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ORIGINAL PAPER

Tribological Properties of Greases Based on Biogenic Base Oils and Traditional Thickeners in Sapphire-Steel Contact

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Abstract Friction and wear tests were performed with a number of greases based on biogenic esters and thickened with two metal soaps and a highly dispersed silica acid gel. The series of experiments was performed on a Nonotribometer in material combination of sapphire ball on steel disks with a range of normal loads from 1 up to 500 mN. Results directly show influences of the bulk grease components on frictional and wear behavior. Comparison of frictional and wear results makes manifest that, while in most combinations of base oil and thickener, the highest influence is found in the thickening agent, some combinations are mainly influenced by the base oil. All frictional results along with wear widths and depths as well as micrographs of the prevailing wear mechanisms are presented and discussed.

Keywords Biodegradable oils · Organic esters · Friction mechanisms · Wear mechanisms · Abrasive wear · Adhesive wear

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1 Introduction

Although undiscovered fossil hydrocarbon occurrences are predicted and technological progress will for some part increase the degree of feasibility of these resources, we will inevitably face the limits of availability of petrochemical resources in future [1, 2]. This will require a large change of ideas in the production of auxiliary and process materials in a large number of engineering fields. Consequently, a demand is made to focus on the use of renewable materials. Moreover, the steadily growing sense of ecological responsibility in society as well as legislation in concern of dealing with substances, which may have lasting harmful effects on environment puts a challenge to producers and users of lubricants to fall back upon biologically compatible alternatives.

It is stressed that lubricants are environmentally highly pertinent substances especially with regard to atmospheric carbon dioxide balance [3]. Also it is commonly known that vegetable oils more readily biodegrade. It was found that owing to their lower molecular weight, vegetable-derived-lubricants are significantly more degradable by maritime microbial communities than their mineral-derived-lubricant counterparts [4]. In combination of these demands, one can easily detect a trend toward regrowing biodegradable process materials.

Lubricating greases are produced as complex multiphase systems in which all component properties are needed for fulfillment of the desired function. In order to comply with all desired functions base oil as well as a thickening agent are needed, the latter of which may take a share of up to 50% and more depending on the required consistency and all the entailed characteristics of the grease. The presence of a metallic soap as a thickener does not only define the consistency of the grease but it also influences its frictional properties [5–9]. When formulating greases, which fully meet both above-mentioned requirements of biogenity and biodegradability, the manufacturer is challenged to take all of this into consideration for the selection of all the ingredients. If, for reasons of feasibility and lack of experience, not all components may be replaced by such of biogenic origin the utilization of biogenic base oil may at least be a step into the right direction. In the present article, some greases based on biogenic base oils are tribologically characterized.

There are only a few studies found in the literature, which directly examine the influences of main components of biodegradable greases on frictional and wear behavior. In this study, greases formulated with combinations of three different biogenic base oils and classical thickener types are compared with each other and to a grease system based on synthetically generated polyalphaolefin (PAO) thickened with the same thickening agents. Results appear to elucidate the role of both the employed base oils and thickener agents in frictional and wear behavior.

Table 1 Base oil characteristics

	ν_{40} [mm^2 s^{-1}]	ν_{100} [mm^2 s^{-1}]	Density [g/cm^3]	Degradability[a]
HOSO	38.8	8.5	0.92	96%
TMPO	48.0	9.8	0.92	96%
OCT	25.5	5.5	0.87	97%
PAO (synth. reference oil)	46.7	7.9	0.83	Yes[b]

[a] According to OECD 301B

[b] According to Materials Safety Data Sheet it is expected to be inherently biodegradable

2 Experimental Procedures

2.1 The Greases

Three different biogenic and biodegradable base oils mainly consisting of organic esters, high oleic sunflower oil (HOSO), octyldodecyl isostearate (OCT), trimethylolpropane trioleate (TMPO), and one synthetic biodegradable low viscous PAO as reference oil with basic characteristics displayed in Table 1, were used as base oils to formulate the lubricating greases studied.

Lithium and calcium soaps both of which are formulated as hydroxy stearates, as well as a highly dispersed silica acid (HDS) gel thickener were used as thickener agents. No additives were applied in the formulation except for swelling aid in the case of HDS greases. Composition and characteristics of the greases are depicted in Table 2.

2.2 The Testing Equipment

Tribological friction and wear tests for all greases were performed on a CSM nanotribometer as shown in Fig. 1. This ball-on-disk apparatus works in linear oscillation and circular rotation mode. In this study, all tests were performed in the rotational mode. The velocity of relative motion between the static ball and the dynamic disk directly depends on the diameter of the set wear track in correlation to the set rpm resulting in gliding friction. The

Table 2 Grease characteristics

Grease label	Thickener		Pu[a] [1/10 mm]	Pw[a] [1/10 mm]	NLGI class	DP[b] [°C]	Oil separation[c] [wt%]
	Type	Content [%]					
HDS PAO	Highly dispersed silica acid	10.3	269	280	2	–	1.12
HDS HOSO		13.9	238	268	2	–	0.92
HDS OCT		11.4	264	287	2	–	1.22
HDS TMPO		14.1	249	287	2	–	0.82
Ca PAO	Ca-stearate	18.8	283	287	2	133	1.16
Ca HOSO		17.6	272	279	2	132	0.80
Ca OCT		15.7	253	272	2	136	0.88
Ca TMPO		13.7	260	272	2	132	0.63
Li PAO	Li-12-hydroxy stearate	9.7	279	283	2	205	2.57
Li HOSO		15.9	264	272	2	195	1.12
Li OCT		13.6	279	294	2	188	1.79
Li TMPO		20.7	264	279	2	190	0.81

