Tribological characterisation and surface analysis of diesel lubricated sliding contacts

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Zusammenfassung

Mittels standardisiertem (ISO12156) Modelltribometer HFRR (High Frequency Reciprocating Rig.) wird die Dieselschmierfähigkeit in experimentellen Untersuchungen anhand der geometrischen Vermessung der Verschleißkalotte beschrieben. Liegt der korrigierte Verschleißdurchmesser der Kugel im Modelltest (WS1,4) unter dem Grenzwert von 460 µm, so erfüllt der Dieselkraftstoff diesbezüglich die Norm EN 590 [1]. Die Schmierfähigkeit des Dieselkraftstoffes für die tribologischen Kontakte in Dieseleinspritzssystemen gilt dann als ausreichend.

Im Rahmen der hier vorgestellten Arbeit wurde ein reiner Dieselkraftstoff ohne Additiv im Vergleich mit Dieselkraftstoff und unterschiedlichen Klassen von Schmierfähigkeitsadditiven sowie einer Beimengung von Biodiesel tribologisch im HFRR unter definierten Beanspruchungsbedingungen charakterisiert. Als Ergebnis wurden die Dieselschmierfähigkeit (lubricity) und die Unterschiede der tribologisch beanspruchten Wirkflächen dargestellt und diskutiert. Die Untersuchungen zeigten, dass neben den Konzentrationen der Additivgehalte, der Testdauer auch die Kraftstofftemperatur das Verschleißverhalten beeinflussen. Die Messung der Verschleißvolumina beider Testkörper (Kugel und Platte) ergab zusätzliche Informationen aus dem Test. Dadurch war, im Vergleich zur genormten Methode, eine bessere Differenzierung der verschiedenen Dieselkraftstoffe möglich. Die erweiterte Auswertung der Verschleißmarken hilft, die unterschiedlichen tribologischen Eigenschaften der verschiedenen Dieselkraftstoffe besser zu verstehen.

Keywords: HFRR, reciprocating sliding, fuel, lubricity additive, wear volume

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Introduction

Tribologically highly stressed tribocontacts which are only lubricated by diesel fuel (EN 590 [1]) appear in every modern diesel automobile. For the determination of diesel fuel lubricity the HFRR was implemented as a short-term standard test method. Since the introduction of low sulphur diesel fuels, diesel fuel lubricity and additive behaviour have been object of a large number of investigations. Lacey and Shaver studied the wear mechanisms with HFRR [2]. Wear volume values were calculated under the assumption of a flat wear scar area on the ball. Wear scar depth and wear scar diameter on the ball were tried to be correlated. The researchers concluded that for most fuels the wear scar diameter (WSD) on the ball is an indirect measure of wear occurring on the disc. The direct measurement of the wear volume as presented in this paper offers the possibility to give evidence to a correlation between wear volume and WSD.

Acids and esters widely used as lubricity additives are characterized by different adsorption strengths on metallic surfaces. Comparing acids, alcohols and esters, absorption strength decreases in the following order: acids > alcohols > esters [3]. The experiments described in this paper should clarify whether the HFRR method is capable to display different behaviour of the tested additives.

The addition of 1 to 2 vol% biodiesel to low sulphur petrodiesel fuel enhances the lubricity significantly according to tests with the standardized HFRR method [4]. These findings are dated back to the presence of free fatty acids and monoacylglycerol contaminants present in biodiesel. Biodiesel from several sources comprising rape seed oil, sunflower oil, corn oil, olive oil and used fried oil were also tested for their lubricating properties [5]. Anastopoulos et al. found a decrease of the wear scar diameter on the test ball tending to stabilize at concentrations higher than 1 vol%, thereby approaching asymptotically a constant WSD value in the range of 200 to 260 µm.

Motivation

Due to the desulphurisation process, the diesel fuel lubricity can reach a level of non acceptable wear taking place in fuel injection components. Hence, the now well established standardized testing method ISO 12156 [6] was implemented to assign a lubricity value (WS1,4) to diesel fuel. Threshold values have been defined in most relevant countries. The WS1,4 should correlate with wear found in diesel fuel injection component tests. The differentiation of good and poor lubricating diesel fuels is possible with this method. Despite this achievement the WS1,4 does not provide sufficient information in case of critical markets and diesel fuels. Possible approaches for improvements are disclosed in this article. The HFRR method is kept as intended, but complemented by additional evaluation methods, e.g. wear volume measurements and various surface analytical methods for a better understanding of the tribological processes happening in the contact zone.

Abstract

This paper presents the results of an investigation concerning diesel lubricated contacts. With the High Frequency Reciprocating Rig (HFRR) and the standardized model test (ISO 12156) the lubricity of diesel fuel is determinated. The tribological results of the model test should correlate with the wear of tribocontacts in diesel fuel injection equipment. The corrected wear scar diameter WS1,4 is a measure of the lubricity of the diesel fuel. A raw diesel fuel with no additives was compared to diesel fuels with various amounts and kinds of additives and biodiesel. Test parameters in particular temperature and time were also varied. More valuable data from the test specimens were gained by wear volume measurements on both specimen, ball and disc. This allowed a more detailed differentiation of the characterized diesel fuels, in comparison with the WS1,4 - value. Moreover, the shape of the wear scars, helped to understand the vast differences in the tribological behavior of various diesel fuels.

1 Experimental setup

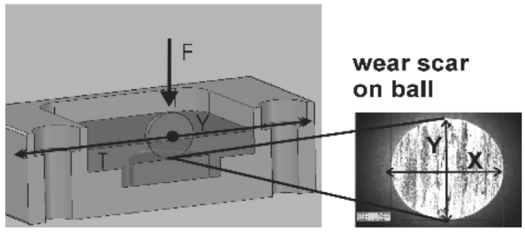
Tested fuel with different admixtures and materials

A raw diesel fuel (DF) without any additives was used as main component in this paper. Selected additives were added to DF, as described in **Table 1**. The lubricity additives

Component	Purpose	Concentration
Diesel Fuel (DF)	Main component	
Neutralized Acid Acid Ester	Lubricity Additive Lubricity Additive Lubricity Additive	25, 50, 100, 150, 200 ppm
FAME Rape Seed Methyl Ester (RME)	Lubricity Admixture, 0.5, 1, 2, 4.7, 10 vol %	5000 to 100.000 ppm
Performance Package (PP)	Detergent, Demulgator, Foam and Corrosion Inhibitor	75 ppm
Conductivity Improver	Antistatic Additive	5 ppm

