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Combining DLC, Shot Blasting, Chemical Dip and Nano Fullerene Surface Treatments to Reduce Wear and Friction When Used With Bio-lubricants in Automotive Contacts

Carrell, J.[1], Slatter, T.[1], Little, U.[2], Lewis, R.[1]
[1]The University of Sheffield, U.K., [2] South West College, U.K.

Abstract

The interaction of three bio-lubricant base oil candidates with seventeen combinations of surface treatment was studied, comparing wear scar volumes and coefficient of friction results. Substrates were initially ground, then a combination of superfinished, Dymon-iC™ DLC, an impact technique of ultra-fine shot blasting method doped with Tin and Molybdenum Disulfide, a calcium based chemical dip containing calcium sulfate and nano fullerene, were used.

DLC is well reported to reduce friction. Some reports suggest wear in coated contacts is independent of the type of lubricant used, whilst others report that bio-lubricants offer reduced friction and wear in combination with DLC. Shot blasting can also reduce wear and friction, due to the surface dimples acting as lubricant reservoirs, making hydrodynamic lubrication more likely. Previous work has also explored the performance of surface texturing in combination with coatings, some reporting higher friction when surface texturing and DLC is used. As a surface coating, fullerene has been shown to have significantly lower wear and friction than DLC coatings. The calcium based chemical treatment used has no published data.

A ball on flat reciprocating wear tester was used with bio-lubricant base oil candidates, jojoba and soybean oil, with a mineral base oil used for comparison. Wear scars were analysed using a scanning electron microscope.

Coefficient of friction results from testing with bio-lubricant base oil candidates' soybean and jojoba oil were lower than tests with mineral base oil. A hybridized coating combination of superfinish, diamond like carbon and chemical dip gave the highest wear protection for tests with the mineral base oil and bio-lubricant base oil candidate soybean oil. A hybridized coating combination of superfinish, impact technique and chemical dip gave highest wear protection when tested with bio-lubricant base oil candidate jojoba oil. Results showed no overall improvement in wear protection when substrates were processed with the impact technique. Superfinishing substrates improved the performance of both the chemical dip and DLC.

Introduction

Interest in bio-lubricants has increased as the need for lower toxicity lubricating fluids has developed. Bio-lubricants are generally derived from vegetable oils, their use is becoming more common in total and potential loss, applications, such as marine engines. The bio-lubricants market was estimated at over 630,000 tonnes in 2015 and is projected to grow to 1.12 million tonnes by 2024 [1]. This represents approximately a 2% share of the total lubricants market.

There are many drivers that may contribute to bio-lubricants increasing their market share, such as the demand for greater fuel efficiency, lower friction, increased wear protection and reduced environmental impact. It is estimated that 50% of all lubricants end up in the environment due to total loss systems or spillages [2] and it is increasingly important that these fluids are biodegradable and have low toxicity. In addition to the environmental benefits of bio-lubricants, they are reported to have lower wear rates [3], higher viscosity index, higher flash point, due to the higher molecular weight, and a narrow molecular weight distribution [4].

Direct frictional losses in automotive engines account for 28% of fuel consumption. Advanced coatings and surface texturing are seen as the major step in reducing these losses by up to 61% in the next 25 years. This would reduce fuel consumption by up to 385,000 million litres [5].

Surface treatments are well documented to reduce friction and wear in a variety of applications. Diamond like carbon (DLC) coatings in particular have become prevalent in the automotive industry to aid increases in efficiency. DLC coatings are generally considered inert [6], but research shows that there is an improvement in wear when lubricants with higher levels of polar and unsaturated groups, such as vegetable based oils, are used [7]. Studies have shown that, particularly in steel/DLC contacts, wear and friction are reduced when bio-lubricants are used. Mobarak and Chowdhury [8] found that with ball on flat tests, with DLC and Canola oil, wear was reduced by 22% in comparison to steel/steel equivalent tests. They suggested that the oil formed OH and CH groups with the surface of the DLC coating, these carboxyl and hydroxyl groups are known to aid the performance of bio-lubricants on steel surfaces. Most vegetable oils have these functional groups situated on the end of their long carbon chains and they give excellent adhesion properties on steel surfaces. DLC coatings are also thought to form hydroxyl groups in the presence of water, leading to the suggestion that DLC is hydrophilic and this also aids the friction and wear properties of the material [9]. Kalin and Vizintin [10] also made the link between increased polarity and saturation and decreasing wear loss in a proportional manner. For relatively inert materials such as DLC, Kalin and Vizintin concluded that as vegetable oils have large amounts of readily available polar elements, the likelihood of interaction is increased. They also observed lower wear in steel/DLC contacts, attributed to a thin layer of wear debris generated from the steel ball in a ball on flat contact, which "accommodated the contact stresses". This was only observed in tests with mineral based oil but tests were also carried out using sunflower oil. The same wear on the steel balls was not observed, probably due to the oil forming a tribo-chemical film on the ball.

