^4 V_m} \left(\sum_i \frac{\left(C_i^k - C_i^\alpha\right)^2}{C_i^\alpha D_i^\alpha} \right)^{-1},\tag{6.9}$$

where d_{latt} is the lattice spacing, V_m is the molar volume, C_i^{α} and C_i^k are the concentration

per unit volume of component *i* in the matrix and in precipitate *k*, and D_i^{α} is the diffusion coefficient of component *i* in the matrix. τ_{inc} is the incubation time expressed as [130, 131]:

$$\tau_{inc} = \frac{1}{2\beta^* Z^2}.$$
 (6.10)

For evaluating the growth of precipitate particles, the Svoboda-Fischer-Fratzl-Kozeschnik model based on the thermodynamic extremum principle [132, 133] is adopted. In this scheme, the total Gibbs free energy of the system (G_{sys}) can be expressed in terms of independent state parameters (q_i) under constant temperature and pressure in a closed system. Then, the rate of G_{sys} dissipation (Q) becomes a function of q_i and $\dot{q}_i = \partial q_i / \partial t$. The dissipative processes considered here are: (i) interface migration determined by its mobility, (ii) diffusion in the precipitate and in the matrix, which are controlled by the diffusivity in the corresponding regions. If Q is the positive definite quadratic form of \dot{q}_i , then

$$\frac{\partial G_{sys}}{\partial q_i} = -\frac{1}{2} \frac{\partial Q}{\partial \dot{q}_i}.$$
(6.11)

Since the radius of the precipitate k (r_k) and C_i^k are q_i in the system, the equation is solved to obtain them.

Other aspects should be also considered when employing MatCalc. Although many different kinds of nucleation sites can be selected, they do not vary in activation energies but in multiplicity instead. Moreover, there is no thermodynamic database for transition carbides; therefore, cementite (other than ϵ) was chosen. In order to consider transition carbide effects, a shape factor and a misfit strain were introduced and varied according to the carbide particle size; these relationships were determined from the experimental data during precipitation in a 100Cr6 martensitic steel [15]. The linear misfit parameter was $0.0013r_{\theta,m}^2 - 0.0137r_{\theta,m} + 0.0635$ ($r_{\theta,m}$ in nm), and the shape factor was adjusted to $3 \operatorname{erf} \left\{ \frac{\sqrt{\pi}}{3} (r_{\theta,m} - 3.5) \right\} + 4$ ($r_{\theta,m}$ in nm), where the error function was used to incorporate an asymptotical limit for large precipitates [125]. During kinetics calculations,

Table 6.3: Ferrite/cementite interfacial energy values from the literature and calculation by generalised nearest-neighbour broken-bond (GNNBB) model (adopted from [125]). The model considered the full composition data and temperature given in the literature although only C content is mentioned in the table.

Temperature / °C	C content / wt $\%$	Reference	Reported / J m ^{-2}	By GNNBB / J m ^{-2}
630–690	0.33	[135]	0.248 – 0.417	0.214 - 0.218
700	0.15	[136]	$0.520{\pm}0.130$	0.183
588	0.34, 0.68	[137]	0.56	0.193 – 0.199
727	0.80, 0.81	[138]	$0.700 {\pm} 0.300$	0.189 – 0.191
645 - 710	0.80	[139]	$\sim 0.73 - 1.1$	0.173 – 0.180
550-720	0.72,0.93,0.61	[140]	$\sim 0.85 - 1.36$	0.167 – 0.284

paraequilibrium condition was assumed, where the same site fraction of substitutional alloying elements are given to the matrix and cementite. Another important aspect is the matrix/carbide interfacial energy. Although MatCalc employs the automatically calculated interfacial energy by the generalised nearest-neighbour broken-bond model [134], the resulting value for the ferrite/cementite interface was rather low compared with the ones from the literature as seen in Table 6.3. Therefore, the interfacial energy was approximated to be 0.391 J/m^2 which well reproduces precipitation kinetics of carbides [15]. Moreover, diffusion enhancement at dislocations and grain boundaries are set to factors of 10^3 and 10^4 , respectively, as suggested by MatCalc. Besides, other factors affecting the nucleation kinetics such as pre-exponential factor for nucleation rate and the incubation time constant are kept as unity during the calculations.

