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DOI:
10.1016/j.triboint.2020.106257

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Document Version
Peer reviewed version

Citation for published version (Harvard):

Link to publication on Research at Birmingham portal

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Damage monitoring of surface treated steel under severe rolling contact loading conditions

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Abstract

The aim of this study has been to investigate the rolling contact behaviour of plasma nitrided and duplex treated (plasma nitrided and diamond-like carbon (DLC)) EN40B steel under overloading and intermittent lubricating conditions using the acoustic emission technique (AE). The signal amplitude and energy were monitored and related to cracking and delamination events observed from scanning electron micrographs. AE technique was found to be capable of detecting cracking and delamination on both surface treatments, proving to be a useful tool for monitoring rotating machinery condition effectively.

Keywords: acoustic emission, rolling contact, plasma nitriding, diamond-like carbon

1. Introduction

Rotating machinery is used in a wide range of industries such as oil and gas, power generation, automotive, and aerospace. As such, rotating machinery is required to operate reliably with as little downtime as possible [1]. Bearings and gears are critical components in all types of rotating machinery. Both bearings and gears may experience structural degradation due to a variety of root causes, including rolling contact fatigue (RCF), overloading, misalignment, or lack of lubrication. If operating conditions are controlled within the nominal range of the design normally degradation will eventually occur through RCF. RCF thus

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remains one of the main causes of failure of components experiencing rolling and sliding surface contact interactions. RCF degradation arises from alternating stress fields either at the surface or near the surface of the material. This leads to the initiation and subsequent propagation of RCF cracks eventually leading to material breaking off in the form of micropitting or at more advanced stages spalling [2]. Catastrophic failure can occur as a result of excessive wear or misalignment leading to overloading within the contact region.

When the contacting elements are lubricated properly, RCF cracking typically will initiate below but near the surface. Subsequently, RCF cracks will propagate towards the surface. RCF cracks usually initiate around inclusions, hence the cleanliness of steel plays a major role in minimising RCF and prolonging the lifetime of bearings and gears [3]. RCF lifetime can also be impacted by other factors such as the material microstructure, residual stresses present, contact pressure, operating temperature and work-hardening response [3 4]. RCF affects only the regions near the surface of the material, due to the stress fields being highly localised there. Contact widths are usually between 200 µm to 1000 µm [3].

Advanced surface engineering techniques are widely employed in order to increase RCF lifetime of rotating components. Nonetheless, surface treated gears and bearings may experience RCF failure due to the shear stress effect between the coating and the substrate [5]. RCF testing is commonly used to simulate the kinematic conditions of bearings and gears [6]. AE techniques can be used to monitor coatings exposed to RCF conditions [7, 8, 9, 10]. Guo and Schwach [11] employed AE to monitor the effect of a white layer formed at the surface of a machined component under RCF conditions. They compared samples of AISI 52100 bearing steel with and without the white layer. They found that the AE technique was sensitive enough to detect cracking initiating due to RCF. Amplitude, energy, and root mean square (RMS) were the most sensitive parameters to RCF crack initiation and propagation. Cockerill et al. [1] used AE in order to determine the condition of cylindrical rolling bearings under rolling contact conditions. The speed and load effects were studied. They
found that the increase in speed had a bigger impact on the RMS signal level than load increase. Additionally, they observed that during a period of constant wear, the resonant frequencies of the bearings increased in amplitude with time, even though parameters such as speed and load remained unchanged.

Kang et al. [5] studied the delamination failure of plasma sprayed composite ceramic (AT40) coating under RCF with the help of an AE monitoring system. They were able to relate the AE signal waveforms and frequencies to three different stages of the delamination process (normal contact, fatigue crack initiation, and delamination failure).

This paper has investigated the applicability of AE testing for monitoring damage during the RCF testing of plasma nitrided and duplex (plasma nitrided and DLC) treated samples subjected to overloading conditions.

2. Experimental procedure

2.1. Surface treatments

The substrate chosen for this work was EN40B steel. The composition of EN40B steel grade is given in Table 1.

Table 1: Chemical composition in weight of the EN40B steel.