Similarly to DLC coatings, surface texturing has become a popular method for improving tribological properties. Shot blasting is one such method and laser surface texturing is also used. Kovalchenko et. al. [11] studied the effects of laser surface texturing on lubrication regimes, with results showing that surface texturing expands the load and speed parameters within which hydrodynamic lubrication could be maintained. As a result friction coefficients (COF) were reduced, in comparison to un-textured surfaces, even when the surface roughness between the textured and un-textured surface was the same. The dimples created through surface texturing were also observed to reduce friction under boundary lubrication, with a lower area dimple density improving lubrication regime transitions. Pattersson and Jacobson [12] studied the effect of surface texturing applied to samples before coating in DLC. When comparing textured and un-textured surfaces in unlubricated sliding contacts, COF results were between 0.12-0.22 for textured DLC samples, in comparison to 0.07 for un-textured. This difference was attributed to the fact that DLC/DLC contacts create a sacrificial layer or tribo-film on the surfaces that reduces friction, the dimples in the textured contact are thought to have removed this layer. In boundary lubricated situations however, the textured surface with a DLC coating proved successful in reducing COF results to a very stable 0.05, with no high starting friction present. This research found that the nature of the surface texturing was important, tests were conducted with grooved textures of 5 and 20 μm. The tests carried out with 5 μm square depressions exhibited low COF values over 200,000 cycles. Etsion [13] also observed the same trend with surface texturing and no lubrication. Lubricated tests were also carried out, varying the amount of lubricant available in the contact. Results showed that for high viscosity lubricants, as lubrication decreased friction increased. Deeper dimple textured surfaces also produced higher friction values. They concluded that the distribution of lubricant across textured and un-textured surfaces is different. For the textured surface it is likely that the lubricant is concentrated in the dimples, whereas a more even distribution is likely on un-textured surfaces.

Nano fullerene is most commonly used as a lubricant additive, but there are some examples of its use as a surface coating. Nano fullerene coatings are comprised of spherical arrangements of carbon atoms. Alverdi, Hatto, Diaz and Csillag [14] developed a nanocomposite coating based on a fullerene like material. In ball on disc tests, performed in unlubricated conditions, and with COF values of around 0.06. Cylinder on flat fretting tests carried out under dry conditions also looked at DLC incorporated in to the fullerene based coating. This reduced COF results from 0.3 for pure DLC to 0.17, with wear rates three times lower for the fullerene based coating.

There is limited research into 'hybridized tribological coatings' and 'adaptive tribological coatings'. Hybridized coatings are either a composite structure or layers of varying solid lubricant, and when subjected to friction adaptive coatings react with surrounding compounds to change the properties of the solid lubricant layer [15]. These types of coating are particularly designed for applications with wide temperature ranges. Sliney [16] carried out work with chromium carbide, sliver, barium fluoride and calcium fluoride that proved to be effective at both low and high temperature ranges, but high surface roughness and dimensional tolerances meant that applications were limited. Calcium fluoride and silver solid lubricants were also shown to exhibit low COF results up to 500˚C by Pauleau, Juliet and Gras [17], when used in hybridized coatings. Calcium and tungsten sulfates in solid lubricant coatings in a composite or layered form, for high temperature applications, were studied by John, Prasad, Voevodin and Zabinski [18]. They concluded that calcium sulfate films exhibited lower COF results than calcium fluoride, but suffered more with the film wearing away. The performance of calcium sulfate can be attributed to the weak sulfur bonds that mean the structure has low shear forces between layers. There appears to be no work that looks at lubricated hybridized coatings.

