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Tribology of polypropylene and Li-complex greases with ZDDP and MoDTC additives

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ABSTRACT

The influence of thickener and additive interactions on grease lubricating performance is examined. Polypropylene and lithium complex thickened (Li-complex) greases were tested both as neat greases and with a 2 wt% addition of ZDDP and/or MoDTC. A combination of ZDDP and MoDTC in the polypropylene grease provided the lowest friction with greater longevity compared to the Li-complex grease with the same additives, independent of sliding speed, contact pressure, temperature or type of sliding: continuous vs. reciprocating. The additive combination of ZDDP and MoDTC provided the best antiwear performance in both greases. Depending on the grease sample type, EDS revealed the presence of iron, zinc, phosphorous, sulfur, and molybdenum within the tribofilms.

1. Introduction

Greases are an important machine element widely used to reduce friction and wear in machinery. Thus, there exists an ongoing industrial demand for the improvement of their performance. Greases are comprised of several components: thickener, oil and various functional additives. Lithium soaps are the most commonly used type of thickener with a global production share in excess of 75% [1]. While lithium greases perform exceptionally well over a wide range of conditions, they tend to exhibit low rates of base oil bleed at low temperatures leaving the lubricating surface dry. In response to the demand for greases with a more constant bleeding rate over a wide range of temperatures, a polymer thickened grease has been developed [2,3]. In addition to enhanced low temperature performance, greases using polypropylene thickeners (referred to hereafter as polymer or polypropylene greases) demonstrate low frictional torque and excellent oil film build-up capability [4–9]. The possibility of further improvement to polymer grease performance via additives, however, has not been widely investigated outside of a single comparison of which found that the friction and wear reducing effect of zinc dialkyldithiophosphate (ZDDP) is less pronounced in polymer greases than for greases where a lithium complex thickener has been used (hereafter referred to as Li-complex greases) [10].

Although additives account for a relatively small portion of the whole lubricant formulation, they have a crucial role in friction and wear reduction, corrosion inhibition and oxidation prevention. Common additives like ZDDP and molybdenum dialkyldithiocarbamate (MoDTC) have been shown to have synergistic effects on the performance of paraffinic mineral oils [11], by effectively reducing friction and wear in conjunction [12,13]. More detailed explanations of the synergistic mechanism of these two additives have been provided by Refs. [14–16], who conclude that ZDDP hinders the formation of the high friction product MoO_3 and extends the friction-reduction time by slowing down the oxidation of low-friction product MoS_2. A more comprehensive description of the scope of the research on the combination of these two additives can be found in Ref. [17]. However, all of the aforementioned results have been obtained for the addition of ZDDP and MoDTC to various oils. Because the rheological properties of greases are more complex than those of oils, the performance of these additives is difficult to predict and is likely to be further influenced (and therefore complicated) by interactions between the additives and thickeners.

This work presents a thorough study on the tribological performance of ZDDP, MoDTC and their combination in a Li-complex grease and in a polymer grease. A combination of SEM, EDS and white light interferometry has been employed for the post mortem surface analysis of the wear scars to study changes in surface morphology caused by various grease/additive combinations.

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Table 1
Grease sample data.

Sample name	Thickener	Thickener content (wt%)	Base oil	ZDDP (wt%)	MoDTC (wt%)	NLGI grade
PP10	Polypropylene	12.5	PAO (93.7%)	0	0	II
PP10Z2	Polypropylene	12.3	+	2	0	
PP10OM2	Polypropylene	12.3	Adipate	0	2	
PP10Z1OM1	Polypropylene	12.3	ester (6.3%)	1	1	
LiX10	Li-complex	18.2	9.4 cSt @100 °C	0	0	
LiX10Z2	Li-complex	17.8		2	0	
LiX10OM2	Li-complex	17.8		0	2	
LiX10Z1OM1	Li-complex	17.8		1	1	

2. Experimental details

2.1. Materials

Polyalphaolefin (PAO10) oil was combined with polypropylene and Li-complex thickeners to make the PP10 and LiX10 greases. The production of the lithium complex greases was done in a pilot reactor with a batch size of 8 kg. The manufacturing procedure followed a standard protocol. The soap was synthesized in around 40% of the total oil volume. The first saponification using 12-hydroxystearic acid was performed at 90 °C with a hold time of 30 min. The temperature was then increased to 115 °C and held for two hours after which the second saponification using azelaic acid took place. The grease was then kept at 115 °C for another 30 min before the temperature was increased to 205 °C. Cooling was performed using 5% portions of oil with a hold time of 15 min between each addition. Finally, the grease was milled and deaerated for one hour.