6.3.2 Calculation procedures

The calculation is composed of two parts as shown by the flow diagram in Fig. 6.7. Before the actual calculation for studying dissolution kinetics in Simulation II, appropriate initial precipitate distributions have to be tailored by Simulation I. As in the previous section, the matrix compositions in Table 6.1 were adopted to exclude 4 vol% residual cementite in the system. For each composition, the aimed microstructure was the matrix with cementite of volume fraction, $V_{f\theta} = 0.05$, 0.10 and the mean particle radius, $r_{\theta,m} = 5$, 10, 20, 50, 100 nm. Note that the cementite volume fraction represents that of temper carbide in the alloy. These microstructures can be obtained by controlling the matrix/cementite interfacial energy ($\gamma_{\alpha\theta}$), and the precipitation simulation time (t_{pcpt}). Precipitation can be suppressed by increasing $\gamma_{\alpha\theta}$, resulting in larger $r_{\theta,m}$ with smaller $V_{f\theta}$. Since Simulation I was only done for designing the appropriate initial microstructure, $\gamma_{\alpha\theta}$ and t_{pcpt} , as well as the calculation process do not have any physical meanings.

Precipitation calculation of Simulation I was done with 500 precipitate classes under the paraequilibrium assumption. The grain size of the matrix was set as $0.2 \times 1 \ \mu m^2$, in agreement with the usual martensitic plate size, and the nucleation sites were set to be at dislocations, the density of which was 10^{15} m^{-2} , generating $4.40 \times 10^{24} \text{ m}^{-3}$ nucleation sites [130]. After obtaining the initial precipitate dissolution, the interfacial energy was set to 0.391 J/m^2 and G_{diss} was added to the cementite Gibbs free energy to destabilise carbide (Simulation II). During the simulation, $G_{diss} = 15 \text{ kJ/mol}$ is always used since this is similar to $|\Delta G_{m,\theta}|$, and at the same time, it was enough for all alloys to cause carbide dissolution. Constant G_{diss} assumes that no dissolution starts until certain G_{diss} is reached for all alloys, which is based on the abrupt dissolution as seen by the calculation in the previous section.

In both simulations, MatCalc version 5.44 (release 1.012) was used with mc_fe thermodynamic (version 1.009) and diffusion (version 0.006) databases¹ and the temperature was kept constant during the calculation; 160 °C for Simulation I, 50 °C for Simulation II.

6.3.3 Initial precipitate distribution

An advantage of Simulation I is that the precipitate distribution contains the particles with various size ranges rather than those with a single size. However, since calculations were done with different $\gamma_{\alpha\theta}$ and t_{pcpt} , these size distributions need to be compared before Simulation II.

¹In the process of performing precipitation calculations, different databases for MatCalc were adopted from Sections 6.2 and 4.2.4. However, this did not compromise the quality of the results.



Figure 6.7: Flow diagram showing two simulation parts for calculating diffusion controlled carbide dissolution kinetics [125]. ($\gamma_{\alpha\theta}$: matrix/cementite interfacial energy, t_{pcpt} : precipitation simulation time, $V_{f\theta}$: volume fraction of temper carbide, $r_{\theta,m}$: mean radius of cementite particles, $r_{\theta,max}$: maximum radius of cementite particles, N_{θ} : number density of cementite particles per unit volume)

A total of forty distribution combinations were obtained with four different alloy compositions, two precipitate phase fractions ($V_{f0} = 0.05, 0.10$), and five mean radii of precipitate ($r_{\theta,m} = 5$ -100 nm). To obtain these distributions, $\gamma_{\alpha\theta}$ was varied from 0.20 to 0.41 J m⁻² and t_{pcpt} from 0.22–156 s. As mentioned, these values do not display any important physical meaning, but are used only to obtain the boundary condition prior to dissolution. The discrepancy between the achieved $V_{f\theta}$, $r_{\theta,m}$ and their aimed values were within 1.8% and 2.2%, respectively.

The precipitate size distributions of 100Cr6 are shown in Fig. 6.8 for each aforementioned condition. Note that abscissa is $r_{\theta}/r_{\theta,m}$ to effectively compare the cases with different $r_{\theta,m}$. In this way, precipitate radius (r_{θ}) is seen to deviate from the average as it spreads from $r_{\theta}/r_{\theta,m} = 1$. Other compositions show similar distributions. When $r_{\theta,m} \ge 20$ nm, the precipitates showed similar size distributions; they gathered in the range of $0.74 \le r_{\theta}/r_{\theta,m} \le 1.14$ (Fig. 6.8b). However, larger scatter in size was observed among the cases with $r_{\theta,m} \le 10$ nm. Precipitates with $r_{\theta,m} = 5$ nm showed the concentrated distribution at $r_{\theta}/r_{\theta,m} = 1$, which may be the result of their underdevelopment due to small t_{pcpt} .