Table 1: *Selected base fuel, additives and admixtures for the HFRR tests*

a) HFRR Concept



b) Test Parameter	Value
Normal load	2 ± 0,01 N
Stroke length	1 ± 0,02 mm
Frequency	50 ± 1 Hz
Test duration	75 ± 1 min
Silding distance total	~ 450 m
Fluid Volume	2 ± 0,2 ml
Fluid Temperature	60 ± 2 °C
Reservoir surface area	600 ± 100 mm²

Figure 1: *HFRR method, a: Concept, b) Parameters*

were common ones, 100 ppm (mg/kg) reflect a realistic amount admixed in field applications. Concentrations from 25 to 200 ppm were tested to evaluate treat rate effects. For some experiments, 4.7 vol% of biodiesel based on rape seed methyl ester (DIN EN 14214, FAME) were added according to EN 590 standard instead of above mentioned lubricity additives. The studies were also extended to lower and higher concentrations than 4.7 vol% to gain data over a wider range. The influence of the performance package on the HFRR results in connection with lubricity additives and biodiesel was evaluated, applying a typical amount of 75 ppm of the performance package (PP). The used sample balls (Ø 6 mm, R_a < 0.05 µm) and discs (Ø 10 mm x 3 mm, R_a < 0.02 µm) were made of 100Cr6 steel according to the HFRR standard, with balls having a considerably higher hardness (~640 to 900 HV) than the discs (190 to 210 HV). HFRR tests were carried out at Robert Bosch GmbH, department CR/ARU2 in Stuttgart (Schillerhöhe) and Drive Technology Center in Schwechat of SGS Austria Controll-Co. GesmbH.

1.2 Tribological test method

The HFRR (**figure 1**, **a** and **b**, [2]) is a ball on disc tribometer with a reciprocating relative movement of ball and disc. As shown in **figure 1 a**, the tribocontact is fully immersed in the diesel fuel. After the tribotest, the wear scar on the ball is measured by means of an optical microscope in sliding direction (Y) – and perpendicular to sliding direction (X). The MWSD value (mean wear scar diameter of X and Y direction) is then corrected using the water vapour pressure (AVP) to give the final value for diesel lubricity, the so-called WS1,4. WS1,4 values as well as the wear volumes published in this paper are with few exceptions the mean values of 2 tests. The AVP dependence of the MWSD is expressed with the Humidity Correction Factor (HCF). This factor must be determined for every tested fluid, what may lead in extensive test series. Alternatively, a factor for unknown fuels as mentioned in the standard can be used (60 µm/kPa). All tests were performed according to ISO 12156.

1.3 Wear volume analysis

A confocal white light microscope µsurf® (NanoFocus AG, Germany) was applied for surface analysis the acquired data of which were used for the wear volume determination of both balls and discs (**figure 2 a**). The measuring system consists of a white light source and a CCD camera mounted behind a Nipkow disc.

Here, the reflected light from the surface reaches a maximum intensity detected by the CCD when the investigated spot on the surface is in focus. This point is pictured. The x-y-scan was provided by means of the Nipkow disc and the z-scan is performed by a piezo actuator.

Balls and discs have to be cleaned before measured with the confocal white light microscope. The wear volume was calculated from the µsurf® data by the use of a Matlab based program developed at AC²T research GmbH. For the balls, an ideal spherical surface is defined as „reference surface“. In order to obtain this reference surface, the approximate non-worn region of the ball is selected interactively (as shown in **figure 2 b** black shaded region) and the reference surface is fitted to this region. In the second step the worn region is selected interactively (white shaded region). Finally, the program calculates the wear volume from the difference between the reference surface and the measured surface in the white shaded region. With this software, it is even possible to determine the wear volume (W_V) of small wear scars. For the discs, an ideal plane is defined as reference surface. Again, the non-worn part of the surface is selected interactively and the wear volume is calculated as difference between reference and measured surface.

2 Results and discussion

2.1 Variation of lubricity additives and the use of biodiesel in DF

All test parameters were according to the HFRR method, the wear evaluation was performed using both microscopical method as described in the standard and volumetrical method as developed at AC²T.

The effect on lubricity by adding 100 ppm lubricity additive or 4.7 vol% biodiesel to the

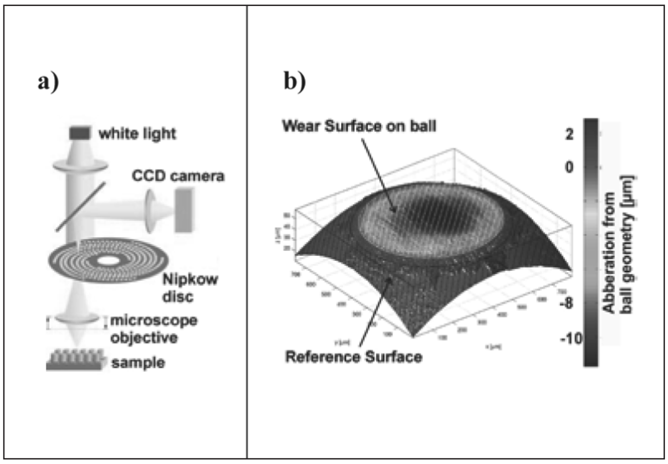


Figure 2: *Wear scar analysis: a) Confocal white light microscope, b) Wear scar region on ball*

raw diesel fuel (DF) was tested. Furthermore the influence of the use of 75 ppm performance package (PP) was investigated.

The standard evaluation (**figure 3**) clearly showed that the WS1,4 is reduced from approximately 600 to 400 µm in the case of 100 ppm lubricity additive. The addition of 4.7 vol% biodiesel resulted in a more pronounced decrease of WS1,4 to approximately 200 µm. The comparison of DF mixtures with or without PP showed no significant difference. Furthermore all tests with the 3 lubricity additives (100 ppm) added to DF gave no significant differences in WS1,4. Thus a differentiation between the 3 lubricity additives was not possible when using WS1,4.

There might be several explanations for this finding:

– No difference in efficiency between acid, neutralized acid or ester as a lubricity additive in a concentration of 100 ppm (same „quality“ in terms of lubricity except 4.7 vol% biodiesel)

– Wear determination based on MWSD is insufficient

– Wear on the disc is not yet considered

An irregular three dimensional material abrasion on ball and disc is most probably. For this reason, our approach is based on the wear volume determination of both ball and disc as a measure for lubricity.

These results are depicted in **figure 4**. The small bars represent the wear volume values (W_V) of the balls. Again, the addition of performance package has no effect concerning ball W_V (same conclusion from WS1,4).

The same applies for the discs (high bars).

