Manuscript No.: TRIBINT-D-18-01076

Title: Debris Development in Fretting Contacts - Debris Particles and Debris Beds

Article Type: SI: 2018 Leeds-Lyon Symposium

Corresponding Author: Mr. Adam Matthew Kirk

All Authors: Adam Matthew Kirk; Philip H Shipway; Wei Sun; Chris J Bennett

Submit Date: Jul 04, 2018

Response to reviewers' comments

We would like to thank the reviewers and the editor for their input on the paper, and for the opportunity to make minor revisions to the text. We appreciate both reviewers' positive comments regarding the overall content of the paper, and have implemented the sole change recommended which is summarised below:

We are familiar with the work of Arnaud and Fouvry et al., whose recent work on debris the reviewer has identified as being relevant to the present work. Of the papers we have identified by these authors there is one paper in particular that we have deemed to be of particular relevance, and have therefore made reference to the contribution of this work to the understanding of contact oxygenation in the discussion section of the revised manuscript. Reference has also been made to the work of Warmuth et al. which is cited in the aforementioned paper and that we consider to also be highly relevant to the subject of contact oxygenation and the formation of metallic wear debris.

The original text from the reviewer is in standard font, *whilst our responses are in italics.* Revisions to the manuscript are highlighted.

Reviewers' comments

Reviewer #1: Very nice work and paper.

One comment - Please refer to and discuss, as appropriate, the recent work on debris by Arnaud and Fouvry et al.

Response : *We are grateful to the reviewer for their recommendation - we are familiar with the work of Arnaud and Fouvry et al., and have identified one paper as being of particular relevance to the present work. As such, we have made a note to this effect in the discussion section of the revised manuscript.*

"An increase in metal-metal contact as a result of increased contact conformity in fretting of steels was observed by Warmuth et al. [26], with the authors attributing the effect to restricted oxygen access to the contact inhibiting the formation of oxide debris, with recent work by Fouvry et al. [27] noting a similar phenomenon in fretting of titanium contacts, relating the effect not only to contact size but other sliding conditions affecting contact oxygenation." (Section 4, page 8)

Reviewer #2: The present study investigates the role of fretting contact parameters on the formation of third body wear debris and subsequent surface transformation / debris particle beds for steel-steel contacts. The conclusions presented are well thought out and supported by the data which has

extensively looked at the debris both in third body form and sintered to the surface within the fretting scar. Chemical changes in the debris composition and particle morphology as a result of displacement amplitude and energy dissipated offer interesting insight into the performance and fatigue of such contacts.

It is my recommendation the article is published as is.

DEBRIS DEVELOPMENT IN FRETTING CONTACTS – DEBRIS PARTICLES AND DEBRIS BEDS

A.M. Kirk, P.H. Shipway, W. Sun, C.J. Bennett
Faculty of Engineering, The University of Nottingham, UK

ABSTRACT

In this study, the formation and destruction of compacted beds of oxidized debris particles are investigated. Fretting tests of steel specimens were conducted, employing a cylinder-on-flat geometry with displacement amplitude being varied. The debris was examined, both in the form of the debris beds and in the form of loose debris; together, these were characterized to better understand the mechanisms of debris bed formation and development throughout the fretting process. XRD was employed to determine the phase makeup of the debris, with SEM imaging and particle size analysis employed to understand the evolution of the debris from nanoparticles into agglomerates and then into sintered bed structures.

1 Introduction

Fretting is defined as small amplitude relative oscillatory motion between two contacting bodies that gives rise to both wear and fatigue in a wide range of industrial contexts, the understanding and mitigation of which have been the subject of extensive study. It is well known that fretting conditions can result in substantial subsurface damage [1, 2], and in contrast to sliding wear, the small relative displacements that characterise fretting have a significant impact on the development of wear through the behaviour of wear debris in the contact itself; specifically, a proportion of wear debris is entrapped in the contact and thus modifies the nature of the contact, whilst the remaining debris is ejected from the contact and thus ceases to play a direct role in the subsequent wear process [3].