[a] Unworked and worked penetration according to DIN ISO 2137

[b] Dropping point according to DIN ISO 2176

[c] According to DIN 51817

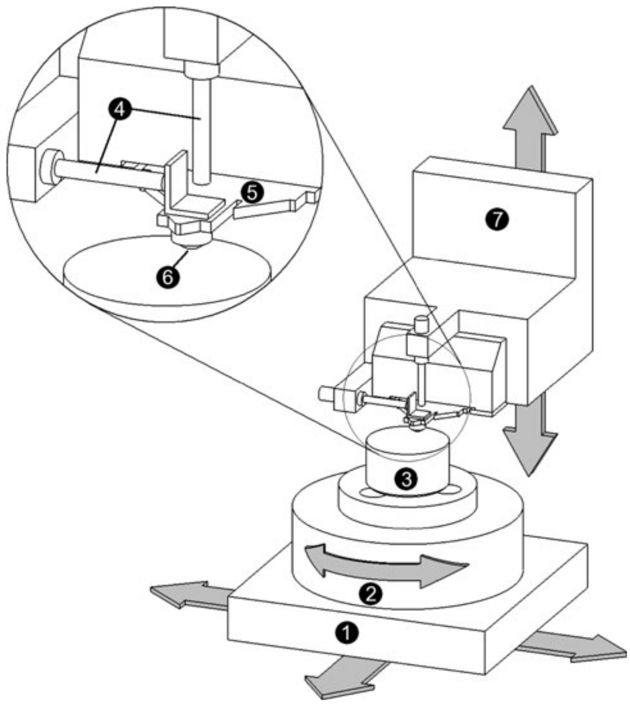

Fig. 1 Experimental set-up—nanotribometer Note: *1* XY-cross-table, *2* Rotational axis, *3* Specimen steel disk, *4* Interferometrical sensors, *5* Cantilever, *6* Specimen sapphire ball, *7* Z-step-motor-axis with integrated piezo-axis

step motor of the rotational axis makes up to 120 rpm and is mounted to an XY-cross-table, which positions the disk under the ball to set up a controlled track radius. Specimen balls are fixed to a cantilever module and normally positioned to the plate by a Z-step-motor-axis. Defined load is applied to the ball by fine positioning and deflecting the cantilever via piezo-axis. The deflection of the cantilever is interferometrically measured and then controlled. By means of two distally attached mirrors, the deflection of the cantilever in radial (frictional force) as well as normal (normal force) direction of the plate revolution is detected and by the very precise calibration of the spring constant of the cantilever in both main directions calculated into frictional and normal forces. Both of these forces, the coefficient of friction, the relative indention of the ball into the plate, as well as the ambient temperature and the relative humidity of the measuring cabin are detected and recorded during the measurements at a set sample rate. The utilization of cantilevers with different spring constants covers a large load range from 0.1 up to 500 mN. The high-precision optical interferometric measurement detects the wear height with a resolution of 50 nm. Despite relatively low normal forces of the nanotribometer, high values of Hertzian stress (up to $\sim$2GPa) can be applied to the measured tribological system. This is due to the fact that very small balls may be applied to the cantilever.

2.3 Tribological Tests

All of the tribological examinations presented below were performed on the above-described nanotribometer in rotational mode with a relative gliding speed of 5.0 mm s^{-1}, using a sapphire ball with 1.5-mm diameter and steel plates (115CrV3, hardness 22.72 HRC). All the steel plates were metallographically grinded and polished with a soft finish diamond paste of particle size 3 μm in the last polishing step. The nanotribometer worked in ambient pressure and temperature. In the test procedures, the normal force was varied with resulting Hertzian stress values as shown in Table 3.

At first, a grease layer of 0.05 mm was applied to the steel plate then the measurement parameters of normal force, relative speed, and track radius were set up. The test duration was 50 min each. In order to allow conclusions to be drawn about the friction and wear influence of each bulk component of the greases the above-described tests were performed with all the greases, each base oil on its own and in completely dry contact situation.

3 Results

3.1 Friction

In general, friction coefficients vary with high fluctuation in most frictional tests. Averaged coefficients of friction of all the examined greases with material combination sapphire ball on steel plate are shown in Fig. 2. Frictional results in the present study have an average standard deviation of more than 10%. Fluctuations in values of coefficients of friction are lower for higher normal forces of 500 and 100 mN. Values for 10 mN and less in fact are so unstable that they become useless. For this reason, the depiction of frictional results for normal forces of 10 and 1 mN is renounced. This typical effect of the nanotribometer is caused by rheological influences evoked by grease layer displacement at every revolution of the steel plate. For this reason, it is very important to always apply grease layers of the same thickness to the steel disks. Thicker grease layers evidently cause higher resisting abilities toward repositioning by the specimen ball.

In attempting to attribute frictional values to bulk grease components, one has to regard results from each component's point of view. At first consideration from the

Table 3 Test conditions

Normal force [mN]	500	100	10	1
Max Hertzian stress [GPa]	1.52	0.89	0.41	0.19

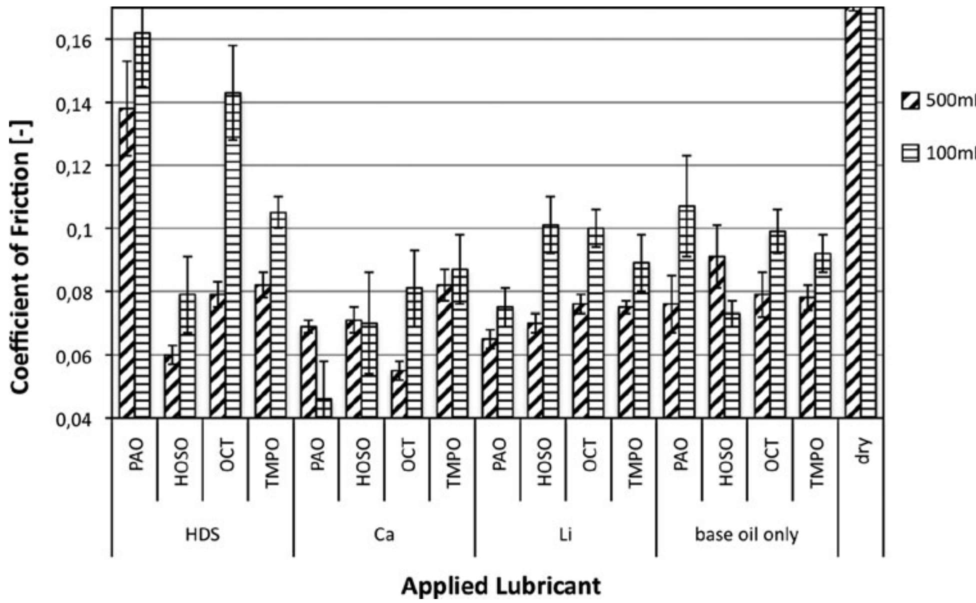

Fig. 2 Friction in contact situation sapphire ball on steel disk

thickener's point of view, one detects a general tendency of the highest frictional values for the series of HDS-thickened greases. The group of calcium-thickened greases stands out because of its lowest friction coefficients in comparison, followed by lithium-thickened greases.