The comparison of the ball W_V shows that the addition of 100 ppm acid to DF resulted in a W_V of ~8 x 10^4 µm^3, neutralized acid leads to a value of ~1.2 x 10^5 µm^3 and ester followed with a W_V of ~3.1 x 10^5 µm^3. Balls run with pure DF have a W_V of ~1.4 x 10^6 µm^3. Summing up, the method based on W_V determination on the ball shows a trend with ester causing more ball wear than the other two lubricity additives. The results also show that the W_V of the ball cannot be simply calculated from the MWSD value, because a two dimensional wear surface does not appear.

The W_V of the discs behaved as follows:

DF containing 100 ppm ester gave the highest W_V values for the balls but the lowest for the discs (1.4 x 10^6 µm^3). DF containing neutralized acid resulted in a low value for the balls but in the highest for the discs (2.1 x 10^6 µm^3). The reason for this result might be found in the different material properties of ball and disc. The disc is relatively soft in comparison to the hardened ball. Furthermore the ball wear scar is in steady contact with the disc, whereas the disc wear scar

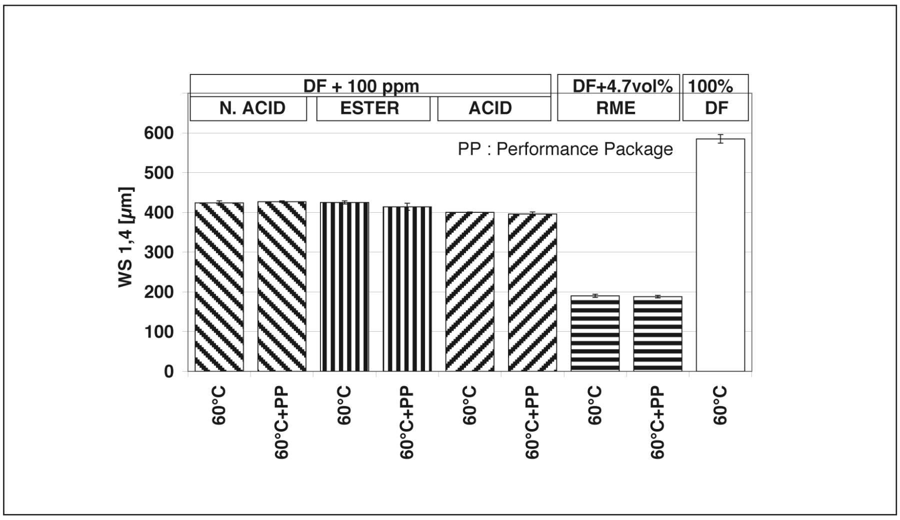


__Figure 3:__ Results of standard lubricity tests of diesel fuel with various additives, (60 °C, 75 min)

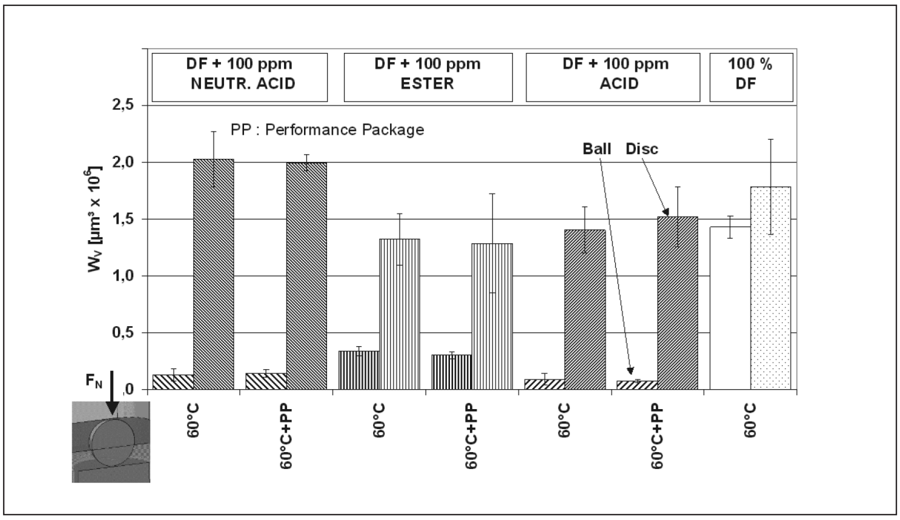


__Figure 4:__ Wear volumes (W_V)for balls and discs at standard test conditions (60 °C, 75 min)

is not subjected to wear the whole time. It can be stated that the additive with low ball wear tends to cause high disc wear and vice versa. Moreover, the results indicate that the wear on the disc can increase at small additive amounts. This effect was investigated in detail and is discussed in 2.2.

Biodiesel (RME) can be used alternatively to lubricity additives. Currently, 4.7 vol% biodiesel in diesel fuel is the standard concentration used in the field. The W_V of the ball with 344 µm^3 and disc with 1.2 x 10^5 µm^3 is much smaller than of DF. Therefore, it is not displayed in **figure 4**. The results of the tests with different concentrations of biodiesel are discussed in 2.2.

Figure 5 and **figure 6** show the ball and disc wear profiles gained by µsurf® from HFRR

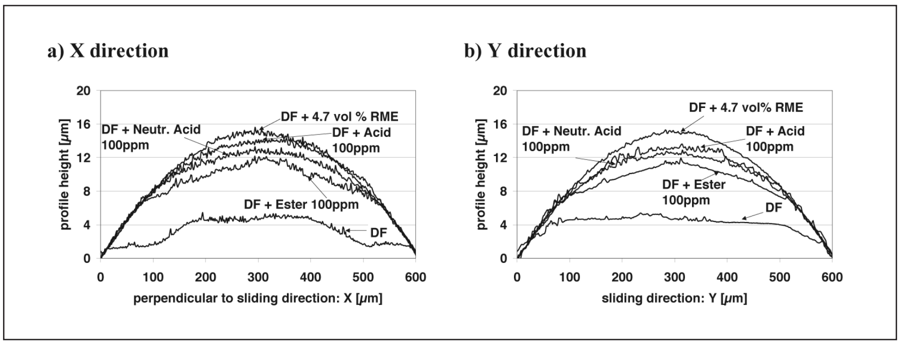


__Figure 5:__ Ball wear profiles for DF mixtures in figure 4 including DF with 4.7 vol% of RME, standard test conditions (60 °C, 75 min); a: X direction; b: Y direction