The precipitate distribution for examining the effects of each microstructural parameter are plotted in Fig. 6.9. The effect of composition was compared between a case $V_{f\theta} = 0.10, r_{\theta,m} = 10$ nm (Fig. 6.9a), that of the precipitate particle size in 100Cr6, $V_{f\theta} = 0.10$ (Fig. 6.9b), and that of the precipitate phase fraction in 100CrMo7-3, $r_{\theta,m} =$ 20 nm (Fig. 6.9c). Since the distributions were similar, the reasonable comparison could be made, except for the case of small particle size in Fig. 6.9b. However, the small particle size did not cause any anomalies in the results.

6.3.4 Carbide dissolution kinetics

Dissolution kinetics are investigated by the evolution of $V_{f\theta}$, $r_{\theta,m}$, $r_{\theta,max}$, $r_{\theta,min}$, N_{θ} , and the C content in the matrix. An example of the general dissolution behaviour is shown in Fig. 6.10 for 100Cr6 with initial $V_{f\theta} = 0.10$, $r_{\theta,m} = 10$ nm. $V_{f\theta}$ decreases monotonically



Figure 6.8: The precipitate distributions of 100Cr6 for (a) $r_{\theta,m} \leq 10$ nm, (b) $r_{\theta,m} \geq 20$ nm [125]. The legends are given in the $(V_{f0}, r_{\theta,m})$ pair.



Figure 6.9: The initial precipitate distributions for assessing the effects of (a) composition, (b) initial mean radius of the precipitate particles, and (c) precipitate volume fraction on the carbide dissolution kinetics [125]. The legends are given in $(V_{f0}, r_{\theta,m})$ pair for (b,c).



Figure 6.10: Carbide dissolution kinetics of 100Cr6 for the $V_{f0} = 0.10$, initial $r_{\theta,m} = 10$ nm in terms of (a) volume fraction of precipitate, (b) C content in the matrix, (c) radii and (d) number density of precipitate particles [125].

with time, which causes C concentration in the matrix to increase. On the other hand, $r_{\theta,max}$ is linearly reduced in the early dissolution stages until it reaches approximately half of the initial value when the particles dissolve completely as N_{θ} drops. These characteristics of dissolution kinetics are observed in all conditions. The servations observed in $r_{\theta,min}$ are the result of the emergence and disappearance of precipitate classes. As a result, $r_{\theta,m}$ starts to oscillate for low N_{θ} where only a small number of classes remains.

Assuming that dissolution is completed at $V_{f\theta} = 0.001$, the corresponding dissolution times are plotted for all compositions and sizes in Fig. 6.11. Although comparison was aimed to assess the effect of the initial mean carbide particle radius, the dissolution times are also given according to the maximum radius, because it governs diffusion controlled

Table 6.4: Diffusion coefficient of C in the matrix (D_C^{α}) provided by MatCalc [125].

	$100 \mathrm{Cr}6$	100CrMo7-3	100CrMnMoSi8-4-6	$100 \mathrm{CrMo7}$
D_C^{α} / m ² s ⁻¹	4.63×10^{-20}	4.72×10^{-20}	5.25×10^{-20}	4.51×10^{-20}

transformation. The influence of both radii seem to be very similar showing that the dissolution is effectively delayed with larger precipitate particles. Diffusion controlled dissolution is obvious from the fact that the dissolution time varies proportionally to the initial $r_{\theta,m}^2$ and $r_{\theta,max}^2$. Assuming spherical precipitate particles, $V_{f\theta} \sim \frac{4}{3}\pi r_{\theta,m}^3 N_{\theta}$. Taking the half distance between the precipitate particles as the C diffusion distance (λ_C) during dissolution, this is proportional to $r_{\theta,m}$ since

$$\lambda_C = \frac{2}{\sqrt[3]{N_{\theta}}} = r_{\theta,m} \sqrt[3]{\frac{32\pi}{3V_{f\theta}}}.$$
(6.12)

For diffusion controlled processes, λ_C can be approximated as $\sqrt{D_C^{\alpha}t}$ where D_C^{α} is diffusion coefficient of C in the matrix and t is the time. Hence,

$$\lambda_C^2 = r_{\theta,m}^2 \sqrt[3]{\left(\frac{32\pi}{3V_{f\theta}}\right)^2} = D_C^{\alpha} t, \qquad (6.13)$$

showing that t is proportional to $r_{\theta,m}^2$; this confirms that dissolution kinetics calculated by MatCalc is controlled by the diffusion of C away from the carbide.