Test Method

A high-speed linear reciprocating wear test rig (Plint/Phoenix Tribology TE-77) was used in a ball on flat configuration. Although ball on flat contacts are uncommon in automotive systems, this contact is a useful method for carrying out comparative tests and has shown to offer a satisfactory level of reproducibility for wear and friction [19]. Bio-lubricant base oil candidates jojoba and soybean oil were used, with a mineral base oil used for comparison (Shell HVI 60).

A 5 mm chrome steel ball, with a material designation equivalent to AISI 52100, was linearly reciprocated against a flat counterface. The surface treatments applied to the counterface varied, with details of the eighteen combinations given in Table 1. All counterfaces were EN9 steel, initially surface ground, then a varying combination of the following surface treatments was used;

- Superfinished (SF)
- DLC
- Impact Technique (IT) using a proprietary two stage process of a spherical ceramic media, followed by a solid lubrication media, where the solid lubricant includes tin and Molybdenum Disulphide
- A proprietary calcium based chemical dip (CD), X-ray fluorescence chemical analysis suggests this is a calcium sulfate
- Nano fullerene

The order of the surface treatments corresponds to the alphabetical order they are listed as in Table 1.

All contact surfaces were degreased before use and a fresh counterface surface was used for each test. 30ml of clean, untested oil was used for each test. Test conditions were similar to those used by Bahari, Lewis and Slatter [20], tests ran for 60 minutes, at a frequency of 10 Hz, a normal load of 40N produced a maximum contact pressure of 2.6 GPa and kept at 100˚C. A stroke length of 15 mm was used.

Table 1, Surface treatment combinations for samples

Sample Number	Superfinished	DLC	Impact Technique	Chemical dip	Fullerene
1		A			
2	A	B			
3			A		
4	A		B		
5	A	B		C	
6	A			B	
7		A		B	
8	A		B	C	
9				A	
10			A	B	
11			A		B
12					A
13	A	C		B	
14		B		A	
15		C	A	B	
16		B	A		
17	A	C	B		
18	A				

Measurement procedure

Once tests were completed, samples were degreased thoroughly. The samples were analysed using a scanning electron microscope (SEM) to assess the type of wear present. The width of the wear scars were then measured using an SEM in order to calculate the volume lost from the sample. Volume lost was calculated using the wear volume calculation for a ball-on-flat surface linearly reciprocating sliding wear described by Sharma et. al. [21], using case one, where it is assumed that the ball does not wear. It is felt that this was a reasonable assumption as the ball was hard enough to resist wear, with no visible signs of wear were present after tests. This is a proven method also used by Green, Lewis and Dwyer-Joyce [22]. The wear scar generated can be represented as the schematic shown in Figure

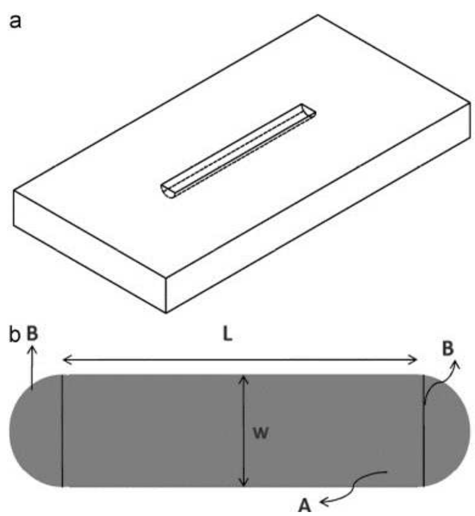

Figure 1, Wear scar volume approximation schematic [21]

1a. Figure 1b shows the top view, the middle section, Section A has cylindrical geometry, the two end sections, Sections B have geometry similar to a truncated part of a sphere.

The volume lost is calculated separately in each of Section A and B, as shown is Equations 1, 2 and 3, where r is the radius of the ball, w is the width of the wear scar and L the length of the wear scar. Figure 2 represents the ball, the area outlined by P, Q and R corresponds to the cross section area of section A from Figure 1b.