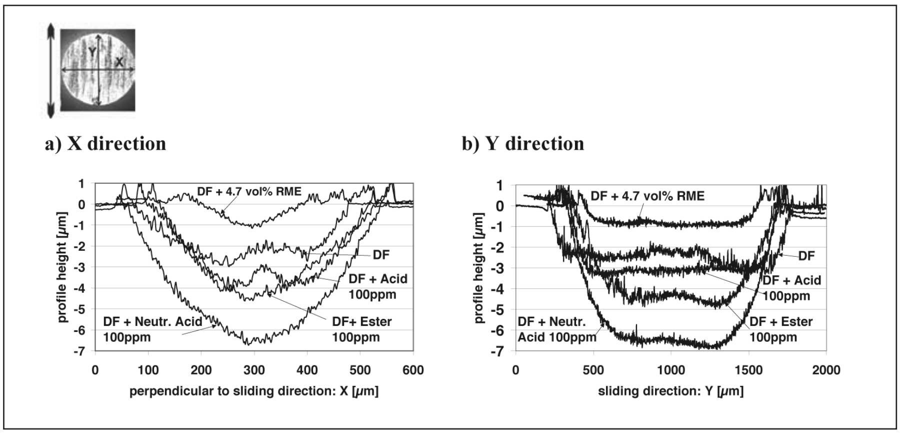


Figure 6: Disc wear profiles for DF mixtures in figure 4 including DF with 4.7 vol% of RME, standard test conditions (60 °C, 75 min); a: X direction; b: Y direction

tests already described in **figure 3** and **figure 4** (WS1,4).

These figures are related to individual measurements; hence do not depict mean values. The surface topographies were measured close to the middle of the wear scars in moving direction and perpendicular to the moving direction.

As shown in **figure 5** (**a** and **b**) the lowest wear on the balls was found for 4.7 vol% of biodiesel. The wear scar is hardly visible. The addition of 100 ppm acid or neutralized acid, respectively, resulted in a convex wear shape on the ball. The wear with neutralized acid is higher, but the wear scar has nearly the same diameter. This was observed for both X and Y direction. DF with 100 ppm of ester leads to even higher wear. The ball profile has more a „roof“ shape in both directions, but with a smoother curve in Y direction (**figure 5 b**). When diesel fuel without additives is tested, the wear profiles in X and Y direction differ clearly from each other. In Y direction (**figure 5 b**) a „plateau“ appears with chamfers on the sides. The X profile (**figure 5 a**) consists of a smaller plateau and a declivity that is followed by a flat. This flat slides on the disc surface. The profiles are relatively symmetric. A plateau with the diameter of the WSD was not found in any investigated case. Therefore, the W_V cannot be calculated by the WSD.

Figure 6 (**a** and **b**) depicts the wear profiles of the discs. DF with 100 ppm of neutralized acid clearly caused the highest wear on the disc. The groove is wider and deeper (**figure 6 a**) but shorter (**figure 6 b**) compared to pure DF. DF with 100 ppm of ester shows similar curves, but the groove is not as deep. The wear scar of DF with 100 ppm acid has a bump in the middle of the X profile. The Y profile shows a more even bottom surface of the groove than the other three curves. DF features this bump also, the groove is the less deepest (except biodiesel) but longest which correlates with the largest WSD on the ball. According to the Y profiles (**figure 6 b**) of the discs, a slight vertical movement of the balls is expected (**figure 11**), causing a recess at the end points of the HFRR stroke.

2.2 Variation of biodiesel content

The main component DF was mixed with various amounts of biodiesel to study the wear dependence on the concentration. Lower contents of lubricity additive than shown in 2.1 were also tested. HFRR Test conditions were in accordance to the standard (60 °C, 75 min). Biodiesel (RME) concentrations ranged from 0.5 to 10 vol%. The WS1,4 values were 475 µm (0.5 vol% of RME), 295 µm (1 vol% of RME), 212 µm (2 vol% of RME), 193 µm (4.7 vol% of RME) and 176 µm (10 vol% of RME), respectively. The addition of 1 vol% of RME to DF would be already sufficient to achieve a lubricity better than the 460 µm (critical value according to EN 590). As shown in **figure 7** the concentration of 1 vol% of biodiesel gave better W_V results than the tested lubricity additives (see **figure 4**). At higher concentrations of biodiesel, the wear scars were so small that the W_V and WSD of the balls were difficult to measure. A steady decrease of W_V was observed from 0.5 to 2 vol% of RME added. The values for 4.7 vol% of RME (standard concentration) and 10 % of RME are comparable (**figure 7**).

2.3 Variation of additive content

The effect of the variation of ester, acid and neutralized acid additive concentration was tested in a range from 25 to 200 ppm. The addition of 25 ppm additive into raw DF shows only a low decrease in WS1,4 (563-575 µm, depending on additive) compared with raw DF (WS1,4 = 587 µm). According to the results in **figure 8** wear decreases with increasing additive content from 25 to 100 ppm.

The WS1,4 values for 150 and 200 ppm additives in the range from 361 – 353 µm (150 and 200 ppm Ester), 305 – 319 µm (150 and 200 ppm Acid), and 424 – 403 µm (150 and 200 ppm neutralized Acid) are not distinguishable. However these values are significantly lower than at 100 ppm concentration in the case of ester (WS1,4 100 ppm= 425 µm) and acid (WS1,4 100 ppm = 400 µm). For the neutralized acid the same WS1,4 was calculated for 100, 150 and 200 ppm (WS1,4 = 424 - 403 µm).

The results for the characterised additive concentrations indicates that there is a tendency towards lower WS1,4 values with DF containing acid than with ester and neutralized acid.

The W_V measurements are represented in **figure 9**. The ball W_V decreases with increasing additive content. The addition of only 25 ppm lubricity additive results in significant decrease of the W_V by a factor of about 2 compared with raw DF. W_V ball with additives from 25 ppm onwards is always significantely lower than W_V disc. For concentrations larger than 150 ppm, the W_V values are

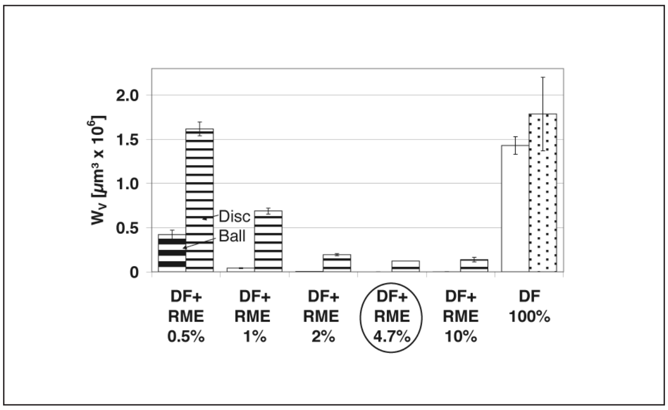


Figure 7: W_V of HFRR specimens of DF with biodiesel (0.5 to 10 vol%, 60 °C, 75 min)