The complex physical processes occurring in fretting wear result in behaviour being dependent on a very large number of parameters [4], with complex interdependencies rendering development of all-encompassing predictive models out of reach in the current state of fretting research. However, many of these parameters influence wear to some degree through their impact on the behaviour of the debris in the fretting contact, and in light of this, a greater emphasis has been placed on the role of third bodies in wear in the past few decades [5]. Most notably, Godet and co-workers [6] developed the “third body approach” where the fretting debris was termed the third body, with the approach being based upon principles of lubrication theory (such as velocity accommodation and load carrying) being applied to the third bodies.

Particles detached from the two surfaces which make up the fretting pair (i.e. the first bodies) are subject to extensive deformation and elevated temperatures in the contact [7-9]. Any nascent metallic surfaces formed (either by detachment of metallic wear debris particles, or by the removal of oxides from the first bodies) are highly reactive and will be subject to rapid oxidation [3, 10]. This is a continuous process, with the wear debris particles themselves becoming active in the damage process; the oxide particles can act as abrasives, increasing wear damage to the first bodies, but can also adhere to the first bodies to form compacted oxide

1 films at the interface [11] which can deform and shear such as to accommodate the relative velocity between
2 the two first bodies, and thus reduce the imposed tractional load on the first body surfaces, thereby protecting
3 them from wear [5, 12].
4

5 Kato and Komai [13] artificially supplied nanometre-scale particles of oxides of several different metals to
6 rubbing surfaces in sliding wear conditions and found that the sliding distance to transition from severe to mild
7 wear (associated with the formation of a stable oxide bed) decreased as the size of supplied particles was
8 reduced, suggesting a strong influence of particle size on debris bed formation. The formation of stable beds
9 was attributed to sintering of the oxide particles, a process which typically takes place at temperatures half
10 that of the melting temperature of the material [14], although under tribological conditions is found to occur
11 at much lower temperatures in a phenomenon known as tribo-sintering [15]. Tribo-sintering has also been
12 found to be strongly dependent upon oxide type; Kato and Komai [13] found that less diffusive oxides did not
13 form a stable layer under the conditions studied, and more recently Viat et al. [16] linked the formation of a
14 stable debris layer to the auto-diffusion coefficients of the alloying elements present in the first bodies.
15
16

17 Formation of a stable load bearing debris bed depends not only on the rate at which the bed grows due to
18 debris generation and consolidation, but also upon the rate at which such beds are broken down and ejected
19 from the contact. Operating conditions conducive to formation of strong bonds between debris particles (and
20 thus to formation of a stable debris bed) can result in the formation of a protective 'glaze' layer which sharply
21 reduces the rate of further wear experienced by the first bodies [17, 18]. This glaze is formed by tribo-sintering
22 [13], and, as such, the factors which affect tribo-sintering are therefore of interest in terms of understanding
23 the development of damage in a fretting contact. Pearson et al. [18] found that while glaze formation occurs
24 more readily at elevated operating temperatures for a self-mated steel fretting contact, there was evidence of
25 glaze formation at temperatures as low as 85 °C under certain conditions; however, it was also shown that the
26 temperature at which glaze formation first occurred was elevated by an increase in the fretting displacement
27 (all other factors being equal) [18].
28
29

30 There have been many attempts over the past decades to characterise loose debris generated in fretting tests
31 of steel specimens [3, 10], with the key characteristics being composition, size and to a lesser extent
32 morphology of the debris particles. It has been found by many researchers that the primary constituent of
33 debris from fretting steel contacts at ambient temperature is α -Fe₂O₃ with metallic iron also being reported in
34 some cases [3, 10], although the relative proportions of oxide and metal are not well established. Much less
35 commonly, there have been reports of other oxides of iron in fretting debris produced by the fretting of steel
36 pairs, such as FeO and Fe₃O₄, although evidence supporting these findings is relatively sparse [3, 10, 19]. It has
37 been noted that in a vacuum or inert gas atmosphere, fretting debris from steels is entirely metallic in
38 character and produced in much smaller quantities than that formed in air [20]. The size of fretting wear debris
39 is less well established in the literature than its composition; early estimates put the size of fundamental
40 debris particles at approximately 1 μ m in diameter [19], although this dimension has been quoted as being
41 nearer 0.01 μ m by some researchers [21], and several microns by others [22]. The wide range of estimates
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65

may be attributed in part to the diverse range of rigs, materials and conditions used employed in the test programmes where debris size was reported.