$$V_A = L\left[r^2 sin^{-1}\left(\frac{w}{2r}\right) - \frac{w}{2}\left(r^2 - \frac{w^2}{4}\right)^{\frac{1}{2}}\right] \quad (1)$$

$$V_B = \frac{\pi}{3}\left[2r^3 - 2r^2\left(r^2 - \frac{w^2}{4}\right)^{1/2} - \frac{w^2}{4}\left(r^2 - \frac{w^2}{4}\right)^{1/2}\right] \quad (2)$$

$$V_{total=V_A+V_B} \quad (3)$$

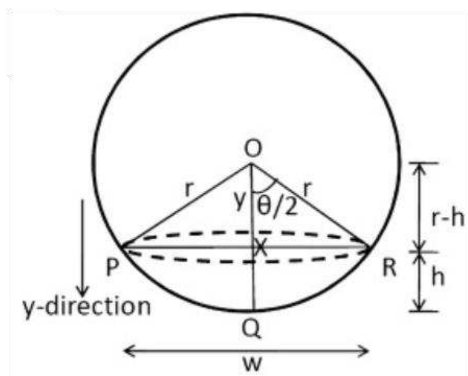

Figure 2, Geometry related to the ball and cross section of the wear scar [21]

Results

Figure 3 shows results for volume lost for all samples tested. Repeats on all samples were not possible due to limited sample availability.

COF results were consistent for each oil, irrespective of coating treatment, there were no notable variations in COF values, tests performed with the mineral base oil produced COF values of 0.1 ±0.01, jojoba oil had COF values of 0.07 ±0.01 and soybean 0.07 ±0.02. Lower COF in the bio-lubricant base oil candidates may reduce heat produced from friction. This in turn may give the benefit of reducing oxidation and temperature driven degradation in bio-lubricants, which is currently a major barrier to the technology being implemented on a large scale.

Basic micro hardness tests were carried out on coated samples, results were typical and no significant trends were found when comparing hardness and wear resistance.