When comparing component attributes and their influence on friction from the base oil point of view, one first of all finds out that the ranking within the group of base oils from PAO with highest frictional values down to HOSO with the lowest coefficients of friction is also found in the group of greases formulated with HDS-gel-thickener with this tendency even more intensified. Reasons for this effect will be discussed later on with respect to wear intensities and mechanisms. It also shows that the group of base oils presents higher frictional values than Ca-soap-thickened formulations in the present frictional contact situation sapphire ball on steel plate. Moreover, the ranking that was found for base oils is almost completely turned to the opposite tendency now with least coefficients of friction for Ca-PAO. Results also show that influences of single base oils on Li-thickened greases are too small to be detected or even interpreted into tendencies. The only conspicuousness is found for PAO thickened with Li-soap—here again it results in smallest coefficients of friction.

3.2 Wear

Different behaviors of grease formulations and their components are generally more explicitly displayed in wear than in frictional results. In the same way, wear measurements fluctuate less than frictional measurements. Considering the full dimensions of wear only after the completion of a frictional measurement implies to break a process factor down into a static view. This way fluctuation is eliminated to some extent. However, it is also important to keep in mind that friction and wear always depend on each other in the total frictional system. In this series of experiments, wear was analyzed statically and process orientated. For the static part, which was measured after the tests, the widths of all the wear tracks on steel disks and the corresponding diameters of all the wear scars on sapphire balls are displayed for all the measured normal forces in Fig. 3. This diagram shows that influences of bulk components differ with the combination of materials in the tribological system. Micrographs of all the wear marks produced with normal load conditions from 500 down to 10 mN are displayed in Figs. 5, 6, and 7. As it has been done previously with the interpretation of frictional results, wear characteristics are also associated with all single bulk grease components. Sorting wear characteristics according to thickening agents shows that ball and disk wear need to be evaluated separately. Some greases e.g., produce high rates in ball wear but leave the disks unaffected and vice versa. It also becomes apparent that each normal force has its own characteristic influence on wear behavior so it does not always prove to be true when simply predicting less wear as a result of less normal force.

The most evident fact for HDS-based greases is that they produce the widest wear scars in sapphire balls, as they are even wider than in completely dry contact situation. For a normal load of 500 mN, this same effect takes place on the surface of the steel plates, resulting in the widest wear tracks measured in the whole series of experiments ranging from 35 µm in case of HDS–OCT up to 62 µm for HDS–PAO. The smaller the normal loads, however, the more these tendencies diverge. While wear scars on the sapphire