It is recognised that characterization of debris is critical to understanding of the fundamental processes of wear in fretting and accordingly, the current work seeks to investigate the evolution of fretting wear debris as a function of both fretting stroke and number of cycles by examining the nature of the debris, both as debris beds and as expelled particles.

2 Experimental procedure

2.1 Specimens and test conditions

Tests were performed on a high strength steel (BS S132), the chemical composition and mechanical properties of which are given in Tables 1 and 2, respectively. The heat treatment cycle used prior to machining to final dimensions is detailed by Mohd Tobi et al. [23].

Table 1. Composition of BS S132

C	Si	Mn	P	Cr	Mo	Ni	V	Fe
0.35-0.43	0.1-0.35	0.4-0.7	<0.007	<3.0-3.5	0.8-1.1	<0.3	0.15-0.25	Balance

Table 2. Mechanical properties of BS S132 [24]

σ_y / MPa	σ_{ut} / MPa	HV30	E / GPa	ν
1247	1697	485 $\pm$ 10	206.8	0.28

Specimens were machined to shape following heat treatment and were arranged in a cylinder-on-flat configuration, as shown in Figure 1, resulting in a line contact with a length of 10 mm perpendicular to the fretting direction. A schematic diagram of the rig is given in Figure 2. The flat specimen was mounted in a fixed lower specimen mounting block (LSMB) and the cylinder specimen mounted in a moving upper specimen mounting block (USMB), with relative oscillatory displacement between the two being generated at a fixed frequency by an electromagnetic vibrator (EMV). Constant normal load was applied to the upper specimen via a dead weight and a lever arm.

Figure 1. Diagram of specimens and their arrangement; $L = 10$ mm and $R = 6$ mm.

Figure 2. Schematic diagram of main components of the fretting rig

The relative applied displacement amplitude, Δ^* , between the LSMB and USMB was measured using a capacitance displacement sensor mounted to the LSMB and recorded throughout the tests. The lateral force,

Q, was measured throughout the tests using a piezoelectric load cell connected to the LSMB. Both sensors were calibrated both externally and in-situ and the test data sampled at 200 measurements per cycle to generate force-displacement traces (i.e. fretting loops) to determine slip amplitude and energy dissipated in fretting (using the method first proposed by Fouvry et al. [25]) and other relevant test data for later analysis.

Tests were conducted at a frequency of 20 Hz with a normal load of 450 N at applied displacement amplitudes of 25 μm and 100 μm for 10 000, 100 000 and 1 000 000 cycles (i.e. covering a range of two decades in fretting cycles). Before testing, specimens were thoroughly degreased using detergent, acetone and industrial methylated spirit. Upon completion of the test, the loose debris ejected was collected for later analysis.

2.2 Characterisation of fretting damage and wear debris

After testing, the specimens were ultrasonically cleaned for ten minutes in industrial methylated spirit to remove any loose debris; profilometry was then undertaken to evaluate the worn surfaces using an Alicona InfiniteFocusSL.

Scanning electron microscopy (SEM) was used for qualitative observations regarding the nature of wear scars and loose debris using a FEI Quanta600 MLA SEM. Secondary electron (SE) imaging was used to examine topographical features and back-scattered electron (BSE) imaging to distinguish between materials of different average atomic number, such as the base metal and oxide debris. Composition was confirmed qualitatively by energy dispersive X-ray spectroscopy (EDX). Loose debris was mounted using adhesive carbon tabs and carbon coated for SEM examination using the same instrument.

Wear debris ejected during tests was analysed by X-ray diffraction (XRD) using a Siemens D500 diffractometer with $\text{Cu-K}\alpha$ radiation to identify phases present in debris samples. Scans were undertaken using a 0.04° step size between 2θ positions of 20° and 120° , with a dwell time of 22 s per step. Samples were prepared using a solvent deposition method in which debris powder was suspended in IMS and pipetted onto a silicon wafer (used to minimise background from the sample holder). Phase fractions in the debris were estimated by performing Rietveld refinements using Topas V6 software, using the fundamental-parameters approach to X-ray line profile fitting. The calculated pattern was fitted to the experimental data by applying a spherical harmonics function to account for discrepancies attributed to complex preferred orientations in the sample.