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>S</th>
<th>P</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>EN40B</td>
<td>0.20 -</td>
<td>0.10 -</td>
<td>0.40 -</td>
<td>2.90 -</td>
<td>0.00 -</td>
<td>0.40 -</td>
<td>0.00 -</td>
<td>0.00 -</td>
<td>Balance</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>0.35</td>
<td>0.65</td>
<td>3.50</td>
<td>0.40</td>
<td>0.70</td>
<td>0.05</td>
<td>0.05</td>
<td></td>
</tr>
</tbody>
</table>

The EN40B was active screen plasma nitrided using a Klockner Ionon 60 kVA plasma nitriding machine at the School of Metallurgy and Materials in the University of Birmingham. The treatment was performed at 550°C for 20 hours, in a working gas atmosphere of 25% N₂ and 75% H₂. The treatment temperature was achieved with an atmosphere of H₂. The pressure of the gas mixture was 4 mbar.

Further to plasma nitriding, half of the samples were in addition coated with diamond-like carbon (DLC). The DLC type chosen for this work was
BALINIT®C, procured from Oerlikon Balzers Coatings Ltd. BALINIT®C is a WC/a-C:H coating. A thin Cr interlayer is used to increase the adhesion to the substrate.

X-ray diffraction (XRD) tests were performed using an X’Pert Philips XRD instrument with Cu Kα (k = 0.145 nm) radiation. The 2θ range was measured from 30° to 110°. A Mitutoyo MVK-H1 hardness testing machine with a Vickers diamond indenter was used to evaluate the microhardness of the plasma nitrided samples. A Nano Vantage (Micro Material, UK) machine was used to characterise the hardness of the DLC coating. The depth of each indent was controlled to 100 nm. A laser Raman spectroscope, containing a Renishaw In-Via reflex spectrometer was used to analyse the DLC coatings. The laser used had a wavelength of 488 nm and a power of 2 mW. The measurements were performed in an Instec HCS621V cell with a pressure and flow of argon gas of 1 bar and 100 ml min$^{-1}$, respectively.

2.2. Rolling contact testing

The tribometer used in this study was an AMSLER A135 wheel-on-wheel universal tribometer, shown in Figure 1.

![Figure 1: AMSLER A135 tribometer.](image)

The test configuration consists of two specimens rotating against one another. The upper specimen rotates with a speed of 360 rpm whereas the lower
rotates at 400 rpm. A load is then applied to one of the wheels. The test can be run in both dry or lubricated conditions. In the case of lubricated conditions, the sample can be partially submerged in oil. Zhou [12] used the same machine to test two steel wheels under lubricated conditions, at the maximum load possible (2000 N), generating 1 GPa of contact stress, for more than 350 hours (or 8.4 million cycles). Little to no wear was observed and running the test for longer would make the experiments impractical due to time constraints of the study. Therefore, to accelerate the occurrence of damage initiation, contact stress had to be increased. Since the applied load was already maximised, the solution selected to address this problem was to use a counter wheel made of tungsten carbide (WC) with an appropriate surface contour. The WC wheel was manufactured having a spherical surface contour resulting in higher contact stress being applied. Hence, wear could accelerate further.

The design of both the WC counter wheel and the steel samples can be seen in Figure 2. The WC wheel was chosen to be the slowest rotating wheel in order to achieve a slip ratio of 18%. Considering the slip ratios between the flanks of gears are usually in between 0% and 20% the slip ratio achieved was within acceptable limits [13]. The slip-ratio $g$ can be calculated with the Equation 1 where $r_1$ and $r_2$ are the radii of the wheels, and $\omega_1$ and $\omega_2$ are their respective rotational speeds [14]. In order to make the tests comparable, each sample was polished to an average roughness of 0.5 µm.

$$g(\%) = 200 \left( \frac{r_1 \omega_1 - r_2 \omega_2}{r_1 \omega_1 + r_2 \omega_2} \right)$$ (1)
The rolling contact tests were performed at room temperature, with ambient humidity, and under intermittent lubricating conditions. The samples were first tested in dry conditions for 800 cycles, and then lubricant was introduced for another 71,200 cycles. After the completion of 72,000 cycles, the samples were removed from the tribometer and inspected. The test was then restarted for another 72,000 cycles increment. This routine was repeated until visual damage was observed or 2 million cycles were reached. For lubrication during testing 15W-40 Shell engine oil was employed. Before and after each test, the wheels were ultrasonically cleaned and subsequently dried.