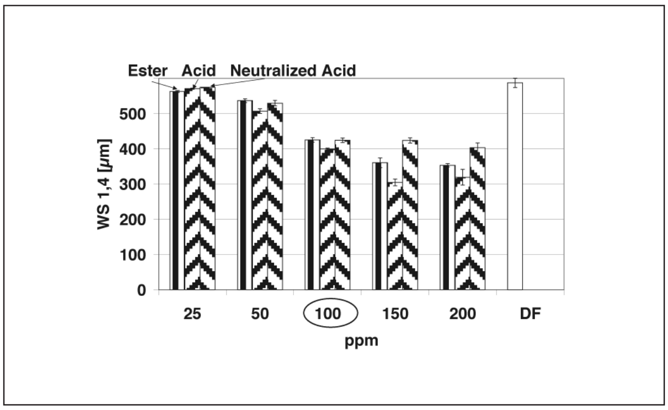


Figure 8: WS1,4 results for DF with variation of additive concentration in a range of 25 to 200 ppm lubricity additives (60 °C, 75 min)

smaller by a factor of about 50 and do not provide a considerable contribution to the cumulative W_V ball + disc.

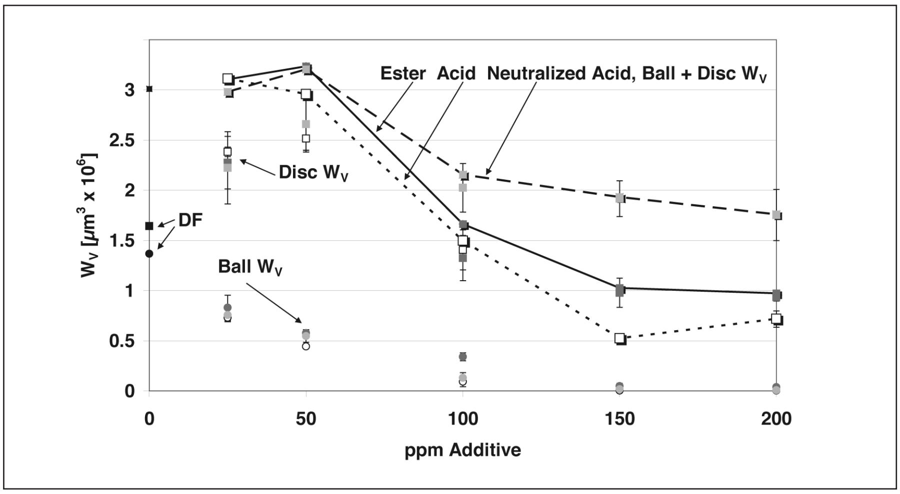


Figure 9: Wear volumes (W_V) of tribological tested specimens in HFRR in DF with 25 to 200 ppm lubricity additives (60 °C, 75 min)

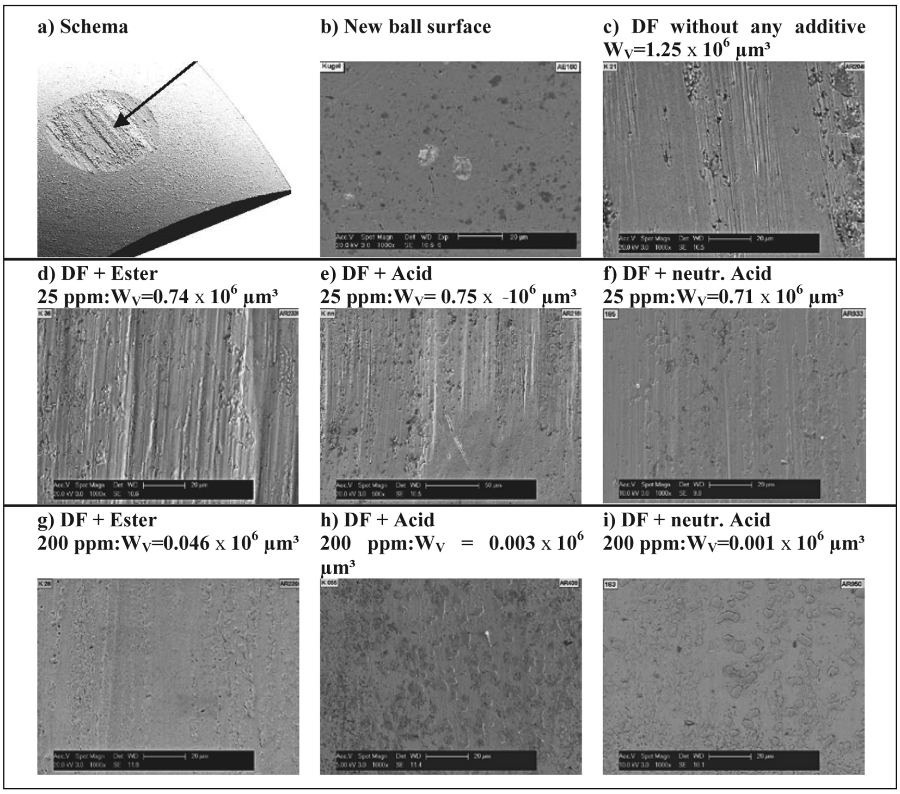


Figure 10: SEM pictures from worn ball surfaces

The SEM images in **figure 10 b** display a new ball surface and in comparison it shows the severe wear when fuel without any additive is used. **Figures 10 d**, **e**, **f** are depicting a corrugated surfaces after the test with the 3 additives in 25 ppm concentrations. The surface roughness according the µSurf evaluations are similar for the 25 ppm samples (Rq = 0,43 - 0,50 µm), also the W_V values are equal.

For concentrations of 100 to 200 ppm, ester tends to generate the highest W_V in comparison with the other additives at the same concentrations. **Figure 10**, g, h, i displays the ball surfaces at 200 ppm additive content. While the ester gave a relatively smooth worn surface (Rq = 0,32 µm) the two other additives resulted in nearly unworn surfaces (Rq = 0,14 - 0,18 µm) where surface analysis by SEM suggested material attached to the surface. These „flakes“ on the surface are composed of the elements iron and oxygen according to the EDX measurements.

