Comparing Tribochemical Film Formation and Durability at Steel and CrN Coating Interface in Boundary Lubrication

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One of the main challenges that engine oil manufacturers are now facing is to reduce friction losses while at the same time increasing the durability of engine components. New materials are being used for engine components, alongside and to replace conventional ferrous materials. More components are being coated with low friction and highly durable coatings. Lubricants are required to lubricate these surfaces as effectively as conventional materials, with no environmental impact. Knowing that the additive package in today’s lubricants is optimised for lubricating conventional ferrous materials it is of paramount importance to understand how effective the current additives are in lubricating non-ferrous materials.

The aim of the current paper is to understand the formation and durability of tribofilms on a Chromium Nitride (CrN) coating formed from two conventional additives; Zinc Dialkyldithiophosphate (ZDDP) and Molybdenum Dialkyl Dithiocarbamate (MoDTC) additives. Tribofilm formation was studied by testing model lubricants containing ZDDP and MoDTC additives on the CrN coating in boundary lubrication, taking the performance on a ferrous system as a reference. The durability was assessed by assessing the tribocouple friction performance in base oil once the tribofilm was formed. Wear scars were fully chemically characterised using X-ray Photoelectron Spectroscopy (XPS). The study shows that the chemical nature of the tribofilms formed on steel and CrN is similar. In both materials, tribofilms were shown to have comparable durability. The factors which affect tribofilm durability and formation, and the reason why ferrous systems and CrN show similarities are discussed.

Keywords: tribofilm formation, tribofilm removal, tribochemistry, ZDDP, MoDTC

1. Overview

Low friction and wear in the engine valve train and in piston ring/cylinder liner tribological contacts is maintained by the additive package in the lubricants. Although there is a range of additives used in lubricants 1), of particular importance are surface active additives such as ZDDP, a very effective anti-wear additive and MoDTC, an effective friction modifier. These two additives react with the lubricated surfaces to form very thin protective films, tribofilms, which provide low friction and low wear. The mechanism of tribofilm formation from these two additives on steel is relatively well understood2,3). Tribofilm formation on steel has been the subject of several review papers which have summarised the knowledge obtained from research going back to the 1950s4,5). However, the effect of substrate material on the tribofilm formation process as well as the effect of the substrate on the subsequent durability of the tribofilm still remains poorly understood.

So et al. 7) have shown in their work that at a lubricant temperature above 80 °C a chemisorbed ZDDP film is formed on the lubricated interface. Besides chemisorbed species, the ZDDP additive components will also physically adsorb to the surface, especially at low temperatures, and will also chemically react with the lubricated materials. In this case, the components that contribute to the growth rate of the effective film thickness, \( h_{\text{effect}} \), can be expressed as8):

\[
    h_{\text{effect}} = h_{\text{phy}} + h_{\text{chem}} + h_{\text{react}}
\]

where

\[
    h_{\text{phy}} - \text{physorbed film growth rate}
\]

\[
    h_{\text{chem}} - \text{chemisorbed film growth rate}
\]

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$h_{\text{react}}$ - chemically reacting film growth rate.

At the interface between boundary lubricated surfaces a dynamic process, in which the tribofilm forms and is removed, exists and the rates of these processes are a function of the contact conditions and interactions of the additives with the substrate. For good tribological performance, the rate of tribofilm removal should always be lower or equal to the rate of the tribofilm formation. This is expressed by Lin et al. \(^9\) using equation 2.

$$h_{\text{effect}} \geq h_{\text{scrape}} \quad (2)$$

where $h_{\text{scrape}}$ defines the tribofilm removal rate.

A change of the substrate material will affect the $h_{\text{chem}}$ and $h_{\text{react}}$ components of the tribofilm growth. At the same time, the tribofilm removal rate will be affected by the nature of chemical interaction between additive molecules or its components with the substrate material. Both these processes confirm the importance of the substrate material in the overall properties of the tribofilm. Understanding them is essential for the process of lubricant blending based on the material being lubricated.