Particle size data for wear debris were obtained using a Coulter LS230 light scattering particle analyser with a detection range from 375 nm to 2 mm in equivalent spherical diameter. Debris samples were suspended in water, with a sample for size analysis being typically 10 mg, several separate particle size samples were measured from a single debris sample to eliminate any potential bias arising from the small volumes required to obtain particle size data.

3 Results

Net wear volumes for the wear tests conducted in this study are shown in Figure 3 plotted against energy dissipated over their course; the wear rate (volume lost per unit energy dissipated) is clearly significantly higher at the higher applied displacement amplitude.

Figure 3. Net wear volume as a function of dissipated energy for the fretting tests conducted in this study; for each displacement amplitude, the data represent the three test durations employed.

3.1 Characterisation of ejected debris

3.1.1 X-ray diffraction

X-ray diffraction data, shown in Figure 4, were obtained for fretting debris collected from tests at both displacement amplitudes after 10^6 cycles, along with diffraction patterns calculated by performing Rietveld refinements on the experimental patterns as a means of estimating relative proportions of the phases in the samples. Distinct peaks at 45° , 65° and 82° align with the diffraction pattern of ferrite, while the further expected peaks at 98° and 116° are less clearly discernible.

The diffraction patterns of both samples indicate that the debris is made up primarily of $\alpha\text{-Fe}_2\text{O}_3$ (haematite) with a small fraction of $\alpha\text{-Fe}$ (ferrite); there was no evidence of other phases being present. Following fretting with a $25\text{ }\mu\text{m}$ applied displacement amplitude, the proportion of ferrite was found to be approximately 2.6 % with this fraction increasing to 5.6 % following fretting with the higher applied displacement amplitude of a $100\text{ }\mu\text{m}$. Although the data have been fitted taking into account preferred orientation of the samples, such orientation in the samples is likely to be complex (given the mechanism by which the debris is formed) and therefore difficult to account for comprehensively; accordingly, the proportions of phases obtained by Rietveld analysis should be considered as approximations.

Figure 4. Experimental and calculated X-ray ray diffraction patterns of debris collected after 10^6 cycles with an applied displacement amplitude of (a) $100\text{ }\mu\text{m}$; (b) $25\text{ }\mu\text{m}$. In each case, the differences between the measured and calculated data are shown.

3.1.2 Particle size analysis

The debris particle size distributions are presented in Figure 5 as histograms; the fractions in the various size ranges are averages of several measurements on different sample aliquots from the same debris sample and the error bars represent the range of the individual measurements. It can be seen that (with an applied

displacement amplitude of 100 μm) the debris particle size distribution does not change significantly with test duration (Figure 5(a)), indicating that steady state conditions are reached very early in the fretting process.

The particle size data obtained for the 10^6 cycle tests at the two displacement amplitudes examined (Figure 5(b)) exhibited a considerable difference in distribution. In both cases, the greatest proportion of debris from both tests fell in the 2 – 7.5 μm range; however, the debris from the test with a 100 μm applied displacement amplitude exhibited a broader distribution, with a greater fraction of particles in ranges both smaller and larger than this.

Figure 5. Particle size distributions of ejected debris (a) for a displacement amplitude of 100 μm at each of the test durations studied; (b) for both displacement amplitudes at 10^6 cycles.

3.1.3 Debris morphology

In order to observe debris morphology, SEM images of ejected debris samples were obtained. An SE image of a sample of debris ejected in the course of a 10^6 cycle test with an applied displacement amplitude of 100 μm (Figure 6(a)) shows a wide range of particle sizes present in the sample, with a small number of large particles being visible. Observing one such large particle (confirmed by EDX analysis to be iron oxide) at higher magnification (Figure 6(b)) confirms that the particle size is significantly greater than the displacement amplitude employed in the test from which it was generated. It is assumed that this is a thin piece of compacted debris, but its depth could not be confirmed from the SEM observations. A striking feature of the large particle in Figure 6(b) is the presence of very different surface textures; Figure 6(c) bears a resemblance to a vermicular structure typically exhibited during sintering processes, and appears much more porous than the smooth and glassy surface texture apparent in Figure 6(d).

Figure 6. SE SEM micrographs of ejected debris collected from a 10^6 cycle test with a displacement amplitude of 100 μm ; (a) overview; (b) higher magnification image of large particle seen in the centre of (a); (c) and (d) are higher magnification images of the surface of the particle shown in (b) as highlighted.