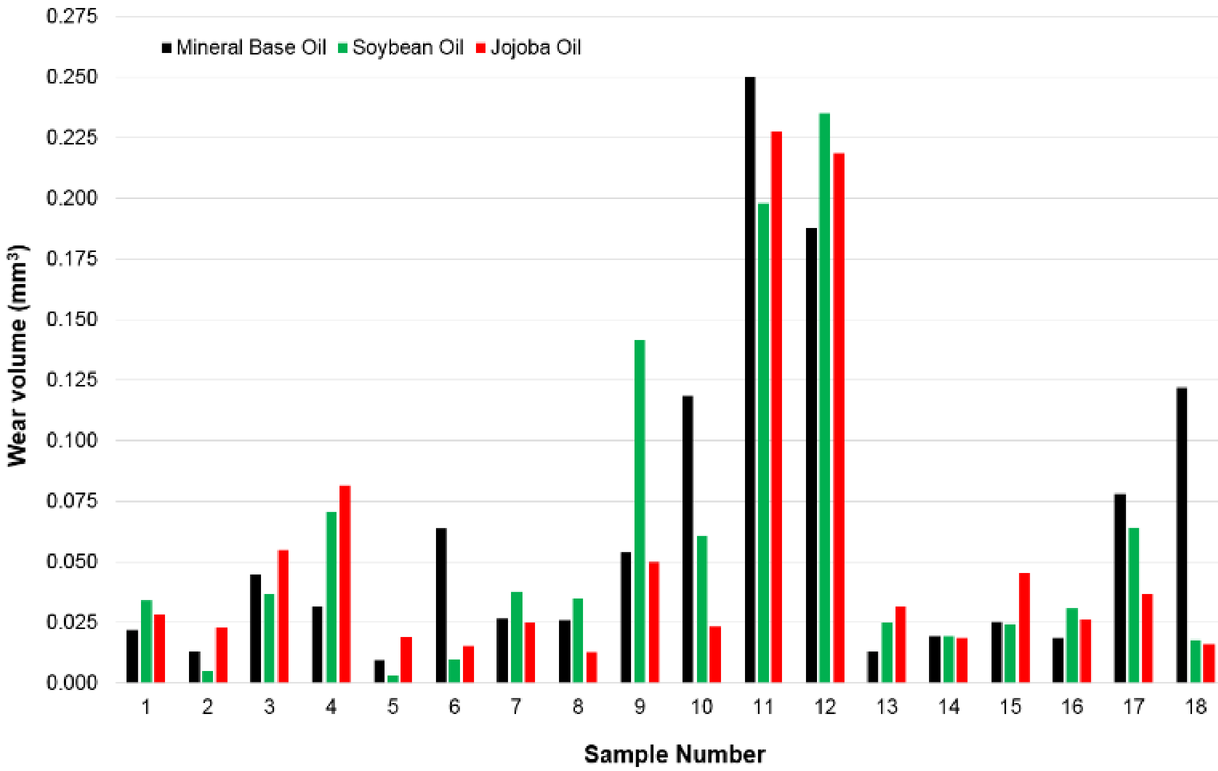

Figure 3, Volume losses for all base oil candidates and samples

Discussion

Combinations of DLC and chemical dip hybridized surface coatings

The samples with the least volume loss in the mineral base oil tests was sample 5, (SF, DLC and CD). Sample 7, as sample 5 but without SF, presented volume losses of three times higher than with SF. This is attributed to an increase in surface roughness causing increased wear, a well-documented trend [23]. SEM analysis showed clear machining marks on sample 7, parallel to wear scars, with accumulation of wear debris across the width. Wear scars on sample 5 were considerably narrower, with intermittently visible wear.

A similar trend was present in samples 5 and 7 when tested with jojoba and soybean oil, but with smaller differences between the volume loss from each sample. SEM analysis shown in Figure 4a, of sample 5, tested with soybean oil, showed very little wear present. When comparing this to tests carried out with mineral base oil in Figure 4b, it is clear there is a noticeable difference in the base oil candidates' ability to reduce wear. CD may act as a sacrificial solid lubricant layer that reduces wear in the initial stages of testing, the performance of calcium sulfate is attributed to the low shear forces between layers [18], as discussed previously.

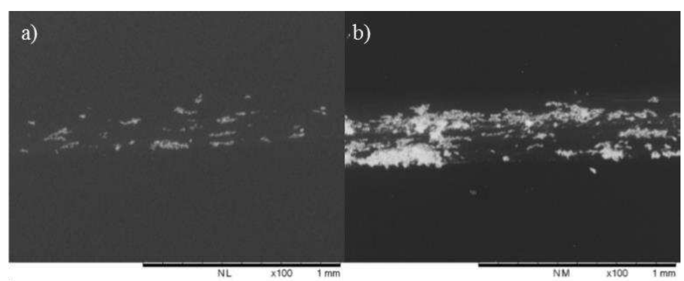

Figure 4, a) Sample 5, tested with soybean oil and b) mineral oil

The smaller difference in wear with tests carried out with the bio-lubricant base oil candidates could be linked to the greater availability of polar elements in the vegetable oils to form tribofilms, as discussed by Mobarack and Chowdhury [8] Kalin and Vizinlin [10]. The variation between the viscosities of the oils tested is not thought to be an influencing factor, soybean oil, with the highest viscosity, 32.6 cSt [24], does not consistently perform better than jojoba oil and mineral oil, with lower viscosities of 24.9 cSt [25] and 23 cSt [26] respectively. Literature shows that compared with mineral oil, vegetable oils have the ability to move the Stribeck curve downwards, meaning that they cause lower COF for shorter periods in boundary and mixed lubrication regimes, leading to earlier film formation [27]. This may contribute to the improved wear protection with vegetable oils on some samples.

Increased grooves due to machine marks left from sample preparation had a negative effect on wear protection when used in combination with DLC. This may be due to the tribofilm formed by DLC being removed by the 'shape' edges of the machine marks, as previously discussed. This shows that in this surface treatment combination, SF has an important effect on overall wear results.

When the coating order is changed to CD then DLC, sample 14, which did not have SF, had volume losses 1.5 times higher (0.013 mm^3) than sample 13, which had SF. This result is the same as for the SF DLC sample, sample 2, showing the CD has no positive effect under DLC. CD under DLC may affect the adhesion properties of DLC, which would account for the higher volume losses present on the wear scar. A similar pattern of wear was also apparent for samples tested with jojoba and soybean oil, tests with sample 2 performed better (wearing less) than tests with sample 13.

When looking at CD surface treatments alone on a SG substrate tested with mineral base oil, sample 9 showed slightly lower wear than the SF sample 6, at 0.0536 mm^3 volume loss, compared with 0.0635 mm^3. Samples tested with soybean and jojoba oil however exhibit the opposite trend, SF, CD samples have less wear than samples that are SG and CD. Figure 5a shows an image of the wear scar from sample 6, 5b shows the wear scar from test with sample 9, both tested with soybean oil. The final surface treatment process is identical (CD), but the finish and resulting wear protection were different, this is attributed to a stable tribofilm formation in sample 6, as well as increased asperity removal from the rougher surface finish in sample 9. Sample 6 tested with soybean oil, showed very little change in the surface composition in the wear scar area compared with the unworn areas.