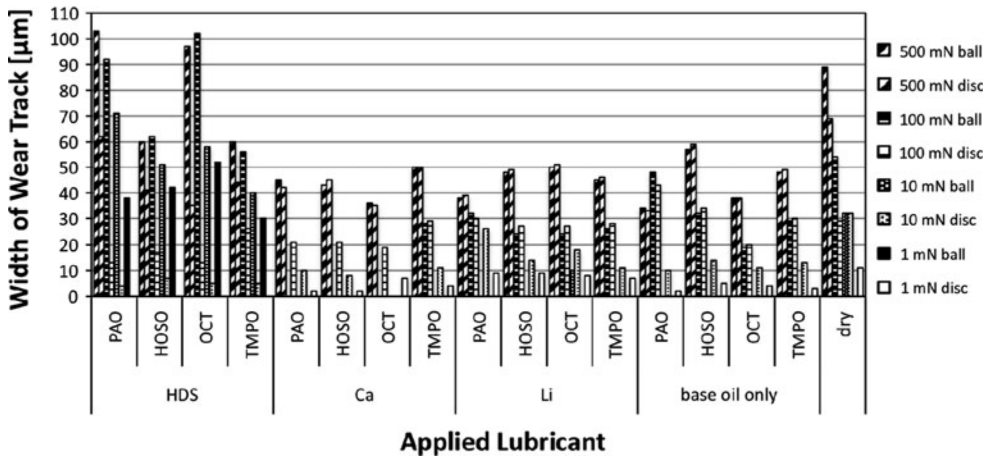

Fig. 3 Width of wear tracks—sapphire ball on steel disk

balls remain the biggest compared to all the other lubricants measured and even when compared to dry contact, the wear tracks on the steel plates become smaller and smaller. This effect becomes the most evident for 1 mN, the smallest of all normal forces applied in this series of experiments. At this load HDS-thickened greases still produce very wide wear scars ranging from 30 µm in case of HDS–TMPO to 52 µm in diameter for HDS–OCT (displayed in black solid bars in the diagram in Fig. 3), while, on the other hand, they do not produce any detectable wear on the steel plates. For soap-thickened greases and base oils only, there is an opposite effect resulting in no scars on the balls with still evident and measurable wear tracks on the plates (white solid bars in the diagram). Moreover, less normal force in HDS-based greases results in much less wear compared with soap greases—wear-force gradients in HDS-thickened greases seem to differ from those in metal soap thickened ones. With all the metal soap greases and base oils only, measured at 500 mN normal force, ball wear equals disk wear with similar wear widths ranging from 33 to 59 µm. Moreover, at these lubricants, with the exception of Li-OCT, there is no detectable wear in sapphire balls at 10 mN normal force. It also becomes clear that at normal forces of 100 mN and less Calcium greases produce the least if any wear in sapphire balls. The only Ca-grease formulation that occasions wear in the sapphire balls is Ca-TMPO. The same thing applies to disk wear at a normal force of 100 mN, here also calcium greases produce the lowest values ranging from 19 µm in case of Ca-OCT to 29 µm for Ca-TMPO. At normal forces of 10 mN, Calcium greases together with base oils only produce least wear in disks with values ranging from 8 µm in case of Ca-HOSO to 14 µm for HOSO base oil only. The test results for Li-greases show that for normal loads of 500 and 100 mN, each width of ball wear approximates to the corresponding wear in the disks with values extending from 38 to 51 µm in 500 mN tests and from 24 to 32 µm at 100 mN of normal load. On the other hand, for 10 mN and less, the only wear to be detected is located on the disk. The only exception to this rule is found for Li-OCT. Similar behavior is observed with Ca-TMPO. The outcomes of tests with applied loads of 500 and 100 mN on base oils only reveal that, just like within the group of Li-greases, the widths of ball wear resemble the according dimensions of disk wear with values stretching from 33 to 59 µm at 500 mN loads and from 19 to 48 µm at normal forces of 100 mN. It also shows that with applied loads of 10 mN in the group of base oils, there is only disk wear revealing values of the same sequence as with higher loads, which is HOSO, TMPO, PAO, and OCT from the highest to the lowest. In the upper spectrum with normal loads of 500 and 100 mN in dry contact situation with sapphire balls on steel disks, wear widths decrease with lower normal loads. At these loads, wear tracks on the disks are 22–46% narrower than those on the corresponding balls. In the lower spectrum of normal loads of 10 and 1 mN in unlubricated contact, however, widths of wear tracks do not decrease with smaller forces in the case of sapphire balls. In case of 10 mN, wears in both contact partners are in equilibrium.

When now sorting wear characteristics according to the applied base oils in the formulation, one soon finds out that the influence of the base oil on wear is much smaller than that of the thickener. It also becomes apparent that the base oil influences differ according to the formulated thickener. Thus, it shows that within the group of HDS-greases, HOSO and TMPO oils generate the least wear, while PAO and OCT originate the highest wear widths. On the other hand, in the tests of plain base oils, there are opposite tendencies to be detected with HOSO and TMPO producing the highest wear widths.

The above-mentioned process-orientated analysis of wear experiments was performed as in situ measurement in every single frictional test on the nanotribometer. As described earlier, the nanotribometer monitors wear in the form of indention of the specimen ball into the testing