The first phase in the manufacturing of the polymer grease is the melting of the polypropylene in the oil. This was carried out in a round flask with a mantle heater with a batch size of 1.2 kg. The polymer/oil solution was then quench cooled to a temperature far below the melting point of the polymers. In the second phase, four batches of quenched material were transferred to a mixing vessel, where the material was deaerated and worked into the desired consistency.

The additives were mixed uniformly into the greases using a DAC600 SpeedMixer. ZDDP and MoDTC were added in the proportions detailed in Table 1 to create eight samples, including the neat greases and samples containing a total of 2% additive by weight (1% of ZDDP and 1% of MoDTC in cases where both additives were included).

Tribological experiments were performed with a ball on disk configuration. The balls and disks were made of AISI 52100 bearing steel. The average disk surface roughness, *Ra*, was in the range of 0.046–0.060 μm. The average roughness, *Ra*, of the balls (3 or 6 mm in diameter) was 0.020–0.025 μm. Disk hardness was 60–62 HRC and ball hardness was 60–67 HRC.

2.2. Test procedure and conditions

In total, four separate sets of tribological experiments were completed, three with unidirectional and one with reciprocating sliding contacts to encompass a wide range of operating conditions. The unidirectional sliding tests were performed with a ball-on-disk geometry using a mini traction machine (MTM). Approximately 0.5 g of grease was used in each experiment, and a grease scoop was used to force the grease back into the contact. The reciprocating tests were carried out using a SRV4 test rig. In all cases, frictional data were collected for the duration of each experiment. New balls and discs were used for each test. The balls and disks were ultrasonically cleaned in an acetone bath for half an hour and then rinsed with isopropanol. For the reciprocating tests, the balls and discs were ultrasonically cleaned in an isopropanol bath and wiped dry

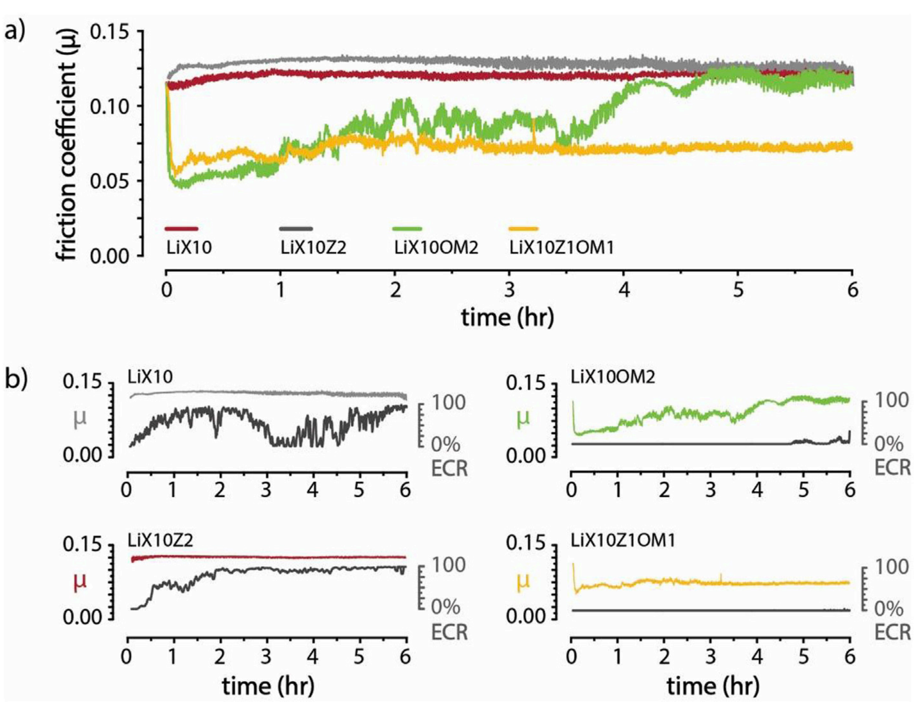

Fig. 1. (**a**) Friction traces for the Li-complex greases and (**b**) corresponding ECR data for each of the friction curves displayed for 6 h of unidirectional sliding (steel ball on steel disk at 80 °C, 60 N, 50 mm/s).