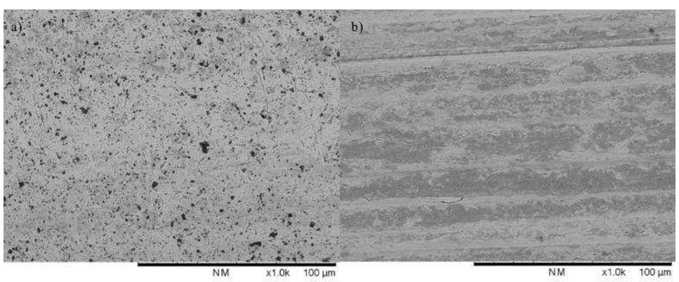

Figure 5, Sample 6, tested with soybean oil, and 3b) sample 9 tested with soybean oil

Comparing Figures 4 and 5, SEM images with sample 5 in Figure 4 appear more characteristic of a DLC coated sample with a uniform dark surface finish visible under the SEM, such as sample 2, even though the outer surface treatment is CD. CD samples, such as samples 6 and 9 in Figure 5, look lighter under SEM imaging with a visible surface finish. Further analysis is needed to assess the surface composition post testing. CD may be a sacrificial layer in the case of sample 5. When assessing the samples by eye, samples that are coated in DLC and CD have a duller finish than samples coated in DLC alone, indicating that overall there is a CD layer present.

When the coating order is changed from DLC followed by CD in samples 5 and 7, to CD followed by DLC in samples 13 and 14, SF samples show lower wear compared to SG samples. This coating combination is least sensitive to initial substrate surface finish, with little difference between wear volume lost with SF and SG samples. Wear scars on sample 13 (SF, CD, DLC), tested with jojoba oil were larger than those arising from the mineral or soybean oil tests. Figure 6a shows the wear scar produced with jojoba oil. When comparing this with an untested sample, shown in Figure 6b, it appears that the DLC layer has been removed and a surface characteristic of CD is visible due to the lighter image produced in the wear scar region. This layer may still offer wear protection as samples that were just coated in CD, such as sample 7, still offered improved wear protection in comparison to the uncoated sample. The wear debris caused by the removal of this layer may accelerate wear by acting as a third body in the contact. This removal of the DLC layer also indicates that CD may hamper the adhesion of DLC.

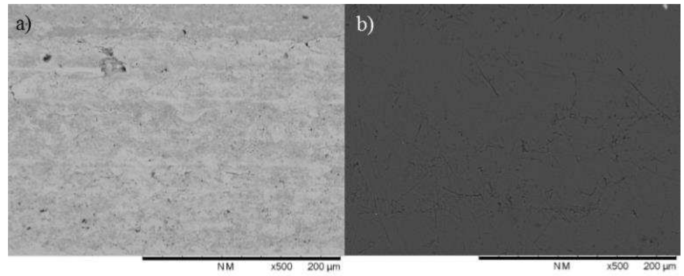

Figure 6, Sample 13 wear scar region tested with jojoba oil and 4b) Sample 13 before testing

Effects of Impact Technique

The effect of IT in various surface treatment combinations was also observed. Figure 7 shows the non-uniform dimple effect produced through IT.

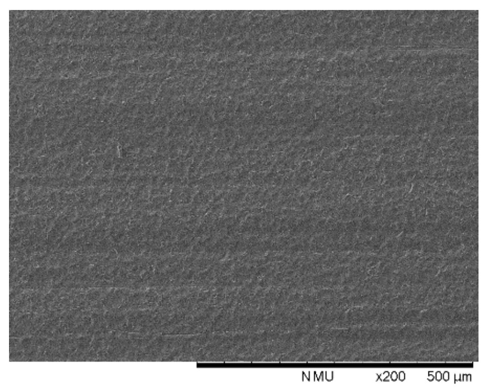

Figure 7, Impact Technique, sample 3

For tests with base oil candidate soybean oil, higher volume losses were observed when IT was combined with CD (sample 8 and 10). Sample 6 (SF, CD) produced volume losses of 0.0095 mm^3, while sample 8 and 10 volume losses were 0.0347 and 0.0608 mm^3 respectively, as shown in Figure 8.