Examining some of the smaller particles in the same debris sample showed a great variation in the nature of the debris particles ejected during one test, although all appeared to consist of a multitude of sub-micron oxide crystallites either agglomerated or bonded to form a larger whole. The BSE images shown in Figure 7 highlight a flat piece of debris (confirmed as oxide by quantitative EDX analysis) consisting of smaller particles bonded to form a flat, coherent structure that appears to have been plastically deformed, suggesting that it is a detached piece of the debris bed that has subsequently been ejected from the contact. Debris particles similar in appearance to that highlighted in Figure 7(b) were also observed in the ejected debris collected after 10^4 cycles, indicating that such detachment and ejection of parts of the debris bed occurred from an early stage of fretting. Due to the samples consisting of debris collected upon completion of the fretting tests, it is

not possible in the current study to determine at what stage individual particles were ejected from the contact during longer cycle tests.

Figure 7. BSE SEM micrographs of debris ejected during a 10^6 cycle test with displacement amplitude of 100 μm : (a) low magnification; (b) higher magnification view of the surface of the right-hand larger particle seen in (a).

The similarity in the nature of ejected particles is evident in the SE images shown in Figure 8; here, images are presented of two particles of similar size found in the debris from tests with both 100 μm and 25 μm applied displacement amplitudes. In both cases, it can be seen that the larger particles (Figure 8 (a) and (c)) are made up of individual crystallites of the order of 0.1 μm in diameter (Figure 8 (b) and (d)). The sub-micron crystallites comprising the ejected debris particles observed were not observed in isolation in any of the samples examined by SEM, suggesting a strong tendency for such particles to agglomerate / sinter during the fretting process.

Figure 8. High magnification SE SEM micrographs of debris particles ejected during 10^6 cycle test with displacement amplitude of (a)-(b) 100 μm ; (c)-(d) 25 μm

3.2 Debris bed development

3.2.1 Plan view SEM

The wear scars developed on flat specimens fretted at both displacement amplitudes were imaged at various magnifications in plan view using BSE SEM (Figure 9). The contrast in BSE images results primarily from differences in average atomic number and allows for clear distinction between regions covered with a layer of oxide (darker grey) and exposed metal surfaces (lighter grey). Qualitative EDX was used to confirm the nature of the regions examined.

Considering Figure 9(a) and Figure 9(d) it is clear that an oxide debris bed forms and is adhered to the surface after a relatively short number of cycles, and that even after a hundredfold increase in the number of cycles, the bed still does not cover the entire surface, leaving metallic regions exposed. It can be seen that while wear scar width increases significantly (from approximately 1 mm after 10^4 cycles to almost 4 mm after 10^6 cycles), debris bed coverage remains incomplete, even after 10^6 cycles.

Higher magnification images of the specimen after 10^6 fretting cycles show an area of developed oxide bed on top of a more exposed metallic surface with a small degree of oxide coverage, evidently a remaining region of the debris bed formed during fretting and adhered to the substrate. The uneven edges of this oxide region suggest that pieces have broken away, exposing the metal surface beneath it. Figure 9(e) shows a crack across

1 a protruding region, suggesting a mechanism by which pieces of such a debris bed may become detached from
2 the surface. It should be noted that the ultrasonic cleaning of specimens after tests to remove loose debris
3 may have dislodged such pieces, and so it cannot be stated whether such regions were ever exposed to such
4 an extent during fretting.
5

6
7 Also visible from the BSE images in Figure 9 are bright regions contained within the oxide debris bed layers. It
8 is proposed that this may be the metallic fraction observed in the debris via XRD analysis (see Figure 4). It is
9 not currently clear how deep these metallic regions extend beneath the surface of the debris bed, that is to
10 say whether the bright regions are caused by a very thin layer of metal smeared across the surface between
11 the contacting bodies or whether the debris beds contain metal particles of several microns in depth.
12
13
14
15
16
17

18 **Figure 9. BSE SEM micrographs of flat specimens after fretting with an applied displacement amplitude of**
19 **100 μm for two test durations: (a) - (c) 10^4 fretting cycles; (d) – (f) 10^6 fretting cycles.**
20
21
22
23
24