In the current study the formation and durability of tribofilms formed from ZDDP and MoDTC additives on a CrN coating in boundary lubrication conditions, in comparison to the formation and durability of tribofilms formed on steel, are evaluated. The chemical nature of tribofilms formed as well as the chemical nature of the same tribofilms when subsequently rubbed in base oil with no additive replenishment was probed using XPS.

2. Experimental

2.1. Tribological experiments

Since the additive effectiveness is most significant in the boundary lubrication regime, the experimental work is devised in such a way to enable testing of model lubricants in this regime. For this, the pin-on-reciprocating plate apparatus is used, in which the friction force is measure via a bi-directional load cell with measuring range to 100 N and combined error of $\pm 0.0037$ N. This error is the unavoidable error and includes errors from non-linearity, hysteresis and temperature effects on load cell sensitivity. The duration of tests to obtain stable tribofilms was 4 hours. Friction force data are collected every 3 minutes for 2 s (120 points), which corresponds to two 10 mm stroke cycles. The 120 data collected are then averaged and this value is used to calculate the friction coefficient. As a final output, friction coefficient is shown as a function of time for the duration of the test. The tests were replicated at least three times showing a good repeatability of friction as a function of time. A repeatability of friction coefficient in the last hour of the test of $\pm 0.004$ is recorded. The tribofilm formation was evaluated by analysing the films formed as a result of testing model lubricants on fresh material couples for 4 hours while the tribofilm removal was assessed by chemically characterising the initial tribofilms after they were rubbed further in base oil with no additive replenishment for 1 hour.

The lubrication regime is determined by the calculation of lambda ratio ($\lambda$), which is the ratio between the minimum film thickness and composite root mean square surface roughness. The test conditions, given in Table 1, ensure a lambda ratio in a region of 0.02-0.03.

### Table 1 Testing conditions

<table>
<thead>
<tr>
<th>Load</th>
<th>Hertzian contact pressure</th>
<th>Average sliding speed</th>
<th>Lubricant temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>188  N</td>
<td>Steel/Steel 650 MPa</td>
<td>0.02 m/s</td>
<td>100 °C</td>
</tr>
</tbody>
</table>

2.2. Materials and lubricants

The CrN coatings were deposited on polished steel plates in an industrial closed field unbalanced magnetron sputter ion plating system with chromium targets. For all experimental work AISI 52100 steel was used as the pin material. The radius of the hemispherical end of the pin was 40 mm and the roughness varied from $R_q 0.15 - 0.2 \mu m$. For comparison, an uncoated steel/steel material combination was also used in this study. Table 2 gives the physical properties of the materials used. The plate wear scar was measured using an optical white light interferometer while wear on the pin was calculated following the measured wear scar diameter.

### Table 2 Physical properties of the materials tested

<table>
<thead>
<tr>
<th>Properties of Coating/Material</th>
<th>Bearing AISI 52100 Steel/Substrate</th>
<th>CrN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness of the coating</td>
<td>-</td>
<td>4.5 µm</td>
</tr>
<tr>
<td>Hardness</td>
<td>6.5 GPa</td>
<td>21.9 GPa</td>
</tr>
<tr>
<td>Roughness, $R_q$</td>
<td>0.15-0.2 µm</td>
<td>0.04-0.06 µm</td>
</tr>
</tbody>
</table>

Although the surface roughness of the CrN coating was lower than the steel roughness, the lubrication mode was still in the boundary regime. The contact pair was immersed in the lubricant to be tested. For each test 3 ml lubricant was used. The lubricants used are defined in Table 3.

2.3. XPS analysis

The chemical nature of the tribofilms formed after 4-hour tests using ZDDP and ZDDP/MoDTC lubricants in both material systems, as well as the chemical composition of the same tribofilms rubbed further in base oil for one hour, was obtained. This methodology gave important information on the nature of species.
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formed on the wear scar as well as the chemical composition of the wear scar once the replenishment of additive has been stopped giving valuable qualitative information on the durability of the species formed.