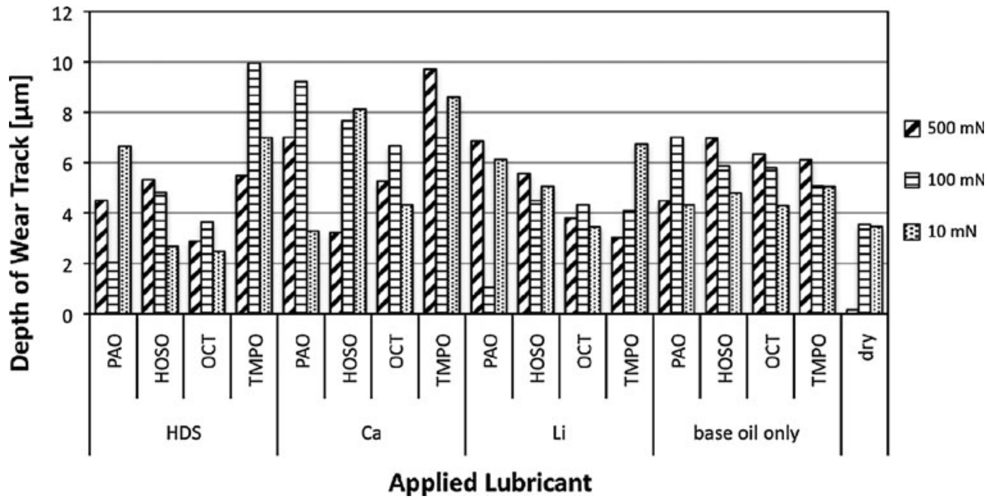

Fig. 4 Depth of wear tracks—sapphire ball on steel disk

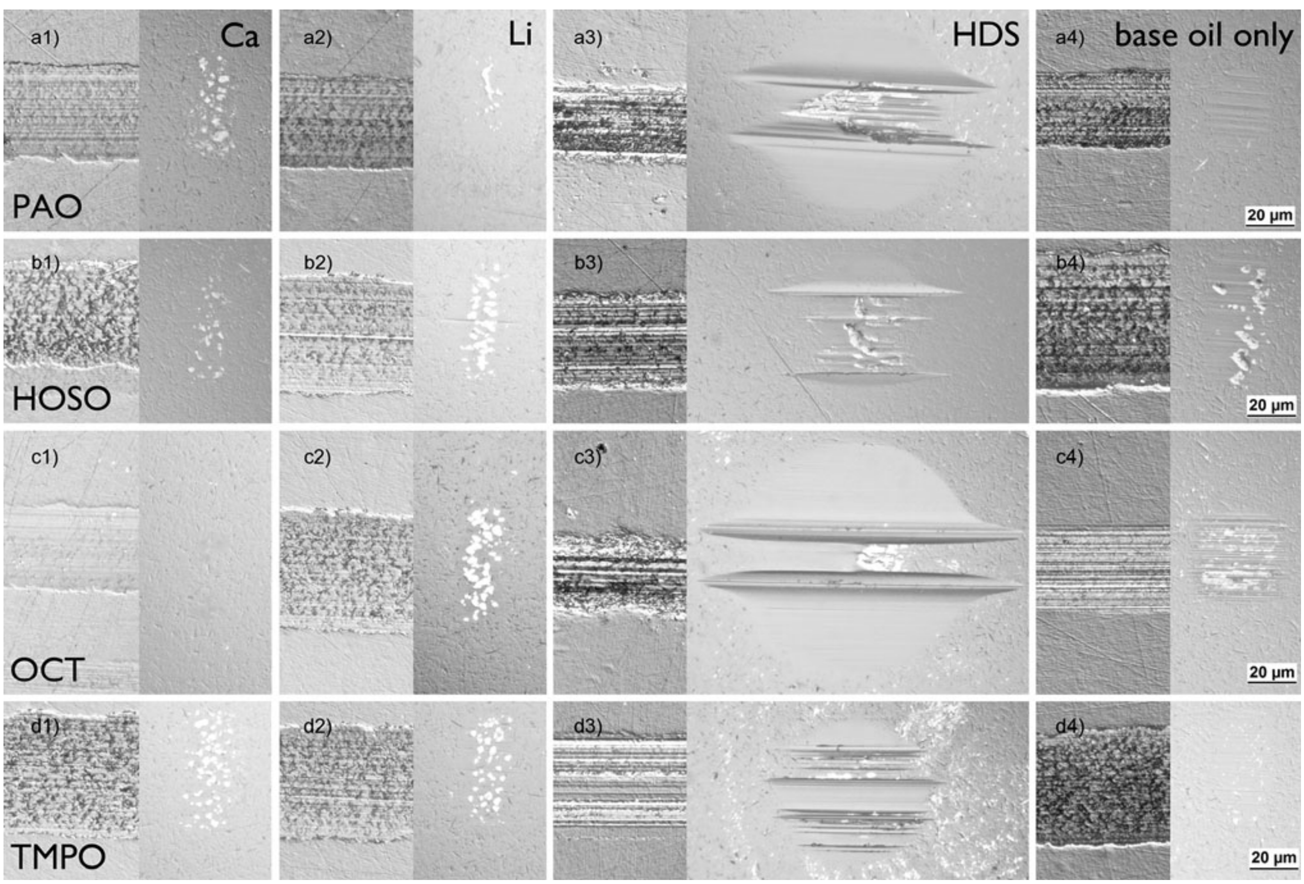

Fig. 5 Matching sets of disk and ball wear—normal load 500 mN, sapphire balls on steel disks Note: Grease compositions and base oils only—sorted by applied thickener in columns and base oil in lines

surface. This way it measures the total depth of penetration but does not, however, judge these indention results according to the location of wear in the surfaces. In other words these wear penetration results do not give any information about whether the wear took place in the ball, the disk or intermediate layers. Measurement results show a steadily declining course of values as expected for indention. Because of the regularity of the decrease of values only the final values are displayed in this study (see Fig. 4).

The biggest noticeable difference between the depths and the widths of wear tracks in this series of experiments

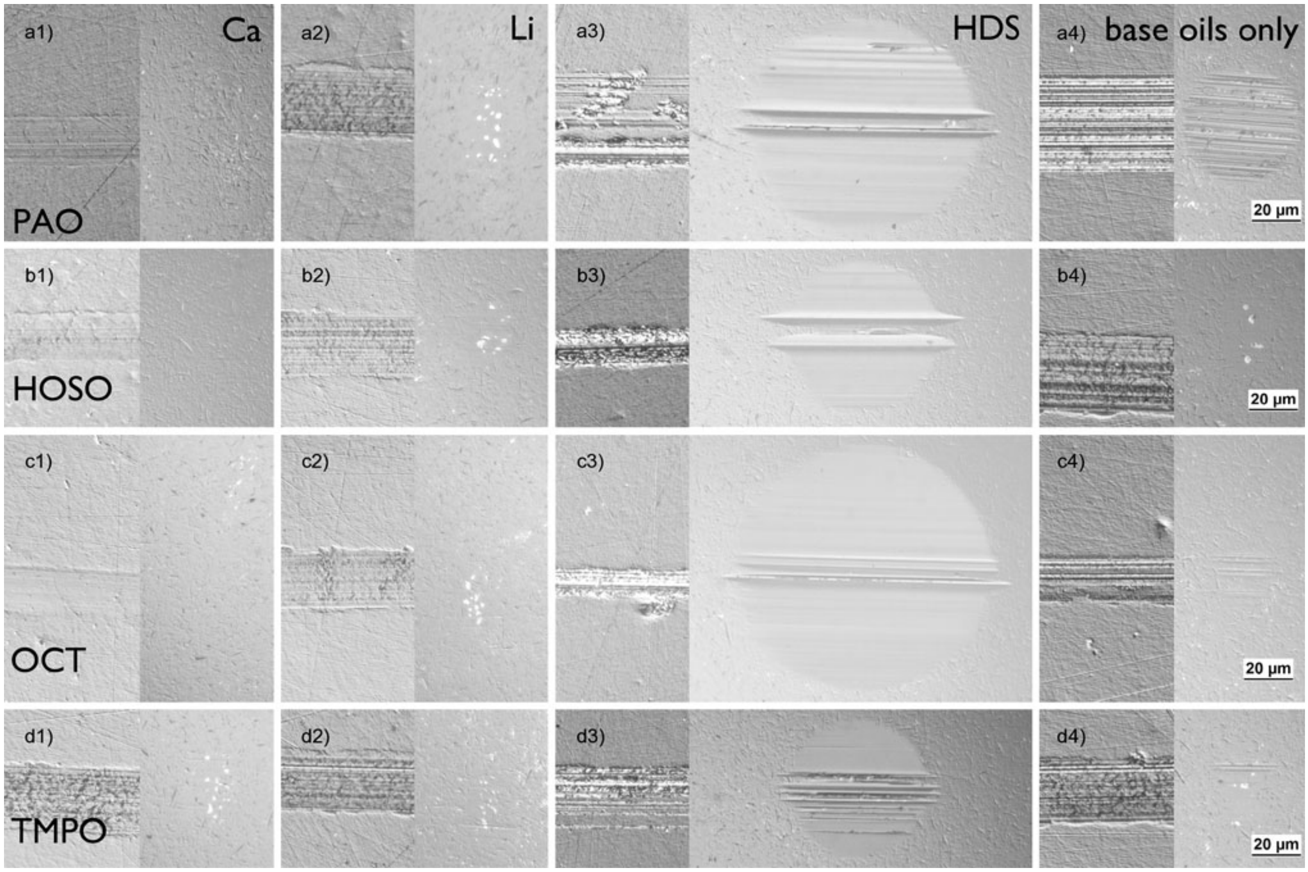

Fig. 6 Matching sets of disk and ball wear—normal load 100 mN, sapphire balls on steel disks Note: Grease compositions and base oils only—sorted by applied thickener in columns and base oil in lines