Concerning disc wear, changes occurred differently to the ball wear and even showed wear increase up to 50 ppm additive concentration. Moreover, different additive behaviour was found than observed for ball wear. The use of 25 and 50 ppm additive did not lead to a decrease of W_V as anticipated, moreover there is an indication for pro-wear behaviour in comparison with DF. The highest W_V was found for 50 ppm lubricity additive. At low concentrations (up to 50 ppm) it is not possible to distinguish the three additives. But at concentrations with 100, 150 and 200 ppm a principal differentiation of the 3 additives by means of the measured W_V can be carried out:

- Neutralized acid shows only a slight W_V decrease from 100 to 200 ppm (mainly in the range of the standard deviation) being in the range of DF W_V. This is remarkable because the ball W_V is less than 15 % of DF.
- Ester and acid resultes in lower W_V from 100 to 200 ppm compared to neutralized acid or DF (up to a factor 2).
- Disc W_V with ester is lowered from 100 to 150 ppm and then remains constant. The acid additive has a minimum in W_V at 150 ppm with W_V = 0,52 x 10^6 µm³.

Summing up, **figure 9** shows that a differentiation of the additives at lower concentrations (up to 100 ppm) is difficult. At higher concentrations different wear behaviour of ester, acid and neutralized acid can be obtained.

Based on the above-mentioned results a categorization according to wear behaviour has been attempted:

Wear behaviour after testing with raw DF

The calculated W_V of ball and disc are comparable with W_V Ball=1.37 x 10^6 µm³ and W_V Disc=1.64 x 10^6 µm³. The ball wear scar with DF is nearly circular. Approximately 10 µm of the ball are worn off in height of the spherical calotte. The maximum depth of the disc wear track is only 3 to 5 µm as depicted in **figure 11**. In the top view (down to the right in **figure 11**), the track is characterized by a neck in the middle.

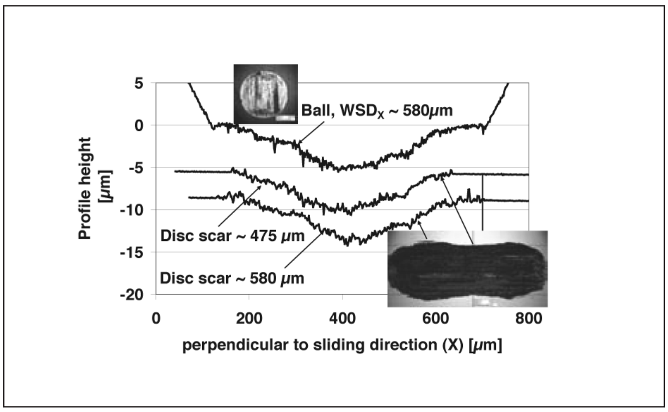


Figure 11: DF wear on ball and disc

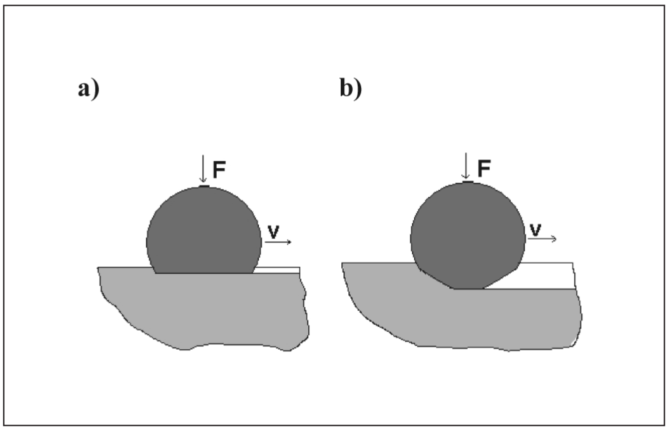


Figure 12: Schematic representation wear behaviour of ball and disc in oscillating HFRR contact - a) DF, b) DF + 25 ppm additive (ester, acid, neutralized acid)

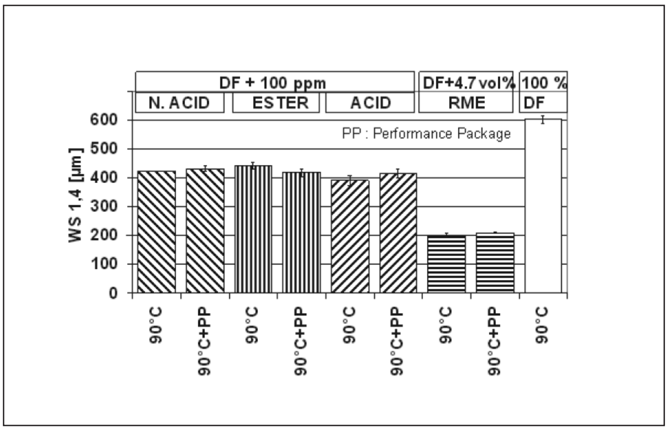


Figure 13: Lubricity following WS1,4 evaluation; DF with various additives tested at elevated temperature (90 °C, 75 min)

That means that the worn ball surface is not in full contact with the disc in the middle of the track. A slight up and down movement of the ball during sliding is assumed as cause for the shape of the disc wear track.

Wear behaviour after testing with raw DF + 25 ppm additive

The additives used were 25 ppm of ester, acid or neutralized acid which lead to W_V at balls of 0.73-0.83 x 10^6 µm^3 and W_V at discs of 2.22-2.38 x 10^6 µm^3. That means the W_V ball decreased to 0.6 x W_V DF, but the W_V disc increased to 1.64 x W_V DF. The wear scar on the ball with 25 ppm of lubricity additives used is nearly circular in top view, but in lateral view one can see, that the wear surface has a 3 dimensional shape. The wear track has an oval shape in top view. Approximately 6 µm of the ball are worn off in height. The wear scar on the disc has a depth of about 8 µm (acid, neutralized acid) being double of raw DF. The depth with the additive ester is between the values of raw DF and acid / neutralized acid.

In comparison of raw DF and DF + 25 ppm additive the linear wear of ball and disc summarized are the same. The distribution is different in the way that the ball shows severe wear with raw DF and low wear with DF + 25 ppm additive. The disc wear is low with raw DF and high with DF + 25 ppm additive (**figure 12**). The worn surface of the ball tested with 25 ppm of additive shows a convex shape, whereas the ball tested without additive displays a more flat wear surface.

Wear behaviour during testing with raw DF + 200 ppm additive

The use of 200 ppm of ester additives leaded to a W_V ball of 0.038 x 10^6 µm^3 and a W_V disc of 0.93 x 10^6 µm^3. This is a decrease to 0.03 x W_V ball DF and 0.55 x W_V disc DF. The wear scar on the disc of DF + 200 ppm ester is as deep as with raw DF. A slight up and down movement is expected. The ball wear is 2 µm in height. Summing up, a low worn ball leads to low disc wear.