The chemical composition was obtained from an area of 500 µm \texttimes{} 500 µm in the wear scar of the plates using a monochromatized AlK\( \alpha \) source in the XPS. The samples were cleaned using n-heptane to remove residual oil and contaminants before doing XPS analysis. Spatial mode was chosen to acquire the spectra and an argon etching was performed using ion gun set at beam energy 3 keV and 1 µA in an area of 1 mm\(^2\) on the wear scar. The peaks obtained from long scans were further analysed by curve fitting determining the species detected. For this the CasaXPS software was used. The data obtained were compared with standard spectra and with the tabulated spectra from references\(^9\)\textendash{}\(^{11}\).

3. Results and discussion

3.1. Friction performance

Figure 1 shows the steady state friction coefficient, averaged from the friction values obtained during the last hour of the test, as a function of the lubricant and material tested.

In the case of the ZDDP lubricant, friction for the CrN/steel system is shown to be slightly lower compared to the steel/steel system. The presence of the MoDTC additive in the lubricant resulted in friction reduction not only for steel, as expected, but also when CrN material was tested. This suggests that MoDTC is also active in friction reduction when CrN coating is lubricated. The wear values of both pin and plate wear scars were in the range of 10\(^{-17}\) m\(^3\)/Nm showing that wear with every material combination used was of comparable values to the wear experienced in the cam/follower system.

3.2. Changing lubricants friction performance

Figure 2 and Figure 3 show the friction performance once the replenishment of the additive has stopped by changing the lubricant from ZDDP and ZDDP/MoDTC, respectively, to base oil (BO). In case of the ZDDP lubricant, Figure 2, even after changing the lubricant to BO the friction performance did not change significantly during the following 60 minute test. The friction performance after changing oil to BO is similar to the friction performance obtained by testing BO on fresh materials. Since the friction coefficient for a ZDDP tribofilm is not significantly different to the one obtained from BO, it is difficult to get any durability data of the ZDDP tribofilm from the friction data. Knowing that ZDDP is a very good antiwear additive, it would be interesting to record the wear data in real time. This will be subject of authors’ future work.

Friction performance of the ZDDP/MoDTC lubricant started high but after 2 minutes sliding the friction started to reduce stabilising at value of around 0.06. Changing the lubricant to base oil, that is stopping the MoDTC additive replenishment, resulted in steady increase of the friction coefficient, reaching 0.08 after 1 hour rubbing at the same testing conditions.

The friction performance on steel is in agreement with authors previous tests of the ZDDP/MoDTC oils on steel sample\(^{12}\) but surprisingly, the rate of friction change, shown in Figure 3 by the slope of the friction coefficient curve as a function of time, does not change significantly when the CrN coating was tested. The chemical nature of the tribofilms formed is shown in the following sections of this study.

3.3. Chemical nature of the tribofilm

3.3.1. ZDDP tribofilm formation on steel and CrN materials

Table 4 shows the binding energies of the peaks obtained from analysing wear scars produced from testing steel and CrN materials with a steel counterbody using the ZDDP lubricant. The numbers in brackets in Table 4 show the peak area, giving an indication of the concentration of the element in the area analysed. All the information shown is obtained by curve fitting of the XPS peaks acquired after 30 seconds Ar\(^+\) etching. Binding energies of P 2p and O 1s peaks indicate that a

<table>
<thead>
<tr>
<th>Designation</th>
<th>Lubricant</th>
<th>Dynamic Viscosity at 100 °C (Pa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BO</td>
<td>Poly Alpha Olefin 6 – PAO6</td>
<td>0.004</td>
</tr>
<tr>
<td>ZDDP</td>
<td>PAO6 + 0.6 mass% ZDDP</td>
<td></td>
</tr>
<tr>
<td>ZDDP/MoDTC</td>
<td>PAO6 + 0.6 mass% ZDDP + 1.1 mass% MoDTC</td>
<td></td>
</tr>
</tbody>
</table>

Table 3 Lubricants composition and designation

Figure 1 Steady state friction coefficient for steel and CrN materials lubricated with ZDDP and ZDDP/MoDTC lubricants

Figure 2  Friction performance when the lubricant is changed from ZDDP to BO

Figure 3  Friction performance when the lubricant is changed from ZDDP/MoDTC to BO