The acoustic emission signals were detected and recorded using a 4-channel AE system procured from Physical Acoustics Corporation (PAC, now Mistras). The data acquisition was performed using AEwin software. One 150-700 kHz PAC R50α piezoelectric AE sensor was coupled to the oil container of the tribometer (to which the steel test wheel was partially submerged) with epoxy adhesive (Araldite®) and connected to a PAC model 2/4/6 pre-amplifier. Hsu-Nielsen source (pencil lead break) tests were performed before each test to confirm the acoustic coupling quality consistency. The AE parameters for the rolling contact tests can be seen in Table 2.
<table>
<thead>
<tr>
<th>AE parameter</th>
<th>RCF test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Threshold</td>
<td>40 dB</td>
</tr>
<tr>
<td>Pre-Amplification level</td>
<td>40 dB</td>
</tr>
<tr>
<td>Analogue filter</td>
<td>0.1 - 1 MHz</td>
</tr>
<tr>
<td>Sampling rate</td>
<td>1 MSPS</td>
</tr>
<tr>
<td>Pre-Trigger</td>
<td>512 µs</td>
</tr>
<tr>
<td>Length</td>
<td>10 k</td>
</tr>
<tr>
<td>Peak Definition Time</td>
<td>300 µs</td>
</tr>
<tr>
<td>Hit Definition Time</td>
<td>600 µs</td>
</tr>
<tr>
<td>Hit Lockout Time</td>
<td>4000 µs</td>
</tr>
<tr>
<td>Duration</td>
<td>100 ms</td>
</tr>
</tbody>
</table>

The resultant contact area between the WC steel wheel is an ellipse. In order to calculate the contact pressure, the reduced radii in the x and y coordinates must be calculated using Equations 2 and 3, where \( r_{1x} \) and \( r_{1y} \) refer to the radii of the WC wheel, and \( r_{2x} \) and \( r_{2y} \) to the steel wheel. The final reduced radii \( (r) \) is given by Equation 4. 

\[
\frac{1}{r_x} = \frac{1}{r_{1x}} + \frac{1}{r_{2x}} \tag{2}
\]

\[
\frac{1}{r_y} = \frac{1}{r_{1y}} + \frac{1}{r_{2y}} \tag{3}
\]

\[
\frac{1}{r} = \frac{1}{r_x} + \frac{1}{r_y} \tag{4}
\]

Additionally, the reduced elasticity modulus \( (E_r) \) has to be calculated by Equation 5, where \( E_1 \) and \( \nu_1 \) are the elasticity modulus and Poisson’s ratio for the WC wheel, respectively, and \( E_2 \) and \( \nu_2 \) correspond to the same for the steel.
wheel.

\[
\frac{1}{E_r} = \frac{1 - \nu^2}{E_1} + \frac{1 - \nu^2}{E_2}
\]  

(5)

The ellipticity parameter is given by \( k_e \) (Equation 6), and \( S_e \) is the elliptic integral of the second kind, which can be approximated by Equation 7. The semi-minor \( (a_e) \) and semi-major \( (b_e) \) axes of the ellipse can then be calculated by Hertzian contact stress analysis following Equations 8 and 9. The load applied is given by \( L \) [16].

\[
k_e = 1.0339 \left( \frac{r_y}{r_x} \right)^{0.6360}
\]  

(6)

\[
S_e = 1.0003 + \frac{0.5968r_x}{r_y}
\]  

(7)

\[
a_e = \sqrt{\frac{3k_e^2S_eLr}{\pi E_r}}
\]  

(8)

\[
b_e = \sqrt{\frac{3S_eLr}{\pi k_e E_r}}
\]  

(9)

Finally, the maximum contact pressure \( (CP_{max}) \) can be calculated by Equation 10 [16].

\[
CP_{max} = \frac{3L}{2\pi a_e b_e}
\]  

(10)

By using the values in Table 3, the contact pressure can be calculated as a function of the load. All samples in this study were tested with a load of 1200 N, thus achieving a contact stress of 5.0 GPa.