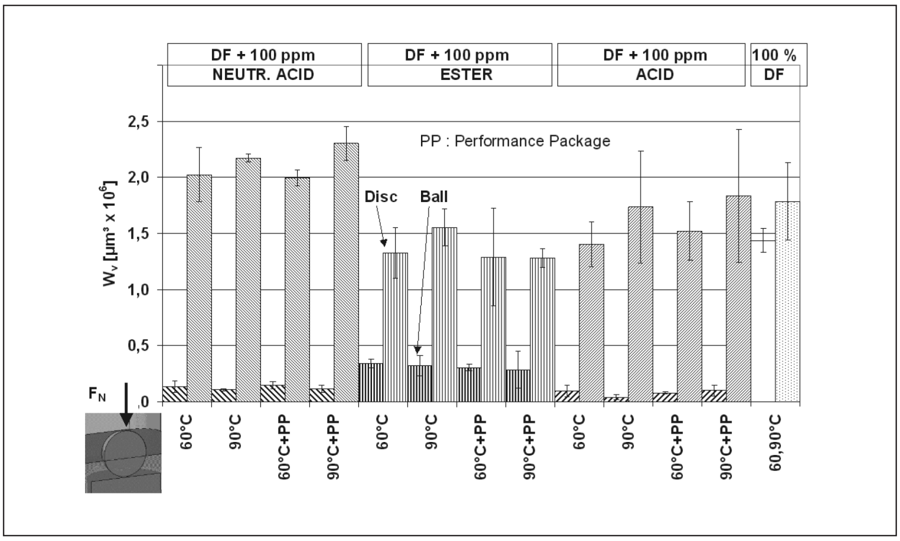


Figure 14: W_V evaluation for balls and discs, standard test time of 75 min, temperature variation (60 °C / 90 °C)

In the case of DF + 200 ppm acid the ball shows even less wear (W_V = 0.004 x 10^6 µm^3), the disc wear is comparable to 200 ppm ester (W_V = 0.715 x 10^6 µm^3). The disc does not seem to be worn equally, the wear depth is 2 to 4 µm. The ball wears 1 µm in height.

With **neutralized acid** additive in 200 ppm concentration, the ball W_V decreased to 0.006 x W_V DF (W_V ball = 0.0079 x 10^6 µm^3), but the disc wear remains constant in comparison to DF (W_V disc = 1.75 x 10^6 µm^3). The ball wears 1 µm in height. The disc wear scar of DF + 200 ppm neutralized acid is deeper (~6 µm) but not as long as with DF. The wear track does not seem to be worn equally. Differently to the previous additives, a nearly unworn ball leads to high disc wear.

Additional to additive testing, the effect of antistatic additive was also studied. For technical reasons, an antistatic additive is needed to avoid static discharging while pumping fuels. Therefore, 5 ppm of antistatic additive was added. The results showed no effects on WS1,4 and W_V at 100 ppm additive concentration.

2.4 Variation of fuel temperature

The standard test temperature for the HFRR test is 60 °C, as used for all tests discussed above. For the following tests, a test temperature of 90 °C was chosen as it relates to the range of high pressure pump supply temperatures. The test-duration remained unchanged (75 min). To find out the influence of the temperature the same tests (as already shown in **figure 3**) were also performed at 90 °C. Basically, the higher temperature in the HFRR fuel bath may result in more evaporation of some fluid components, but no effect on wear formation was observed. **Figure 13** displays the WS1,4 values.

There is no significant change in the values for DF containing various lubricity additives with or without 75 ppm of performance package. The same was observed for DF with 4.7 vol% of RME. The W_V remained almost unchanged in comparison to the values displayed in **figure 4**. In **figure 14**, a compilation of all W_V results from lubricity tests of DF with lubricity additives at 60 and 90 °C is given (see chapters 3.1 and 3.3). The W_V evaluation of HFRR tests performed at 90 °C (2nd, 4th, 6th, 8th, 10th and 12th bar) gave comparable results to those at 60 °C. Concluding from the above results it can be stated that no differentiation of lubricity performance based on temperature (60 and 90 °C) was achieved. Furthermore, no differentiation concerning the addition of 75 ppm of performance package was possible. The W_V differed in dependence on the additive used whereas the WS1,4 values where found in the same range of 400 µm. Accordingly, the W_V values for RME at a test temperature of 90 °C for the balls were again difficult to determine, the W_V for the discs remained in the range of 1 x 10^5 µm^3.

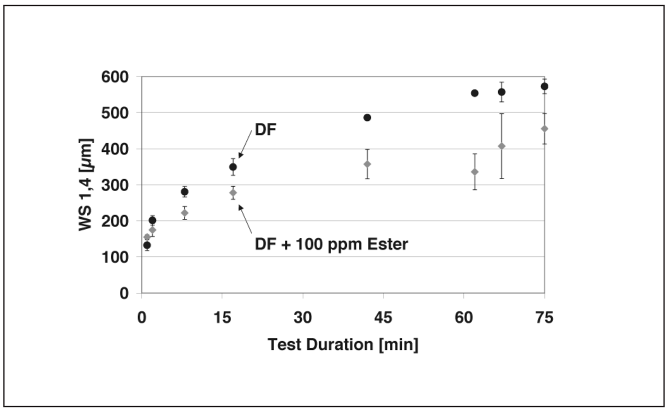


***Figure 15:** Variation of test duration, 1, 2, 8, 17, 42, 62, 67 and 75 min at 60 °C*

2.5 Characterisation of running-in process

The following studies aimed at the observation of the wear progress over time. Thus, the test duration was varied from 1, 2, 8, 17, 42, 62, 67 min to the standard test time of 75 min corresponding to sliding distances from 6 to 450 m. For each test duration, two individual tests with new balls and new discs were performed. These HFRR tests were performed for the pure DF and DF with 100 ppm of ester. As it can be seen in **figure 15** the WS1,4 increase of DF with ester additive is much flatter than with pure DF.

The wear scar images of the balls (**figure 16**) show, that the wear scars are more oval when DF with ester was used.

From the surface analytical data the wear scar profiles of the balls can be printed out. The wear progress in X direction (perpendicular to the moving direction) over the time is shown in **figure 17 a** and **b**. When using pure DF (**figure 17 a**), wear started with flat spots left and right from the ball center. The wear in the „flat spot“ region took mainly place on the ball, the disc (**figure 18 a**) is barely affected. A „dome“ remains in the middle of the ball wear scar.

In the case of DF with 100 ppm of ester (**figure 17 b**) the wear profiles have a „roof“ shape in X-direction.

The profiles appeared to have a nearly symmetric shape. The wear profiles from the discs (**figure 18 a** and **b**) showed for DF with 100 ppm of ester deeper and more V-shaped wear tracks compared to DF. Furthermore the bottom of the track is more even than that obtained with DF, where a recess at the end points of the stroke appeared (not displayed).