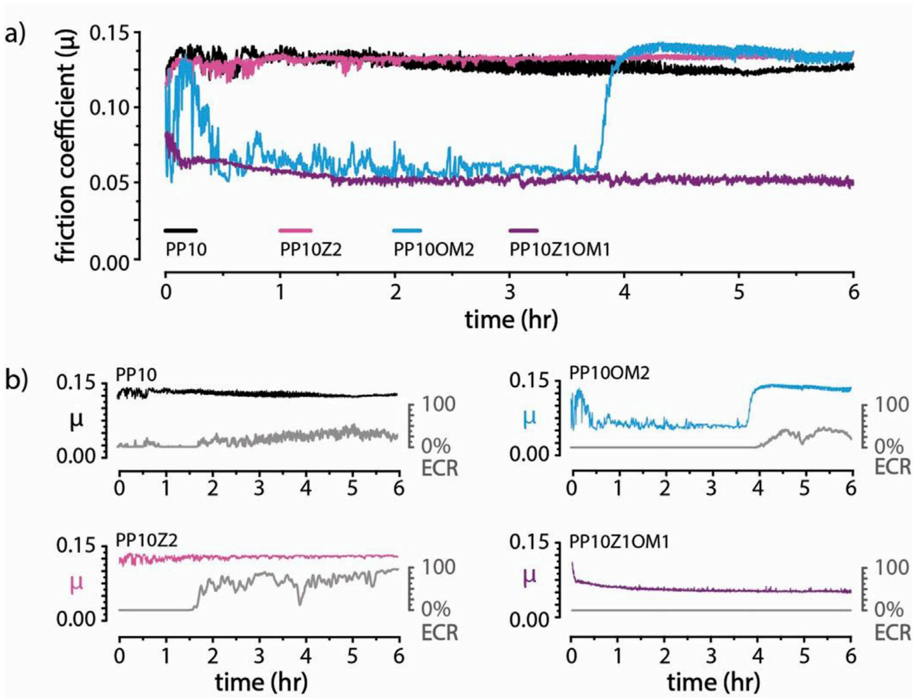

Fig. 2. (**a**) Friction traces for the polypropylene greases and (**b**) corresponding ECR data for each of the friction curves displayed for 6 h of unidirectional sliding (steel ball on steel disk at 80 °C, 60 N, 50 mm/s).

with dust free tissues.

In the first set of experiments in the MTM, a continuous sliding contact was formed by a rotating disc and a stationary ball (6 mm diameter) loaded to 60 N (2.57 GPa). Sliding proceeded at 50 mm/s for six hours at 80 ± 1 °C. Each test was repeated three times in order to compare the effects of the two additives on their own, and in conjunction. Electrical Contact Resistance (ECR) data were also collected for each of these tests.

Wear scar diameters (WSDs) were measured by using an optical profilometer (Zygo, 7300). Each WSD value presented in the Results section is an average value of six measurements (3 times in X direction