In tests with soybean oil, the introduction of surface texturing had a negative effect. Particularly with samples 3 and 4, when combined with CD the effects of IT are less noticeable. The surface characteristics of the wear scar region are also noticeably different, as shown in Figure 9. If sample 10 is compared with sample 9 (SG, CD shown in Figure 5b, it appears that CD adheres and coats rougher surfaces differently to SF surfaces, such as with sample 6. Sample 6 appears to have an even coating of CD with small pockets (small dark regions) remaining in the wear scar region, where samples 9 and 10 have larger areas of darker material (indicating a variation in the material) in the wear scar region that could be due to the removal of

CD during testing, but could also be due to a less stable tribofilm formation on rougher surfaces due to higher contact pressures.

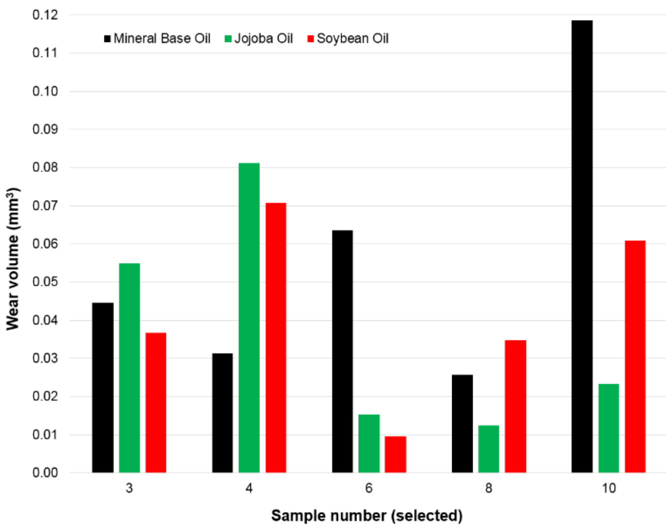

Figure 8, volume losses for sample with varying combinations of Impact Technique

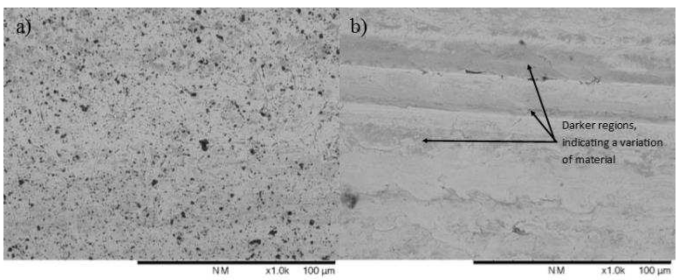

Figure 10, sample 6, and 8b) sample 10 tested with bio-lubricant base oil candidate soybean oil

Tests carried out with jojoba and mineral oil showed slightly lower volume losses for sample 8 than 6. Sample 8 presented the lowest volume losses out of all the coating combinations tested with jojoba oil. The presence of the solid lubricants in IT and particularly CD may have reacted with jojoba oil to form more stable tribo films, in a similar reaction to that discussed by Mobarack and Chowdhury [8], with carboxyl and hydroxyl groups forming on the surface of the samples, therefore reducing wear. Jojoba oil is a unique vegetable oil in that it is made up of a mix of long chain monhydric alcohol esters and carboxylic acids, as opposed to the triglyceride structure mostly seen in vegetable oils [28]. With large amounts of carboxyl groups present in the oil, there is an increased likelihood that they will react with contact surfaces. Figure 8 shows that bio-lubricant base oil candidate, jojoba oil demonstrates improved wear protection capabilities when IT is combined with CD, as sample 8 (SF, IT, CD) has lower volume losses than sample 6 (SF, CD).

When comparing IT with samples coated in DLC, Figure 10 shows little difference with sample 14 (SG CD DLC), compared with sample 15 (SG IT CD DLC), demonstrating that the addition of IT under CD and DLC makes no difference in terms of wear protection. Comparing samples 14 and 15 to sample 10 (SG IT CD), this also shows the addition of DLC in samples 14 and 15 improves wear protection considerably.