25 4 Discussion

26
27 As previously identified, the loose debris and debris beds are shown to be made up of an iron-based oxide with
28 a small fraction of metallic particles. The presence of metal in the debris bed is visible in the bright specks
29 observed in BSE images of the beds in plan-view (Figure 9), suggesting that metal particles several microns in
30 diameter are present inside the contact and are incorporated into the third body layer. Analysis of the X-ray
31 diffraction data (Figure 4) demonstrates an increased proportion of metallic iron ($\alpha\text{-Fe}$) in ejected debris as the
32 displacement amplitude was increased under the fretting test conditions explored. The presence of metal in
33 fretting debris is commonly attributed to adhesive metal-metal contact between surfaces during wear, which
34 may result from a low rate of oxidation of the surface insufficient to prevent metal-metal contact by formation
35 of a separating third body layer, or detachment of parts of such a layer leaving metallic regions exposed. An
36 increase in metal-metal contact as a result of increased contact conformity in fretting of steels was observed
37 by Warmuth et al. [26], with the authors attributing the effect to restricted oxygen access to the contact
38 inhibiting the formation of oxide debris, with recent work by Fouvry et al. [27] noting a similar phenomenon in
39 fretting of titanium contacts, relating the effect not only to contact size but other sliding conditions affecting
40 contact oxygenation. Another proposed mechanism for debris formation is the delamination theory of wear, in
41 which subsurface fatigue cracks in the substrate parallel to the contact surface become connected to the
42 surface and plate-like debris particles are released [28, 29]. Waterhouse proposed that such delamination
43 occurs in fretting and is responsible for 1.5-3.5 μm thick plates of oxide-coated metal in fretting debris, which
44 become increasingly oxidised as they become comminuted in the contact [29].
45
46
47
48
49
50
51
52
53
54
55
56

57 Qualitative observations of the debris particle morphology showed significant variation in size and the nature
58 of surfaces of the ejected debris particles, although a common feature was that a majority of the particles
59
60
61
62
63
64
65

examined can be seen to consist of many sub-micron crystallites which by XRD and EDX analyses were identified to have the $\alpha\text{-Fe}_2\text{O}_3$ structure. The crystallites were not observed in isolation in the SEM observations, suggesting a strong tendency for agglomeration and sintering of these particles. A general expression for the sintering rate of particles, S , is shown in Equation 1 as a function of oxygen diffusion coefficient, D , particle size, d , and constants, a , m and n [13]:

$$S = a \frac{D^m}{d^n} \quad (1)$$

Kato and Komai proposed that the value of n (and hence the strength of dependence of sintering rate on particle size) in tribological conditions is substantially lower ($n = 0.6$) than that in conventional sintering processes (where $n = 3 - 4$) [13], although this still suggests that particle size is a strong driver in tribo-sintering, and that small particles will sinter very much faster than larger particles. Kato and Komai observed that particles 300 nm in diameter were observed to sinter during a sliding wear test; this observation is in line with SEM observations of ejected debris undertaken in this study, in which particles of various sizes ejected during fretting were shown to be made up of haematite crystallites on the order of 0.1 μm in size.

The heavy deformation of contacting asperities in fretting and the consequent high reactivity of nascent metal surfaces exposed results in rapid oxidation. The oxidation process itself along with the frictional power dissipation associated with the contact result in very significant increases in local surface temperature (known as flash temperatures) [30]. The normal and shear loads in the contact result in third body particles being compacted, which along with elevated temperature and small particle size fulfil the necessary conditions for sintering.

Different stages of sintering are indicated by the surface textures observed in debris particles ejected over the course of 10^6 cycles, as shown in Figure 6; for example, Figure 6(c) shows a smooth, glassy region of the surface of a larger particle (most likely to be a piece of detached debris bed) suggesting an advanced degree of sintering has taken place, while the more porous surface observed in Figure 6(d) is similar in appearance to vermicular structures exhibited during conventional sintering processes.

Pieces of debris of up to at least 100 μm in size were observed in the ejected debris (illustrated in Figure 7), consistent in appearance and composition to debris beds adhered to the worn surfaces (Figure 9). The presence of such 'particles' suggests that there are several distinct mechanisms underlying the formation of ejected debris. Many particles examined did not appear similar to debris beds, suggesting that they were ejected before becoming incorporated in debris beds. It is recognised that the debris particles which have been ejected from the contact may have very different histories, but the following is proposed.