is the relatively low penetration depth of HDS-greases. In the measurements of wear widths, the values for HDS-greases jutted out of all other results, while in the depth of wear, they take the lowest segment. The only exception to this rule is HDS–TMPO, which is ranked with the highest values. Another conspicuousness about theses results is the fact that categorization by thickening agents, which showed distinct differences between the single groups in the analysis of wear widths does not induce the same effect when evaluating the wear depths. What also becomes clear when considering wear depth results categorized by thickener is that the group of Ca-greases now reveals the highest wear depths, while during the analysis of wear widths, it had the lowest rates. Moreover, in contrast to the evaluation of wear widths, now base oil results show quite steady behavior and do not seem to give any room for interpretation toward a ranking. Probably, the most peculiar and unexpected outcome of this analysis are the very low indention depths measured for the completely dry contact. Categorizing the indention depths by the formulated base oil results in a ranking that starts with the highest values for TMPO (6.48 ± 2.14 μm), followed by HOSO (5.38 ± 1.62 μm), PAO (5.21 ± 2.36 μm), and OCT (4.44 ± 1.33 μm).

4 Discussion

At the examination of friction and wear as two interrelating dimensions, it is insufficient only to take frictional values and wear widths and depths into consideration to receive significant information about the tribological system. When trying to understand and correctly evaluate friction and wear, one must also comprehend their states, types, and especially their mechanisms. Different wear states, as defined by Fleischer [10] and Czichos [11], evoked by specific wear types are made manifest through different wear mechanisms, which make a deep impact on frictional and wear behavior. As a consequence, different wear mechanisms result not only in different wear heights and widths but also in altering frictional values. Moreover, it is possible even to have several wear mechanisms present in one tribological contact system. Micrographs of wear scars on the disks and balls of all tribological systems examined

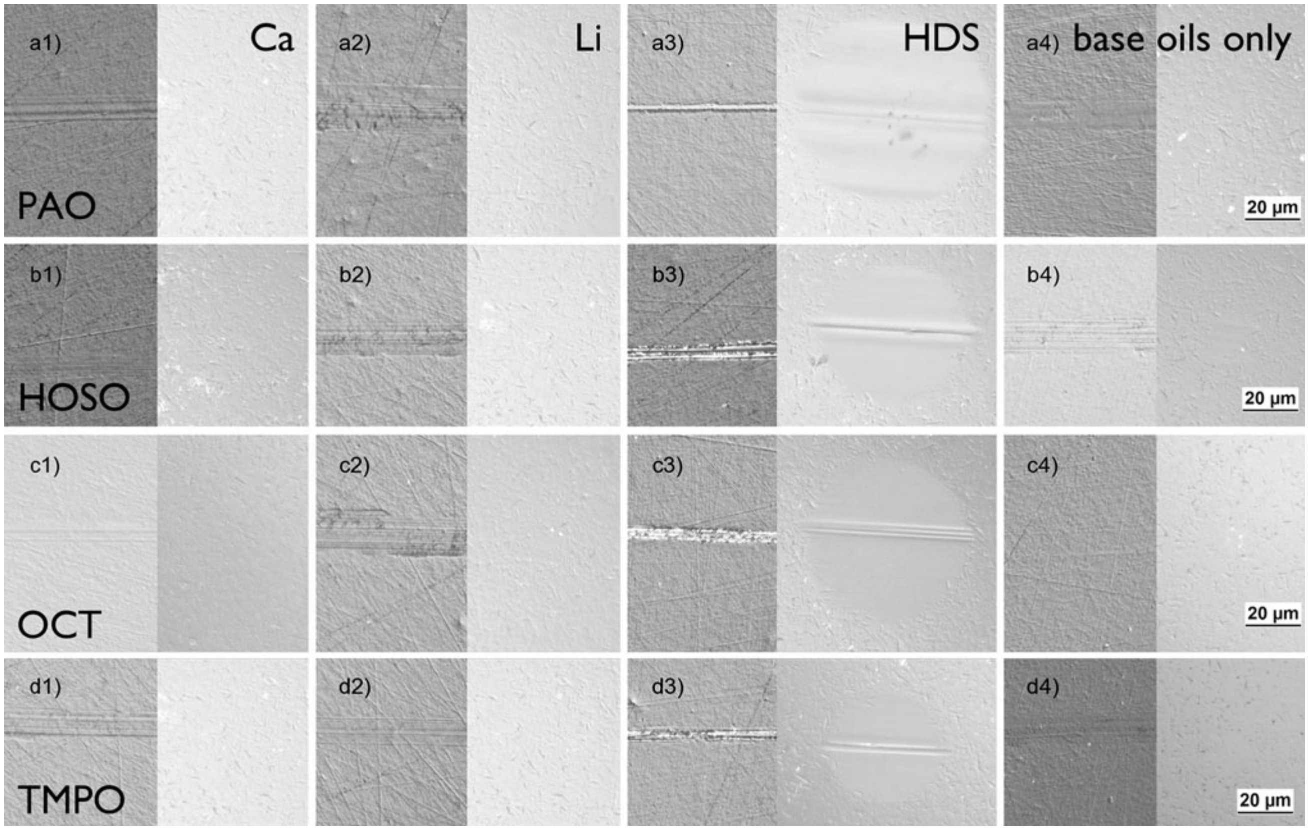

Fig. 7 Matching sets of disk and ball wear—normal load 10 mN, sapphire balls on steel disks Note: Grease compositions and base oils only—sorted by applied thickener in columns and base oil in lines

earlier in this study are displayed in Figs. 5, 6, and 7; they supply an opportunity to interpret prevailing wear mechanisms. Wear widths and depths depicted in these micrographs were analyzed earlier in this study and are only to be mentioned again with reference to comparison of images and data presented earlier in diagrams. When evaluating these images according to wear mechanisms, one has to be very careful to only objectively consider present facts. These facts are e.g., the number and the intensity of wear grooves when appraising abrasive processes, number, and size of fretting particles that adhere to counter surfaces when assessing the adhesive wear process and the number with its accompanying size of cracks and their intensity orthogonal to the direction of relative motion which point to the fact that a surface delamination process took place in the tribological system. Measuring depth profiles of the wear tracks would help in analyzing the only just mentioned intensities. However, profile measurements were not possible in this study, and so intensities of wear mechanisms will be rated and set in relation to each other as displayed in Tables 4, 5, and 6. Every single figure has its own rating so that intensities are compared to the average of each single figure with a rating from less than average up to superior impact (see notes of Tables 4, 5, 6).