Table 4  The binding energies (eV) of XPS peaks detected on the ZDDP tribofilm formed on steel and CrN coating. Numbers in brackets show the peak area

<table>
<thead>
<tr>
<th></th>
<th>ZDDP</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>P 2p3/2</td>
<td>Zn 2p3/2</td>
<td>S 2p3/2</td>
<td>O 1s</td>
<td>C 1s</td>
<td></td>
</tr>
<tr>
<td></td>
<td>P 2p3/2</td>
<td>Zn 2p3/2</td>
<td>S 2p3/2</td>
<td>O 1s</td>
<td>C 1s</td>
<td></td>
</tr>
<tr>
<td>Steel</td>
<td>133.5 (335)</td>
<td>1022.2 (9480)</td>
<td>161.8 (225)</td>
<td>530.3 (3130)</td>
<td>531.7 (2290)</td>
<td>533.3 (683)</td>
</tr>
<tr>
<td>CrN</td>
<td>133.3 (3865)</td>
<td>1022.4 (37694)</td>
<td>162.1 (1426)</td>
<td>531.6 (20245)</td>
<td>533.3 (5870)</td>
<td>284.9 (2931)</td>
</tr>
</tbody>
</table>

phosphate film has formed on both steel and CrN coating. On the steel sample, an oxide is also detected from the oxygen peak with binding energy of 530.3 eV\(^6\),\(^13\). The binding energy of the S 2p peak suggests formation of a sulphide film\(^9\)\(^,\)\(^13\) on both steel and the CrN coating while the exact nature of these sulphides cannot be deduced from XPS analyses.

ZDDP lubricant was shown to form similar species on CrN as on steel. Detailed analyses of the Cr 2p XPS peak from the CrN coating prior to testing (not shown) showed a peak with binding energy of 575.5 eV which corresponds to CrN. The lack of an oxide peak in conjunction with the lack of Cr peak at 576.2 eV in the coating wear scar indicates clearly that no CrO\(_2\) is present in the wear scar. No interaction between Cr in the coating and the additive elements could be observed.

3.3.2. ZDDP/MoDTC tribofilm formation on steel and CrN materials

Table 5 shows the binding energies of the peaks obtained from analysing wear scars produced by testing steel and CrN using the ZDDP/MoDTC lubricant. Similarly to when ZDDP lubricant was used, the highest concentration of P, S and Zn is obtained on the wear scar in the CrN coating. From Table 5 it can be observed that Zn and P binding energies, around 1022.5 ±0.3 eV and 133.3 ±0.3 eV respectively, illustrate that tribofilms formed on steel and the CrN coating are similar and correspond to a phosphate glass\(^9\). AFM images of the ZDDP/MoDTC tribofilms have shown a much more uniform structure compared to the tribofilms formed from ZDDP lubricant\(^2\). This structure with further etching was removed, after which the oxide peak was detected\(^2\), S 2p binding energy corresponds to the sulphide region\(^9\).

Mo 3d peak, as shown in Figure 4, was obtained from the wear scars on both materials clearly indicating formation of a tribofilm from the friction modifier. The XPS curve fitting shows formation of Mo (IV) (~229 eV) and Mo (VI) (232.2 eV), peaks which

Table 5 The binding energies of XPS peaks (in eV) detected on the ZDDP/MoDTC tribofilm formed on steel and CrN coating. Numbers in brackets show the peak area

<table>
<thead>
<tr>
<th></th>
<th>ZDDP/MoDTC</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>P 2p3/2</td>
<td>Zn 2p3/2</td>
<td>S 2p3/2</td>
<td>O 1s</td>
<td>C 1s</td>
<td></td>
</tr>
<tr>
<td>Steel</td>
<td>133.2 (420)</td>
<td>1022.8 (4665)</td>
<td>162.4 (195)</td>
<td>530.1 (83)</td>
<td>531.2 (800)</td>
<td>532.3 (2370)</td>
</tr>
<tr>
<td>CrN</td>
<td>133.4 (1706)</td>
<td>1022.4 (18674)</td>
<td>162.1 (1670)</td>
<td>530.4 eV (1740)</td>
<td>531.7 eV (9640)</td>
<td>533.3 eV (1680)</td>
</tr>
</tbody>
</table>
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correspond to MoS$_2$ and MoO$_3$ species$^{9)}$. Formation of the MoS$_2$ is likely to be the main reason for the friction drop on both material couples$^{14-19)}$.