Oxide particles with a haematite structure of the order of 0.1 μm in size are formed. These rapidly agglomerate and sinter to form particles which cover a range of sizes up to a few microns. Some of these particles are ejected from the contact before they sinter into beds, whilst others will be retained in the contact and will form part of a debris bed. The surface of the debris bed is more fully sintered to form a glaze-like structure. After a certain time, the debris bed becomes detached and is then removed from the contact; some of these

bed fragments are removed with little comminution (yielding debris particles of $> 100 \mu\text{m}$ in size) whilst others may be more finely comminuted.

The particle size distributions (Figure 5) indicate that the debris resulting from a test of only 10^4 cycles is not significantly different from that resulting from a test of duration 10^6 cycles, suggesting that the process has reached steady state even by 10^4 cycles. SEM observations of wear scars in plan view (Figure 9) show qualitatively that there is a significant increase in scar width and a greater extent of debris bed coverage, suggesting that the bed becomes more developed and therefore detached pieces may be larger, although the particle size data in Figure 5(a) suggests that such pieces are likely comminuted before ejection from the contact in most cases.

Figure 5(b) indicates a distinctly different particle size distribution for debris ejected over 10^6 cycles when stroke is varied, with a narrower distribution of particle sizes with the smaller applied displacement. This may indicate that particles are not as easily removed from the contact with the smaller stroke; small particles are therefore sintered to form larger particles (thus reducing the fraction of smaller particles), and large pieces of debris bed which become detached are more effectively comminuted (thus reducing the fraction of larger particles). It is also notable that the metallic fraction in the ejected debris is smaller for the smaller applied displacement amplitude, again indicative of less aggressive conditions in this test, but also of debris particles spending longer in the contact resulting in more complete oxidation of any metallic particles produced.

5 Conclusions

Fretting tests were conducted using steel specimens and ejected debris was collected and analysed to investigate debris composition, particle size and morphology. The ejected debris was found to consist of oxide with a haematite ($\alpha\text{-Fe}_2\text{O}_3$) structure and a metal fraction (ferrite); the ferrite fraction fell from $\sim 5.8\%$ to $\sim 2.6\%$ as the applied displacement amplitude was reduced from $100 \mu\text{m}$ to $25 \mu\text{m}$.

The debris particles were shown to be made up from haematite crystallites of the order of $0.1 \mu\text{m}$ in diameter which had been sintered to different levels. In some cases, sintering had resulted in a vermicular structure whilst in others, a fully sintered, glassy structure was observed. The particle size in the ejected debris was found to be largely independent of number of fretting cycles in the range studied, indicating that a quasi-steady state in debris ejection was reached before 10^4 cycles. However, the particle size was found to vary with displacement amplitude, with a narrower distribution being observed for a shorter stroke. It is proposed that the debris is made up of particles, some of which have been ejected before they were sintered into a debris bed in the contact, whilst others have been sintered into a bed and then expelled as the debris bed has become detached.

Acknowledgements

The authors would like to acknowledge Rolls-Royce plc for their support and funding of this research through the Transmissions University Technology Centre (UTC) at the University of Nottingham, UK.

The authors thank the Nanoscale and Microscale Research Centre (nmRC) for providing access to instrumentation.