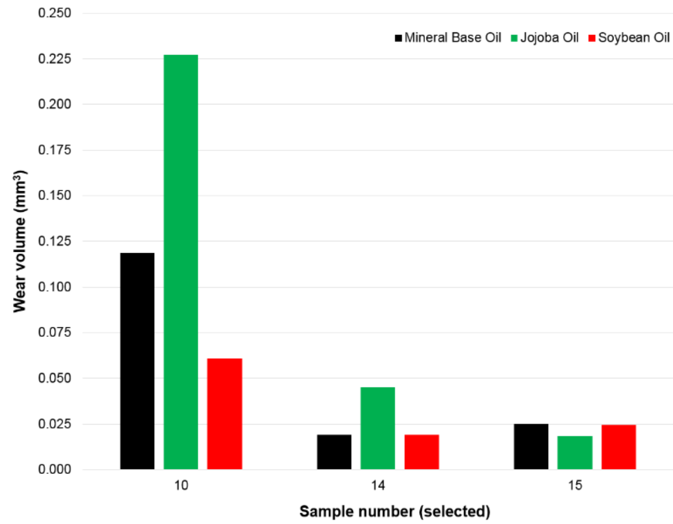

Figure 9, volume losses for samples with Impact Technique, Chemical dip and DLC

Figure 11a shows that for sample 8 IT has a dominant effect on the surface finish under CD, as with sample 15 in Figure 11b even under 2 layers of additional coating.

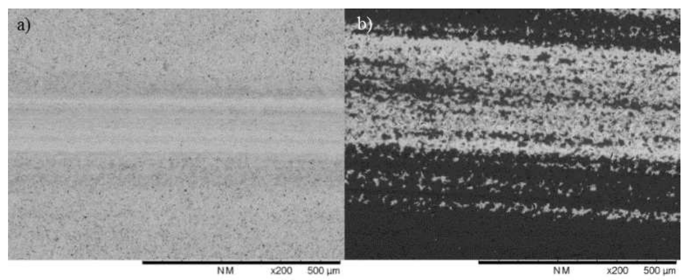

Figure 11, Figure 10a), Sample 8, and 10b) sample 15, both tested with jojoba oil

Nano fullerene

Samples coated in nano fullerene performed poorly. The resulting wear volume losses were considerably larger than any other coated sample, or reference tests carried out with SG or SF samples, with volume losses in the range of 0.1875 to 0.2503 $mm^{3.}$ For nano fullerene samples, SG samples were in the range of 0.0317 to 0.0467 mm^3 and SF samples 0.015 to 0.1217 mm^3. The nano fullerene coating did not adhere well to the substrate using a dipped method of application, untested samples had a 'flaky' and patchy finish. After testing, in the contact region the coating was completely removed, the coating surrounding the contact region showed flaking and blistering. The high wear for these samples is attributed to three body abrasive wear, with the coating being easily removed at the beginning of the tests and then getting entrained in the contact area and increasing wear. Further work is required to improve the application method for this sample, before any conclusions can be made on its ability to reduce friction and wear.

Conclusions

The use of hybridized coatings in combination with bio-lubricant base oil candidate soybean oil have been shown to be particularly effective at reducing wear, most notably with combinations of the chemical dip surface treatment and DLC. A surface treatment combination of superfinish, DLC and chemical dip resulted in the highest wear protection for tests performed with the mineral base oil and bio-lubricant base oil candidate soybean oil. The solid lubricant properties for calcium sulfate, a component of the chemical dip treatment is thought to contribute to the improved wear protection of this coating.

Tests performed with bio-lubricant base oil candidate jojoba oil demonstrated the highest wear protection with a surface treatment combination of superfinish, impact technique and chemical dip. It is thought that jojoba oil, made up of large amounts of highly polar carboxyl function groups, was able to react with the solid lubricants present in the impact technique and chemical dip, to provide a stable tribofilm. Four of the best five performing coating combinations tested with jojoba oil contained the chemical dip surface treatment in either the outer layer or sub layer of the hybridized coating. Tests performed with nano fullerene were unsuccessful due to poor adhesion of the surface coating to the substrate material.

Further work is required to assess the stability of the various layers, in various combinations after tests have been carried out, in order to fully determine which layers influenced performance most. The use of hybridized coatings in combination with bio-lubricants could be beneficial in increasing the use of bio-lubricants in automotive engines. Bio-lubricant base oil candidates have been shown to improve the wear protection, as well as offer lower coefficient of friction results compared with mineral base oil. Lower coefficient of friction results may also aid in reducing temperature driven degradation of the oils, which is currently a major barrier to their implementation.

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Contact Information

J.Carrell, Mechanical Engineering, The Portobello Centre, The University of Sheffield, Congress Street Sheffield, S14ET
j.carrell@sheffield.ac.uk,

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