With the same size and volume fraction of precipitate, the effect of composition results in a difference in carbide stability, as shown in Section 6.2. Although D_C^{α} may affect the kinetics, it is not so different among the alloys as indicated in Table 6.4 and the order of the dissolution follows that of increasing $|\Delta G_{m\theta}|$ listed in Table 6.2.

The effect of the initial $V_{f\theta}$ on the dissolution kinetics is shown in Fig. 6.12. 100CrMo7-3 was chosen because it is the only case showing a very similar $r_{\theta,m}$ (= 20 nm) and $r_{\theta,max}$ (= 23 nm) between the different $V_{f\theta}$, so that the dependence on the initial $V_{f\theta}$ can be solely appreciated. It took 0.48 and 0.44 days for dissolution to complete for the initial $V_{f\theta}$ =0.05 and 0.10, respectively. With larger initial $V_{f\theta}$, the dissolution occurred faster and $V_{f\theta}$ becomes the same for both conditions after ~0.32 days, which suggests that more



Figure 6.11: The dissolution time for all compositions with $V_{f0} = 0.10$ according to (a) the square of initial mean radius $(r_{\theta,m}^2)$ and (b) that of the initial maximum radius $(r_{\theta,max}^2)$ of the precipitate particles [125].

developed precipitates are more susceptible to dissolution. This is mainly because the microstructure with more developed carbides contains less C in the matrix resulting in larger C concentration gradient during dissolution.

In order to assess the assumption that constant G_{diss} suddenly appears, G_{diss} was varied from 12 to 15 kJ/mol for 100Cr6 with the initial $V_{f\theta} = 0.10$, $r_{\theta,m} = 10$ nm and the result is shown in Figs. 6.13 and 6.14. As expected, a threshold value for G_{diss} over which dissolution was induced existed and it was similar to $|\Delta G_{m,\theta}|$. If G_{diss} was smaller than the threshold, carbide coarsening took place. Coarsening occurred because precipitation is not fully developed when G_{diss} was added to the system. Therefore, an increase in precipitate fraction would be preferred if insufficient G_{diss} is provided, which is related to the DER formation; however, since the calculation was done with the thermal diffusivity which is different from the dislocation assisted diffusivity in Section 4.1.2, and the time shown here does not represent that for the DER formation.

6.4 Discussion

6.4.1 Controlling the kinetics of carbide dissolution

In order to completely appraise the time for carbide dissolution, both processes, (i) deformation energy accumulation (Section 6.1), and (ii) C diffusion away from the carbide (Section 6.3), should be considered together. However, it is seen in Section 6.3 that the dissolution takes around 0.5 days to be completed for the case similar to the real bearing where $r_{m,\theta} \leq 20$ nm (Fig. 6.12). Since the rolling speed of the bearing is in the range of 200-800 Hz (Table 2.3), it corresponds to 8.64×10^6 – 3.46×10^7 cycles, which is much later than when WEA is observed to form ($\sim 7 \times 10^4$ cycles [35]). It is suggested this may be because diffusion enhancement is not properly taken into account. In Section 5.2, C pipe diffusion during the dissolution is suggested to occur through the boundaries of dislocation cells formed due to deformation. Therefore, the diffusion enhancement factor is likely to depend on the dislocation cell size and would vary with time during the deformation.



Figure 6.12: Carbide dissolution kinetics of 100CrMo7-3 for the initial $r_{\theta,m} = 20$ nm showing its dependence on the initial precipitate volume fraction in terms of (a) volume fraction of precipitate, (b) C content in the matrix, (c) mean (solid line) and maximum (dotted line) radius, and (d) number density of the precipitate particles [125].



Figure 6.13: Carbide dissolution kinetics of 100Cr6 with the initial $V_{f\theta} = 0.10$, and $r_{\theta,m} = 10$ nm showing its dependence on magnitude of the driving force in terms of (a) volume fraction of precipitate, (b) C content in the matrix, (c) mean (solid line) and maximum (dotted line) radius, and (d) number density of the precipitate particles [125].



Figure 6.14: The same results shown in Fig. 6.13 with the different abscissa scale [125].