Summing up the image interpretation of all wear marks in Figs. 5, 6, and 7, one may detect a good correlation to the frictional and wear results presented earlier in this study. Within the group of Ca-greases, the images reveal surface delamination as well as plastic deformation and abrasive wear mechanisms in the disks and mostly exclusively adhesion mechanisms in the sapphire balls. The same mechanisms occur in the group of Li-greases with generally higher intensity. This behavior can be elucidated only when taking the microphysical mode of action of lubrication into consideration. Lubrication only comes into effect by the application of a lubricant film that mechanically separates the contact partners [9, 11]. Both, base oil and thickening agent influence the formation of this separating film in lubricating grease [7, 12]. The results of performed series of experiments show that the thickener's influence is more substantial in this context. To some extent, explanations for these effects can be found in the microstructure of the thickening agents. Metallic soaps usually form three-dimensional networks [13] of polymorphic soap fibers that confine the oil with secondary valence bonds. It is well known that lithium-greases generate longer and more densely arranged and entangled fibers than calcium-greases [14–17]. This difference could be an explanation for the

Table 4 Wear mechanisms in sets of disk and ball wear—normal load 500 mN, sapphire balls on steel disks

	Ca	Li	HDS	Base oil only
PAO	Disk	Disk	Disk	Disk
	Surface delamination	Surface delamination+	Plastic deformation++	Surface delamination
	Plastic deformation	Plastic deformation	Abrasion+++	Plastic deformation
	Abrasion+	Abrasion+		Abrasion
	Ball	Ball	Ball	Ball
	Adhesion+	Adhesion−	Adhesion+	Abrasion+
			Abrasion+++	
HOSO	Disk	Disk	Disk	Disk
	Surface delamination+	Surface delamination+	Plastic deformation+	Surface delamination+
	Plastic deformation+	Plastic deformation+	Abrasion+++	Plastic deformation+
	Abrasion−	Abrasion+		Abrasion
	Ball	Ball	Ball	Ball
	Adhesion	Adhesion+	Adhesion+	Adhesion+
	Abrasion+	Abrasion+	Abrasion+	Abrasion+
OCT	Disk	Disk	Disk	Disk
	Plastic deformation−	Surface delamination+	Plastic deformation++	Abrasion++
	Abrasion−	Plastic deformation+	Abrasion+++	
		Abrasion+		
	Ball	Ball	Ball	Ball
	No wear marks	Adhesion+	Adhesion+	Adhesion+
			Abrasion+++	Abrasion++
TMPO	Disk	Disk	Disk	Disk
	Surface delamination+	Surface delamination+	Plastic deformation+	Surface delamination+
	Plastic deformation+	Plastic deformation+	Abrasion++	Plastic deformation+
	Abrasion−	Abrasion+		Abrasion
	Ball	Ball	Ball	Ball
	Adhesion+	Adhesion+	Abrasion+	Adhesion−
				Abrasion+

Notes: Relative rating in comparison to each other: − less than average; + more than average; ++ much more than average; +++ superior impact

lower coefficients of friction and the generally lower wear rates of examined calcium-greases compared to the greases formulated with lithium-soap. (Table 5).

The group of HDS-gel-greases tested under all normal load conditions presents itself with much different wear marks than those known for soap greases. In all sapphire balls used to measure single greases, there are marks of the highest abrasive wear in the central region surrounded by a circular area of homogenous fine particle erosion that almost looks like polished material. The surface roughness of these polished areas, even though not measured, stands out from the processed surface of the rest of each sapphire ball. The central grooves of the strongest abrasive wear in the sapphire balls created deep highly abrasive marks in the corresponding surfaces of the steel disks. The fact that apart from these deep marks, the steel surfaces appear completely unspoiled, while on the surfaces of the sapphire counter parts, there are much larger worn-off areas is the most conspicuous for the group of HDS-greases. Again, reasons for this effect have to be found in the microstructure of the thickener. HDS-thickener mostly consists of SiO_2 particles of size 0.1 up to 1.0 µm [18]. The presence of theses particles has been proven in the residue of wear debris via EDX-analysis [16, 17]. The high proportion of abrasive wear with the use of HDS-greases especially in the sapphire balls is to be explained by the extreme hardness of SiO_2 particles (5–7 on the hardness scale according to Mohs). Although sapphire is even harder (up to 9 on the Mohs scale), it yields the persistent influence of SiO_2 particles. It is assumed that these SiO_2 particles are elastically embedded in the metal surface of the steel plates during the course of performed frictional tests without abrasively influencing the steel. This would explain why, in some parts, the sapphire balls wore off, while the corresponding contacted area in the steel plates was left unaffected.