Table 3: Parameters used for calculating contact pressure.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( r_x ) (mm)</th>
<th>( r_y ) (mm)</th>
<th>E (Gpa)</th>
<th>( \nu ) (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC</td>
<td>5</td>
<td>17.5</td>
<td>650</td>
<td>0.2</td>
</tr>
<tr>
<td>EN24T</td>
<td>( \infty )</td>
<td>19</td>
<td>187</td>
<td>0.3</td>
</tr>
</tbody>
</table>
3. Results

3.1. Characterisation of plasma nitrided steel

The cross-section of a nitrided EN40B steel can be seen in Figure 3. The overall diffusion layer can be seen in Figure 3a. A closer look of the nitriding layer surface (3a) shows a thin compound layer. This can be observed in more detail in Figure 3b. The compound layer is brittle and can have an adverse effect to the wear resistance of nitrided steels [17]. It is also softer than the nitrided layer [18]. More often, the compound layer is formed from a mixture of $\gamma'$-Fe$_4$N and $\epsilon$-Fe$_{2-3}$N phases. In the nitrided EN40B steel, an interface in the compound layer is distinguishable, with a columnar $\epsilon$ phase at the top and an angular $\gamma'$ at the bottom. The existence of both phases is confirmed by the XRD shown in Figure 4.

Figures 3c and 3d show a continuous network of prior austenite grain boundary precipitates. Mridha and Jack [18] found that the microstructure of a EN40B nitrided steel evolves due to the formation of fine CrN plates in the nitrided zone. These plates create large compressive stresses parallel to the specimen surface. If following nitriding, no compound layer is formed, decarburisation is likely to occur. However, as this is not the case for the nitriding used in this study, the released carbon from the cementite is instead driven deeper into the bulk of the sample as a result of the nitrogen-rich surface, creating a carbide-enriched sub-layer. Due to the compressive stress, the cementite precipitates in the prior austenite grain boundaries, in a direction parallel to the surface of the specimen. This is confirmed with an EDS line scan of a carbide, as Figure 5 shows. This continuous network of carbides is usually detrimental to the ductility of nitrided steels [19].
Figure 3: SEM micrographs showing the nitrided layer at different regions of the nitrided sample. Etched with 2% nital. Figure (c) shows the whole sample at a low magnification. Figure (a) shows the nitrided layer with no prior austenite grain boundary precipitates. Figure (b) shows nitrided layer with the columnar $\epsilon$ on top and $\gamma'$ at the bottom. Figure (d) shows the continuous network of prior austenite grain boundary precipitates.
A microhardness profile of the nitrided EN40B samples is shown in Figure 6. The hardness of the case is 800 HV, and the case depth, defined as the distance to the surface to a point where the hardness is 400 HV [20], is 300 µm. The hardness decreases at 200 µm, which is comparable with the size of the diffusion zone shown in Figure 3.
Figure 5: SEM micrograph of the EN40B carbide and the location on the EDS line scan, etched with 2% nital (a), and the results of the EDS line scan for carbon and nitrogen (b).

Figure 6: Microhardness profile of the nitrided EN40B steel.
3.2. RCF and AE behaviour of plasma nitrided surfaces

The AE system captured several high energy signals at the very beginning of the rolling contact test. This was due to the removal of the white layer, which is brittle and breaks off easily at such high contact pressures, as shown in Figure 8. As soon as this layer was removed, no signals were detected up to 1.2 million cycles. AE signals begun to be detected again in the latter half of the test. These signals can be associated with cracking at the edges of the wear track, as shown in Figure 9. The nitrided layer is quite hard, and thus less ductile. Instead of deforming, it cracks and breaks off at the wear track edges.

![Figure 7: AE hits and cumulative energy of the plasma nitrided intermittent sample as a function of the number of cycles.](image-url)
cracking/delamination  white layer removal

Figure 8: SEM micrograph of the cross-section of the plasma nitrided intermittent sample showing cracks and removal of the white layer. Etched with 2% nital.

Figure 9: SEM micrographs of the wear track edge of the plasma nitrided intermittent sample after 1.3 million cycles (a), and 2 million cycles (b).

It was thought that the carbides located at the prior austenite grain boundaries could reduce RCF lifetime through initiation of cracks. However, no defects
were found, as shown in Figure 10. The AE system proved to be able to detect the white layer removal and cracking at the wear track edges.

Figure 10: SEM micrograph showing the cross-section of the wear track at the carbide-enriched zone. Etched with 2% nital.

3.3. Characterisation of duplex treated steel

The duplex surface system was created by applying DLC coatings to the nitrided EN40B. The DLC layer is distinguishable from the chromium interlayer, which is thinner and acts as a bonding agent between the coating and the substrate. The elements are confirmed by EDS shown in Figure 11.
Figure 11: SEM micrograph of the DLC layer for the EN24T sample, etched with 2% nital, with the yellow line showing the position of the EDS line scan (a), and the results of the EDS line scan (b).