Figure 6.15: The relationships among parameters which can influence the kinetics of carbide dissolution. (a) is from Eqs. 6.3 and 6.6, (b) is from Sections 6.2 and 6.3, and (c) is from Section 6.3.

However, both this factor and the dislocation density are regarded to be constant during the simulation, which may have resulted in slow kinetics.

Still, the comparison made in the study in Section 6.3 is valid since the diffusion enhancement factor would not be greatly influenced by the factors considered here (composition, the size and amount of precipitate particles). Moreover, with the results from Sections 6.1 and 6.2, the resistance to carbide dissolution can be controlled as shown in Fig. 6.15. Composition will affect $|\Delta G_{m\theta}|$ as well as $\Delta \gamma_C$, and heat-treatment can control the microstructure $(V_{f\theta}, r_{\theta})$. However, microstructure should be carefully tailored since it also affects $\Delta \gamma_C$.

6.4.2 From white-etching area to material failure

Although a WEA is formed as a result of carbide dissolution, it cannot directly lead to failure of the components. Adjacent to a WEA, usually a microcrack is found, which has to grow to the surface to cause spalling. The growth rate might be slow until the crack reaches a critical length (l_c) , but after exceeding this, it would propagate rapidly with applied stress. This critical length is set by mode II fracture toughness, K_{IIc} , which is reported to be 22.71–22.92 MPa \sqrt{m} for a 1C-1.4Cr (in wt%) steel [141]. Note that mode II is selected instead of mode I since, the normal stress close to the crack is always in the

direction of closing up the crack. From the fracture mechanics [142],

$$K_{IIc} = \tau \sqrt{\pi \frac{l_c}{2}},\tag{6.14}$$

where τ is the responsible shear stress for the crack propagation. Considering the two dimensional stress state during bearing operation (Section 2.2, Table 2.2) and the stress concentration (Section 5.1), $\tau = \alpha_{\tau_{xz}} \tau_{xz} = 1.5 \times 0.25 p_0$ leading to $l_c \sim 260 \ \mu\text{m}$ with $p_0 = 3$ GPa. In order to obtain the time for failure from WEA, the crack growth rate is necessary and it is suggested to be related to WEA formation kinetics due to cooperative growth between the crack and the WEA [34]. However, the growth rate is not yet available and needs further study.

6.5 Summary

The driving force for the carbide dissolution was estimated either by cyclic stress-strain behaviour or by the interfacial area increase due to the carbide deformation. The value was used to calculate the thermodynamic equilibrium and a sharp decrease in carbide amount was detected, implying the carbide dissolution takes place. Moreover, diffusion controlled dissolution was simulated with a constant driving force to find the effects of composition as well as the size and amount of the carbides. It was found that the carbide dissolution kinetics can be controlled by changing (i) deformation energy accumulation rate, (ii) carbide precipitation driving force, and (iii) C diffusion rate with the alloy composition and the heat treatment. Although the WEA and the failure of the component can be related, the crack (WEA) growth rate is necessary which is yet to be established.

Chapter 7

General conclusions and suggested future work

Rolling contact fatigue (RCF) of bearing steels is manifested by subsurface microstructural damage, often manifested in the form of dark-etching regions (DERs) and white-etching areas (WEAs). The mechanisms of such damage forms have been investigated from a metallurgical point of view. This study is a first attempt to enhance bearing performance by looking into the microstructure at scales going down to the atomic level.

The stress state during bearing operation is special in that it keeps evolving depending on the position under the surface and time, which makes it impossible to directly measure strain during bearing operation. Although Hertzian elastic contact theory can explain the principal shear stress responsible for plastic deformation, reaching its maximum at the subsurface, and resulting in the alteration of the microstructure at such region, cyclic plasticity needs also to be considered as well. During plastic deformation, residual stresses develop in the material and change both the magnitude and orientation of the principal shear stress. This causes a stress state in the material different from the one predicted by Hertzian elastic contact theory.

It has been examined whether simpler test methods can be implemented to replace a full-scale bearing test. The first method being the ball-on-rod test, which is one of the bearing element tests that reproduce repetitive rolling cycles with simple sample geometry. Microcracks were observed adjacent to inclusions after such test and these were sometimes decorated by WEAs. Also, the matrix hardness increases at the subsurface. Both the frequency of the inclusions decorated with the microcracks and the degree of hardness increase depend on contact pressure. However, the microcrack orientation was inconsistent with previously reported values, and DERs did not form. The reason is suggested to be related to different residual stresses developing during testing, which depend on the test conditions and the contact geometry.