Formation of the Mo species on the wear scar when CrN coating is lubricated with a MoDTC-containing lubricant is also reported by Yajun et al$^{20}$ using Electron Dispersive X-ray spectroscopy. The drawback of EDX in identifying the Mo peak is that the energy of this peak overlaps with the energy of S, making the distinguishing between S and Mo difficult. In addition, EDX only gives elemental information from the wear scar. Surface analyses in the present work show clearly the formation of the MoS$_2$ and MoO$_3$ on both CrN and steel.

Tribofilms formed from lubricants containing both ZDDP and MoDTC in steel are reported to comprise a carbon-rich Zn/Mo phosphate film with dispersed MoS$_2$ sheets$^{2,21,22)}$. Table 5 and Figure 4 suggest that this lubricant forms a similar tribofilm on the CrN coating.

Figure 4  Mo 3d curve fitted XPS peaks obtained from ZDDP/MoDTC tribofilms formed on steel and CrN surface

3.4. Chemical nature of the wear scar after no additive replenishment

To understand the nature of the tribofilm once the additive replenishment has stopped, the tribofilms formed from ZDDP and ZDDP/MoDTC lubricants on both materials have been rubbed further for one hour in base oil. The wear scars were then analysed using XPS.

Table 6 shows that even after no ZDDP replenishment and after one hour rubbing, there was still phosphorus, sulphur and zinc detected on the steel and CrN wear scars. For steel samples, the high tenacity of the ZDDP tribofilm was observed in authors previous work$^{23)}$ while Miklozic et al$^{24}$ showed no ZDDP film thickness reduction when rubbing in base oil. It is evident that the $h_{\text{crepe}}$ of the ZDDP tribofilm formed on steel substrate is very low. This current study shows that in a similar manner to the steel wear scar, the phosphate tribofilm formed on the CrN coatings is not removed after 1 hour when rubbing in base oil. These results indicate that the removal rate of the ZDDP tribofilm ($h_{\text{crepe}}$) does not change significantly no matter if the substrate material is steel or CrN coating.

Table 6 The binding energies of XPS peaks (in eV) detected on the ZDDP tribofilm formed on steel and CrN coating and then rubbed in BO for 1 hour. Numbers in brackets show the peak area

<table>
<thead>
<tr>
<th>ZDDP changed to BO for 1h</th>
<th>P 2p3/2</th>
<th>Zn 2p3/2</th>
<th>S 2p3/2</th>
<th>O 1s</th>
<th>C 1s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>133.0</td>
<td>1021.6</td>
<td>161.7</td>
<td>530.1 (6379)</td>
<td>284.8 (5128)</td>
</tr>
<tr>
<td>CrN</td>
<td>133.7</td>
<td>1022.7</td>
<td>162.2</td>
<td>530.2 (100)</td>
<td>284.8 (3560)</td>
</tr>
</tbody>
</table>

As Table 6 indicates, the chemical nature of the species observed in the test after the ZDDP replenishment is stopped does not change significantly from the chemical nature of the initial tribofilm. After 1 hour rubbing in the base oil of the ZDDP tribofilm formed on both materials, the phosphates and sulphides could still be detected. The only obvious difference is the increase of the oxide peak (530.1 ±0.3 eV) in relation to the other peaks. Looking at the areas which the observed peaks cover (the data shown in Table 4 and Table 6 in brackets), it can be seen that in case of steel sample, further rubbing in the base oil results in a film richer in sulphur and oxides. ZDDP tribofilm is a layered structure with phosphates covering the sulphide and oxide film$^{25}$, so the wear scar enrichment in oxides and sulphides after rubbing in base oil could be due to the removal of upper layer of the tribofilm (phosphate species).