Table 5 Wear mechanisms in sets of disk and ball wear—normal load 100 mN, sapphire balls on steel disks

	Ca	Li	HDS	Base oil only
PAO	Disk	Disk	Disk	Disk
	Plastic deformation	Surface delamination+	Plastic deformation+	Abrasion++
	Abrasion	Plastic deformation+	Abrasion+++	
		Abrasion+		
	Ball	Ball	Ball	Ball
	No wear marks	Adhesion+	Abrasion+++	Abrasion++
HOSO	Disk	Disk	Disk	Disk
	Surface delamination−	Surface delamination	Plastic deformation+	Surface delamination+
	Plastic deformation	Plastic deformation	Abrasion++	Plastic deformation+
	Abrasion−	Abrasion		Abrasion+
	Ball	Ball	Ball	Ball
	No wear marks	Adhesion	Abrasion++	Adhesion+
				Abrasion
OCT	Disk	Disk	Disk	Disk
	Plastic deformation−	Surface delamination	Plastic deformation+	Surface delamination
	Abrasion−	Plastic deformation	Abrasion+	Plastic deformation
		Abrasion		Abrasion
	Ball	Ball	Ball	Ball
	No wear marks	Adhesion	Abrasion+++	Abrasion+
TMPO	Disk	Disk	Disk	Disk
	Surface delamination+	Surface delamination+	Plastic deformation+	Surface delamination+
	Plastic deformation+	Plastic deformation+	Abrasion+++	Plastic deformation+
	Abrasion	Abrasion+		Abrasion+
	Ball	Ball	Ball	Ball
	Adhesion+	Adhesion	Abrasion++	Adhesion−
				Abrasion+

Notes: relative rating in comparison to each other: − less than average; + more than average; ++ much more than average; +++ superior impact

Because of major differences in the prevailing wear mechanisms in the group of base oils as sole lubricant, there seems to be smaller correlation between wear and friction. There is slight abrasive wear in the ball and the disk and surface delamination in the disk evoked by the use of PAO. The same mechanisms in addition to adhesive wear work with HOSO. OCT is the only base oil that exclusively causes abrasive wear on both frictional partners. TMPO on the other hand only produces surface delaminations in the steel plate with only marginal abrasive and adhesive wear in the ball.

Although in the formulated greases the general influence of base oils on friction and wear only played a subordinate role, there are still some observations worth mentioning. Since all tests were performed in rotational mode and the grease layer was applied only before the test, there might be a tendency toward starvation of the frictional track. It is assumed that the lower the viscosity of the base oil, the more it tends to flow back into the frictional track. Comparing the base oil viscosities to frictional and wear results, however, substantiates this assumption only to the extent of a general tendency.

Wear results also make clear that base oils on their own behave differently in the tribocontact than base oils in formulation with different thickeners. The OCT ester and PAO measured in 500 and 100 mN of normal load regime are the oils with the highest rates of abrasive wear in the sapphire balls. This characteristic seems to be even more intensified in combination with HDS-gel-thickeners where they result in the widest wear diameters. In combination with Li-soap-thickener, however, this characteristic is limited to the same extent of all other base oils; in addition, most interestingly, Ca-thickener seems to change this effect to the opposite in the case of OCT-ester resulting in the smallest wear diameters with the least abrasive wear. These results directly correlate to the frictional examinations displayed in Fig. 2. Microstructural analysis of some exemplary Li-greases by means of atomic force microscopy may help elucidate these effects. AFM images displayed in Fig. 8 show a direct influence of the base oil on

Table 6 Wear mechanisms in sets of disk and ball wear—normal load 10 mN, sapphire balls on steel disks

	Ca	Li	HDS	Base oil only
PAO	Disk	Disk	Disk	Disk
	Abrasion+	Surface delamination+	Plastic deformation+	Plastic deformation+
		Plastic deformation	Abrasion++	Abrasion
		Abrasion		
	Ball	Ball	Ball	Ball
	No wear marks	No wear marks	Abrasion+++	No wear marks
HOSO	Disk	Disk	Disk	Disk
	Abrasion	Surface delamination	Plastic deformation+	Surface delamination
		Plastic deformation	Abrasion+++	Plastic deformation
		Abrasion		Abrasion++
	Ball	Ball	Ball	Ball
	No wear marks	No wear marks	Abrasion ++	Adhesion
				Abrasion
OCT	Disk	Disk	Disk	Disk
	Abrasion	Surface delamination	Plastic deformation+	Abrasion
		Plastic deformation+	Abrasion++	
		Abrasion		
	Ball	Ball	Ball	Ball
	No wear marks	Adhesion	Abrasion+++	Adhesion+
TMPO	Disk	Disk	Disk	Disk
	Surface delamination−	Abrasion	Plastic deformation+	Surface delamination+
	Plastic deformation−		Abrasion++	Abrasion+
	Abrasion+			
	Ball	Ball	Ball	Ball
	No wear marks	No wear marks	Abrasion++	Abrasion

Notes: relative rating in comparison to each other: − less than average; + more than average; ++ much more than average; +++ superior impact

Fig. 8 AFM—micrographs of some exemplary Li-greases

the formulation of soap fibers. In combination with HOSO and TMP, the Li-soap resulted in long, mostly parallel and less entangled fibers. However, in combination with OCT, it resulted in rather short and more entangled fibers. As explained earlier in this study, it is assumed that the shorter soap fibers result in less friction and harmful wear mechanisms. In deduction, it is assumed that the less abrasive character of shorter soap fibers in general and specifically in combination of Li-soap-thickener and OCT base oil counteracts the higher abrasive properties of OCT resulting in less-to-equal wear intensities and frictional values.

Different behaviors between HDS greases and base oils only provide some examples for diverging tendencies.

With all HDS-greases, wide wear tracks result in high values of coefficients of friction—with base oils only, this effect is turned to the opposite—and the high values in wear width result in low friction.

5 Conclusions

While highly dispersed silica acid gel thickener most substantially affected the wear behavior toward a highly abrasive nature, lithium and calcium thickeners mostly resulted in a combination of plastic deformation with slight surface delamination in the steel plates and adhesive wear in the sapphire balls. While base oil influences retain a high impact with the use of HDS-gel and Ca-soap as thickeners, they are limited in the group of greases thickened with of Li-soap. Synthetic PAO and biogenic OCT ester as base oils maximized the abrasive nature of HDS-gel thickener. OCT ester, on the other hand, minimized the effects of adhesion of particles to the sapphire ball surface and the intensity of surface delamination of the steel surface in combination with Ca-soap thickener. Ca-thickened grease formulations generally resulted in least wear and coefficients of friction. Lithium greases resulted in values for the coefficient of friction and wear widths similar to base oils, although wear depths and wear mechanisms were much different. HDS greases resulted in the highest wear rates and frictional values as well as the most destructive wear mechanisms.

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