A newly proposed method to examine microstructure evolution during RCF is the repetitive push test, which employs a uniaxial fatigue tester. Although rolling contact is not reproduced in the test, the plastic strain due to the rolling cycles can be obtained. The measured strain is employed in this thesis for models describing microstructural alteration. The degree of hardness increase was observed to be similar to the one displayed by ball-on-rod testing, suggesting that the developed residual stresses are comparable. In this respect, the suggested future work is:

- Residual stress measurement for different test methods employing X-ray diffractometry. This will enable proper comparison of the stress state with actual bearing operation.
- Testing the samples without retained austenite would rule out the possible hardness and ductility increase due to austenite to martensite transformation. This may allow to assess the small changes in hardness more easily.

A model for carbide coarsening in DERs is introduced. A driving force for carbide coarsening is already present due to the matrix supersaturation with C, stemming from incomplete tempering. Without deformation, carbide coarsening is suppressed due to C segregation to crystal defects at low temperature. During deformation, dislocations with C atmospheres glide around carbide particles and transfer C. As a result, carbides coarsen and the matrix gets depleted of C, causing a hardness to decrease. This process is quantitatively described as a function of number of cycles, and the results agree with experimental data. According to the proposed model, DER formation kinetics can be controlled via tailoring test conditions such as operation speed, temperature, contact pressure as well as the microstructure, e.g. carbide size and fraction. The most important factor is plastic strain which strongly depends upon microstructure. Further study can be done on:

• Failure due to DERs formation. DERs have different mechanical properties compared with the surrounding matrix. Although failure caused by DER has never been reported, the associated mechanism would be scientifically and technologically interesting.

WEAs are different from DERs in that they are localised; they appear near stress raisers such as inclusions and cracks, and show distinctive boundaries with the matrix. The stress concentration at the cracks is estimated to be ~ 1.5 times higher than the case without crack. The microstructure inside a WEA is found to be nanocrystalline; the nanocrystalline boundaries are decorated with C and Si atoms. Nano-sized nonstoichiometric carbide particles are observed suggesting carbide dissolution in the region. Based on these observations, it is suggested that a stress concentration adjacent to cracks causes larger deformation accumulation in such a region than in the matrix, which acts as a driving force of the carbide dissolution. Suggested future research may focus on:

• Rolling simulations employing the finite element method involving a pressure moving over the contact surface. This would allow to assess space and time stress concentration variations caused by a crack. Also, by introducing more than one stress cycle, the residual stress development and its effect on stress raisers could be predicted.

The carbide dissolution driving force can be estimated by (i) macroscopic deformation behaviour of the material adopting the results obtained from stress concentration calculation and repetitive push test, and (ii) the capillary effect due to interfacial area increase from carbide deformation. Then, the corresponding driving force can be added to the carbide Gibbs free energy to perform an equilibrium calculation, which results in abrupt carbide dissolution. Although the order of magnitude of the number of cycles when dissolution happens to agree with reported values, this approach neglects the contribution of C diffusion during dissolution. Therefore, diffusion controlled dissolution was simulated and its dependence on microstructure and test conditions are appreciated. These two processes, deformation energy accumulation and C diffusion away from carbide, need to be summed up to completely explain carbide dissolution kinetics. The factors controlling the kinetics are carbide stability depending on alloy composition, morphology of carbide including its volume fraction and particle size, and the experienced strain per cycle. Related future research may focus on:

- Pipe diffusion behaviour of C atoms in nanocrystalline structures, which causes diffusion enhancement. The interaction of C with Si needs to be considered further, since Si influences C activity.
- Development of dislocation cell structures including their size and misorientation. This may affect the diffusion of C and Si via pipe diffusion. Moreover, the high hardness in WEAs can be predicted by C content in the matrix after the carbide dissolution with the dissolution model suggested in this work.

Moreover, it is suggested that a crack and the accompanying WEA may cooperatively grow. After the crack reaches the size defined by mode II fracture toughness, it would propagate fast and result in failure. Therefore,

• The cooperative growth rate of the crack with WEA needs to be further studied in order to relate WEAs to bearing life.

Although experimental data support the suggested models, the models only separately explain each form of microstructural damage and it is not yet correlated. However, some evidence shown in the study suggests that some forms of microstructural damage sometimes interact and even suppress the formation of others. Therefore, • Studying the interaction between different forms of the damage, and incorporating all models simutaneously would be the most important task in future.

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