In the case of the ZDDP tribofilm formed in the CrN substrate, all tribofilm constituents (Zn, P and S) showed reduced concentration, indicating that the XPS layer analysed contains less phosphate and sulphides after rubbing the ZDDP tribofilm in base oil. It is obvious that phosphate layer film formed on CrN has high durability similarly to the phosphate layer formed on steel but it is not clear how the reduced tribofilm content on the near surface layer influences wear.

Similarly to the ZDDP tribofilm, Table 7 shows that phosphate film formed from the ZDDP/MoDTC lubricant is still detected on the wear scar even after 1
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Curve fitting of the Mo XPS curve, shown in Figure 5 for the peaks obtained in the wear scars produced following the rubbing of ZDDP/MoDTC tribofilm in BO, shows that Mo sulphide and Mo oxides are formed on both materials. A steady increase of friction with lubricant change, observed in Figure 3, and the XPS analyses of wear scars shown in Figure 5 indicate that Mo species have a certain durability. In the case of steel, previous work has shown that the level of friction reduction from the ZDDP/MoDTC lubricant depends on the Mo sulphide/Mo oxide ratio determined by near surface chemical analysis. Table 8 shows this ratio for the ZDDP/MoDTC tests done in this work and it is obvious that no MoDTC replenishment resulted in an increase of the Mo oxide peak, in relation to the Mo sulphide peak, indicated by their ratio. No difference was seen depending on the substrate material.

Table 7 The binding energies of the XPS peaks (in eV) detected on the ZDDP/MoDTC tribofilm formed on steel and CrN coating and then rubbed in BO for 1 hour. The numbers in brackets show the peak area.

<table>
<thead>
<tr>
<th>ZDDP/MoDTC changed to BO for 1h</th>
<th>P 2p3/2</th>
<th>Zn 2p3/2</th>
<th>S 2p3/2</th>
<th>O 1 s</th>
<th>C 1 s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>133.3 (690)</td>
<td>1022.2 (3784)</td>
<td>162.1 (480)</td>
<td>530.5 (2180)</td>
<td>284.8 (10050)</td>
</tr>
<tr>
<td>CrN</td>
<td>133.8 (821.7)</td>
<td>1022.8 (8400)</td>
<td>162.1 (490)</td>
<td>532 (5758)</td>
<td>284.8 (1350)</td>
</tr>
</tbody>
</table>

4. Conclusions

The current study has highlighted the importance of the tribofilm durability formed from ZDDP and MoDTC additives on both steel and CrN coating in maintaining good frictional properties even when there is no additive replenishment. The concise conclusions that can be made are:

1. A tribofilm with similar chemical nature is formed on CrN coating and steel lubricated by ZDDP-containing oil in boundary regime. On both materials, phosphate and sulphide species are formed.
2. Similarly to steel, use of the MoDTC additive results in friction reduction when the CrN coating is lubricated. Surface analyses have shown formation of MoS$_2$ and MoO$_3$ from the MoDTC additive.
3. Phosphate and sulphide tribofilms formed on both steel and CrN coating from the ZDDP oil show low removal rate. Even after one hour rubbing in oil with no additive replenishment the film was still on the wear scar.
4. In case of the ZDDP/MoDTC tribofilm, Mo sulphide is shown to have certain durability. No MoDTC additive replenishment led to increase of Mo oxide species on the wear scar and gradual loss of friction performance.

Table 8 Ratio between low friction material (Mo sulphide) and high friction material (Mo oxide) formed from the MoDTC additive.

<table>
<thead>
<tr>
<th></th>
<th>Mo sulphide/Mo oxide ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZDDP/MoDTC</td>
<td>After rubbing</td>
</tr>
<tr>
<td>ZDDP/MoDTC in BO</td>
<td></td>
</tr>
<tr>
<td>Steel</td>
<td>1.9</td>
</tr>
<tr>
<td>CrN</td>
<td>2.4</td>
</tr>
</tbody>
</table>

Figure 5 Mo 3d curve fitted XPS peaks obtained from ZDDP/MoDTC tribofilms formed on steel and CrN surface and then rubbed in base oil for 1 hour.
Future work will focus on developing the methodologies for quantifying the tribofilm durability as a function of loading conditions and substrate material.

5. References