6 References

- [1] Nurmi V, Hintikka J, Juoksukangas J, Honkanen M, Vippola M, Lehtovaara A, et al. The formation and characterization of fretting-induced degradation layers using quenched and tempered steel. *Tribology International*. 2019;131:258-67.
- [2] Kirk AM, Shipway PH, Sun W, Bennett CJ. The effect of frequency on both the debris and the development of the tribologically transformed structure during fretting wear of a high strength steel. *Wear*. In Press 2019.
- [3] Hurricks PL. The mechanism of fretting - A review. *Wear*. 1970;15:389-409.
- [4] Dobromirski JM. Variables of Fretting Process: Are There 50 of Them? Standardization of Fretting Fatigue Test Methods and Equipment: ASTM; 1992. p. 60-6.
- [5] Berthier Y, Vincent L, Godet M. Velocity accommodation in fretting. *Wear*. 1988;125:25-38.
- [6] Godet M. The third-body approach: A mechanical view of wear. *Wear*. 1984;100:437-52.
- [7] Uhlig H. Mechanism of fretting corrosion. *J Appl Mech*. 1954;21:401-7.
- [8] Uhlig H, Feng IM, Tierney WD, McClellan A. A fundamental investigation of fretting corrosion. Technical Note 3029, NACA. 1953.
- [9] Waterhouse RB. Fretting corrosion. *Proceedings of the Institution of Mechanical Engineers*. 1955;169:1157-72.
- [10] Waterhouse RB. Fretting corrosion: Pergamon press; 1972.
- [11] Iwabuchi A, Hori K, Kubosawa H. The effect of oxide particles supplied at the interface before sliding on the severe-mild wear transition. *Wear*. 1988;128:123-37.
- [12] Berthier Y, Vincent L, Godet M. Velocity accommodation sites and modes in tribology. *European Journal of Mechanics, A/Solids*. 1992;11:35-47.
- [13] Kato H, Komai K. Tribofilm formation and mild wear by tribo-sintering of nanometer-sized oxide particles on rubbing steel surfaces. *Wear*. 2007;262:36-41.
- [14] German RM. Sintering theory and practice. A Wiley-Interscience Publication. 1996:568.
- [15] Adachi K, Kato K. Formation of smooth wear surfaces on alumina ceramics by embedding and tribo-sintering of fine wear particles. *Wear*. 2000;245:84-91.
- [16] Viat A, Dreano A, Fouvry S, De Barros Bouchet MI, Henne JF. Fretting wear of pure cobalt chromium and nickel to identify the distinct roles of HS25 alloying elements in high temperature glaze layer formation. *Wear*. 2017;376-377:1043-54.
- [17] Hayes EK, Shipway PH. Effect of test conditions on the temperature at which a protective debris bed is formed in fretting of a high strength steel. *Wear*. 2017;376-377:1460-6.
- [18] Pearson SR, Shipway PH, Abere JO, Hewitt RAA. The effect of temperature on wear and friction of a high strength steel in fretting. *Wear*. 2013;303:622-31.
- [19] Dies K. Friction Oxidation as a Chemico-Mechanical Process. *Archiv fur das Eisenhullenwesen*. 1943;10:399-407.
- [20] Feng IM, Uhlig HH. Fretting corrosion of mild steel in air and in nitrogen. *Journal of Applied Mechanics-Transactions of the ASME*. 1954;21:395-400.
- [21] Fenner AJ, Wright KHR, Mann JY. Fretting corrosion and its influence on fatigue failure. *International Conference on Fatigue of Metals, Institution of Mechanical Engineers*. 1956:368.
- [22] Halliday J, Hirst W. The fretting corrosion of mild steel. *Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences*. 1956;236:411-25.
- [23] Mohd Tobi AL, Ding J, Pearson S, Leen SB, Shipway PH. The effect of gross sliding fretting wear on stress distributions in thin W-DLC coating systems. *Tribology International*. 2010;43:1917-32.
- [24] Leen SB, Richardson IJ, McColl IR, Williams EJ, Hyde TR. Macroscopic fretting variables in a splined coupling under combined torque and axial load. *Journal of Strain Analysis for Engineering Design*. 2001;36:481-97.
- [25] Fouvry S, Liskiewicz T, Kapsa P, Hannel S, Sauger E. An energy description of wear mechanisms and its applications to oscillating sliding contacts. *Wear*. 2003;255:287-98.
- [26] Warmuth AR, Shipway PH, Sun W. Fretting wear mapping: The influence of contact geometry and frequency on debris formation and ejection for a steel-on-steel pair. *Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences*. 2015;471.
- [27] Fouvry S, Arnaud P, Mignot A, Neubauer P. Contact size, frequency and cyclic normal force effects on Ti-6Al-4V fretting wear processes: An approach combining friction power and contact oxygenation. *Tribology International*. 2017;113:460-73.
- [28] Suh NP. An overview of the delamination theory of wear. *Wear*. 1977;44:1-16.

- [29] Waterhouse RB, Taylor DE. Fretting debris and the delamination theory of wear. Wear. 1974;29:337-44.
- [30] Blok H. The dissipation of frictional heat. Applied Scientific Research, Section A. 1955;5:151-81.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9