The nanoindentation results are shown in Figure 12. The DLC coating was found to have a hardness of 10.2 ± 0.5 GPa and an elastic modulus of 111.0 ± 3.6 GPa.
Figure 12: Nanoidentation results for 5 different indents on the DLC layer.

Figure 13 shows the Raman spectrum of the DLC coating. Two bands can be distinguished from the spectrum. These are defined as the sp² graphite band and the sp³ disordered band [21]. The disordered peak is located at 1392 cm⁻¹ with an intensity of 605, while the graphite peak is located at 1559 cm⁻¹ with an intensity of 910. The ratio of the intensity of the disordered peak over the intensity of the graphite peak gives the component ratio of sp² and sp³ clusters, which in this analysed DLC coating is 0.66.
Figure 13: Raman spectrum of the DLC coating.

3.4. RCF and AE behaviour of duplex treated surfaces

The AE data of the duplex sample during the rolling contact test are shown in Figure 14. A large number of signals were detected at the start of the test. These signals are related with the fracture of both the DLC and the white layer, as shown in Figure 14. After this, no signals were detected until 0.5 million cycles. These signals are related to cracking at the wear track edges, shown in Figure 16. This was due to the low ductility of the plasma nitrided and DLC layers. Cracking continued, with high energy signals being detected until the test was stopped at 1.3 million cycles due to the wheel being severely damaged. Similarly to the plasma nitrided samples, no cracks were found at the carbide enriched zone.
Figure 14: AE hits and cumulative energy of the plasma nitrided and DLC intermittent sample as a function of the number of cycles.

Figure 15: SEM micrographs of the cross-section of the plasma nitrided and DLC intermittent sample after 1.3 million cycles, showing fracture of the DLC layer. Etched with 2% nital.
The addition of a DLC coating to the plasma nitrided layer did not exhibit any improvement in comparison with the use of nitrided layer only. The DLC coating, being only 4 µm thick, could not sustain the 5 GPa stress and was quickly worn away. The AE monitoring system detected the cracking of the DLC coating, as well as the wear track edge cracking.

4. Discussion

A typical wavelength and its corresponding Fast Fourier Transform (FFT) for both samples can be seen in Figure 17. The peak frequency identified was between 230 and 250 kHz, which is in line with the peak frequency of 249 kHz observed by Kang et al. [5] and identified as the peak frequency for fatigue crack initiation of plasma sprayed ceramic coatings under RCF. The similar wavelengths and FFT spectra suggest a similar mode of failure for both samples.
Figure 17: Typical AE wavelength for both samples (a), and Fast Fourier transform of the wavelength showing peaks at between 230 and 250 kHz.

The cumulative AE energy plot can be a good indicator of accumulated damage. From Figure 7 it is possible to see that most of the AE energy for the plasma nitrided sample is captured at the very beginning of the test due to the cracking and removal of the white layer. Following this there are no acoustic emission signals detected up to 0.9 million cycles. This suggests that there was no damage taking place during this period. In order to compare the plasma nitrided sample with the plasma nitrided and DLC sample, the signals from the removal of the white layer were subtracted and plotted in Figure 18. This was not done for the nitrided and DLC sample as its signals are sparser and contain much less energy, having much less influence in the total cumulative AE energy.
The cumulative AE energy graph for both samples show a similar trend, suggesting that the damage evolution of both samples are similar. This is corroborated by the SEM micrographs for both samples, which show cracks of similar length. The main difference, however, is that the damage accumulation for the nitried and DLC sample starts more than a million cycles earlier. Therefore, in overloading and intermittent lubrication conditions the addition of a DLC layer on top of the nitriding treatment reduced the lifetime of the specimen.

5. Conclusions

AE testing has been proven to be a reliable method of detecting defect initiation and propagation during rolling contact testing of nitride and DLC-
coated EN40B steel samples. Even though the AE sensor had to be coupled to the oil container, which results in reflection and absorption of part of the energy of the AE signal, it was still successful in detecting RCF cracking initiation and propagation, and delamination events. The present study clearly demonstrates that there is strong potential for the meaningful application of AE monitoring of rotating machinery, as it is proven to be a reliable method for detecting RCF damage initiation and propagation. Further investigation will focus on the quantification of the damage detected using AE signals.

Acknowledgements

The authors wish to express their gratitude to the European Commission and the National Council of Technological and Scientific Development of Brasil (CNPq) for the financial support received through the OPTIMUS FP7 project and Science without Borders Scheme respectively.

References