3 Conclusion

The importance of diesel lubricity for highly stressed tribocontacts lead to this study of the standardized HFRR model test. The investigation showed that a modified wear characterisation of ball as well as of disc can lead to a better understanding of the tribological processes.

The W_V of the ball cannot be simply calculated under the assumption that the wear volume only depends on the MWSD value. The

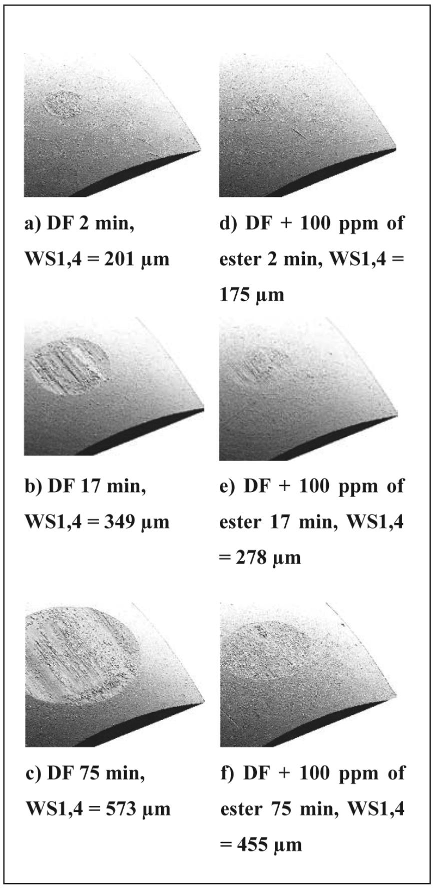


***Figure 16:** Wear scars of balls after 2, 17 and 75 min of test duration at 60 °C*

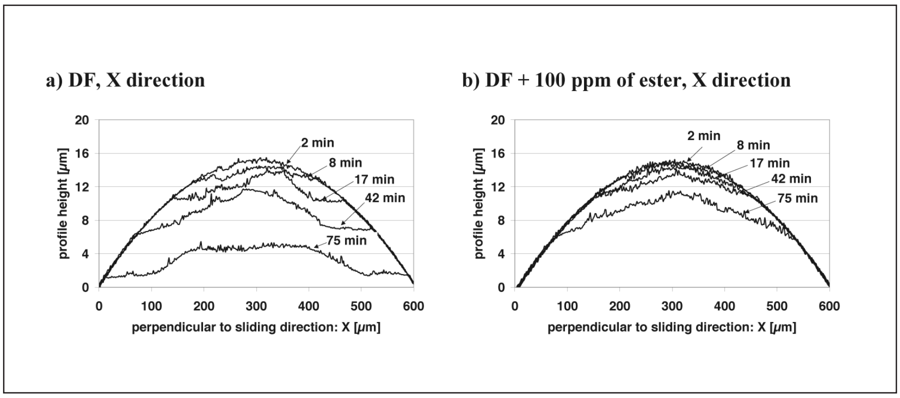


***Figure 17:** Ball wear profiles in X direction after different test durations, standard test temperature of 60 °C*

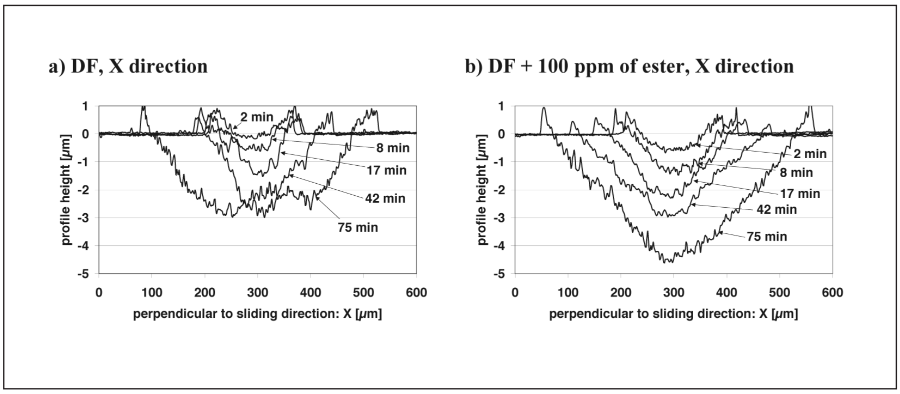


***Figure 18:** Disc wear profiles in X direction after different test durations, standard test temperature of 60 °C*

reason is that the wear of the spherical cap does not depict an even geometry. The topography depends on the used diesel fuel and additives as clearly shown in the figures of wear profiles. Thus, no correlation between the wear scar diameter and the wear volume of the ball was found. The wear volume evaluation (done by means of a confocal white light microscope and a matlab program) gave a more detailed picture of the lubricity and wear behaviour of the tested diesel fluids.

W_V evaluation enabled partly a differentiation where the standard WS1,4 values were not distinguishable. Besides wear on the ball, wear on the disc was also taken into account and gave different wear volumes depending on the diesel fuel samples. A temperature rise of diesel fuel from 60 to 90 °C showed no significant effects on wear.

The tendency for the investigated lubricity additives (100 ppm in DF) was recognized that low wear on the ball causes high wear on the disc and vice versa. For some additive concentrations the W_V of the balls can be ranked in accordance to the adsorption strength described in [3] of the lubricity additive, where acids are strongly adsorbed (low wear volume) and esters are the weakly adsorbed additives (high wear volume). The wear volumes on the discs behaved differently which could be explained with a different input of energy in the disc.

The additive content variation shows that a differentiation of the additives at lower concentrations (from 25 up to 100 ppm) is difficult or impossible, respectively. The comparison with DF depicts that a small amount of lubricity additive decreased the wear on the ball significantly, whereas the wear on the disc increased. At concentrations up to 200 ppm different wear behaviour of ester, acid and neutralized acid can be revealed.

The addition of more than 1 vol% of biodiesel (RME) to DF leads to a significant reduction of wear expressed as WS1,4 as well as W_V compared to the concentration of 100 ppm of lubricity additive.

The addition of 75 ppm of performance package as well as 5 ppm of antistatic additive did not affect wear.

The main conclusion of this research work is, that the usefulness of the High Frequency Reciprocating Rig for studies in diesel fuel tribology can be significantly increased when profile and surface evaluation is performed. The wear volume values of both ball and disc help to differentiate diesel fuels with different additives. The standard evaluation method delivering WS1,4 value turned out to be insufficient for in-depth research.

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