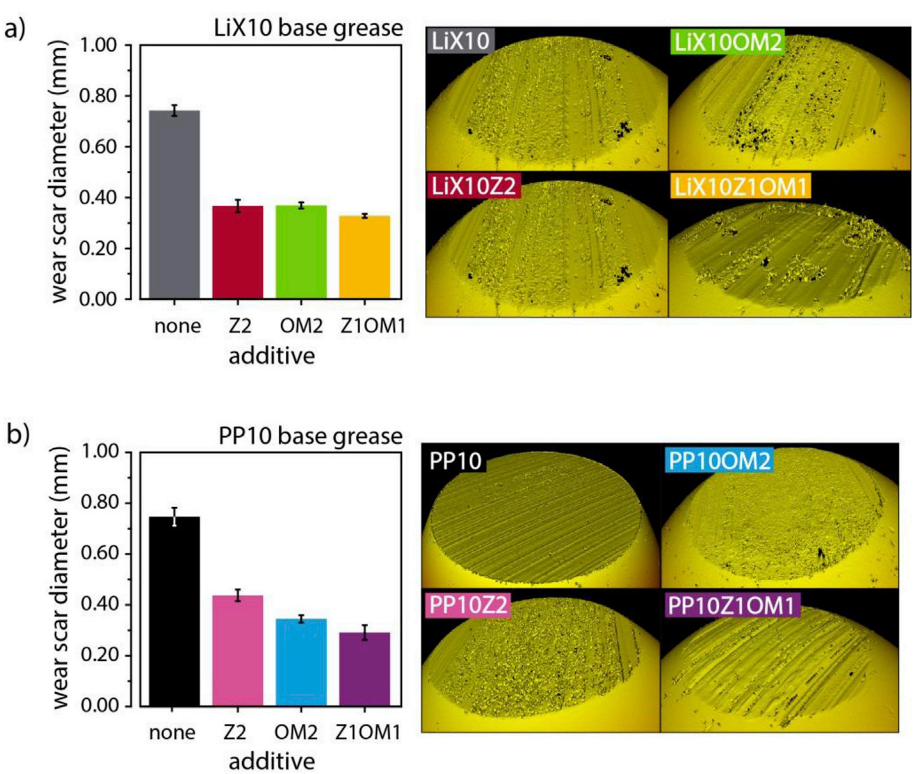

Fig. 3. Wear scar diameters/wear rates and corresponding optical profilometry scans of the wear scars on steel balls tested in (**a**) neat and modified Li-complex greases and (**b**) neat and modified polypropylene greases after 6 h of continuous sliding on a steel counter surface at 50 mm/s and 60 N at 80 °C.

and 3 times in Y direction). A JEOL 7800 F Scanning Electron Microscope (SEM) was used to image the wear scars on the balls. Elemental analysis of the tribofilms formed on the balls was performed in the same SEM using a Bruker Quantax Energy Dispersive X-ray Spectroscopy (EDS) system.

In the second set of experiments, using the MTM with the same ball-on-disk configuration and normal load, hour long tests were run (again in triplicate) at three temperatures (40 °C, 80 °C and 120 °C, at 50 mm/s) and two speeds (50 mm/s and 200 mm/s, at 80 °C). The upper temperature limit was set by the highest allowable continuous operating temperature for the polymer grease [18]. These tests were limited to the neat greases, and the greases containing 1 wt% each of ZDDP and MoDTC (PP10, PP10Z1OM1, LiX10, and LiX10Z1OM1).

Third, the neat greases and the greases containing both additives were tested in the same continuous sliding arrangement at a range of contact pressures. To achieve higher contact pressures, a 3 mm ball was used in the load tests. Each system was allowed to run in for an hour under a 10 N (2.25 GPa) load, and the same temperature and sliding speed (80 °C, 50 mm/s) used in the first set of MTM tests. After run-in was complete, the load was increased in 20 N steps up to a final load of 70 N. Each load was held for five minutes before proceeding to the next step. These tests were also limited to the neat greases, and the greases containing 1 wt% each of ZDDP and MoDTC.

The final experimental set was carried out using the SRV4 according to ASTM D5707-16, but with an extended running time of 6 h. A 10 mm ball was pressed against a steel disk producing a maximum contact pressure of 2.74 GPa. The ball oscillated with a 1 mm stroke at 50 Hz at 80 °C.

3. Results and discussion

3.1. Direct comparison of additive combinations

Representative friction traces were selected for each sample in the six-hour continuous sliding experiments and are provided in Fig. 1a for the Li-complex greases and in Fig. 2a for the polymer greases. On average, neat PP10 returned slighter higher friction (~0.125) as compared to neat LiX10 (~0.115). As expected, the addition of ZDDP on its own (LiX10Z2, PP10Z2) did not significantly affect the frictional behavior of either grease. In contrast, the addition of MoDTC on its own (LiX10OM2, PP10OM2) significantly reduced the average friction for both greases. The LiX10OM2 lubricated contacts demonstrated low friction at the beginning of the tests, but friction gradually increased over time to the higher values measured for LiX10 and LiX10Z2. The contact lubricated with PP10OM2 experienced relatively high, unstable friction during the first thirty minutes of the test before maintaining lower, more stable friction for the next three to four hours (depending on the test) and eventually returning to the higher friction of PP10 and PP10Z2. Both greases benefitted immensely from the addition of a combination of ZDDP and MoDTC, maintaining significantly lower friction throughout the duration of the tests.

Figs. 1b and 2b superimpose the corresponding ECR data for each test below the friction traces. The ECR parameter represents a relative value of contact resistance from 0% (conductive) to 100% (nonconductive).

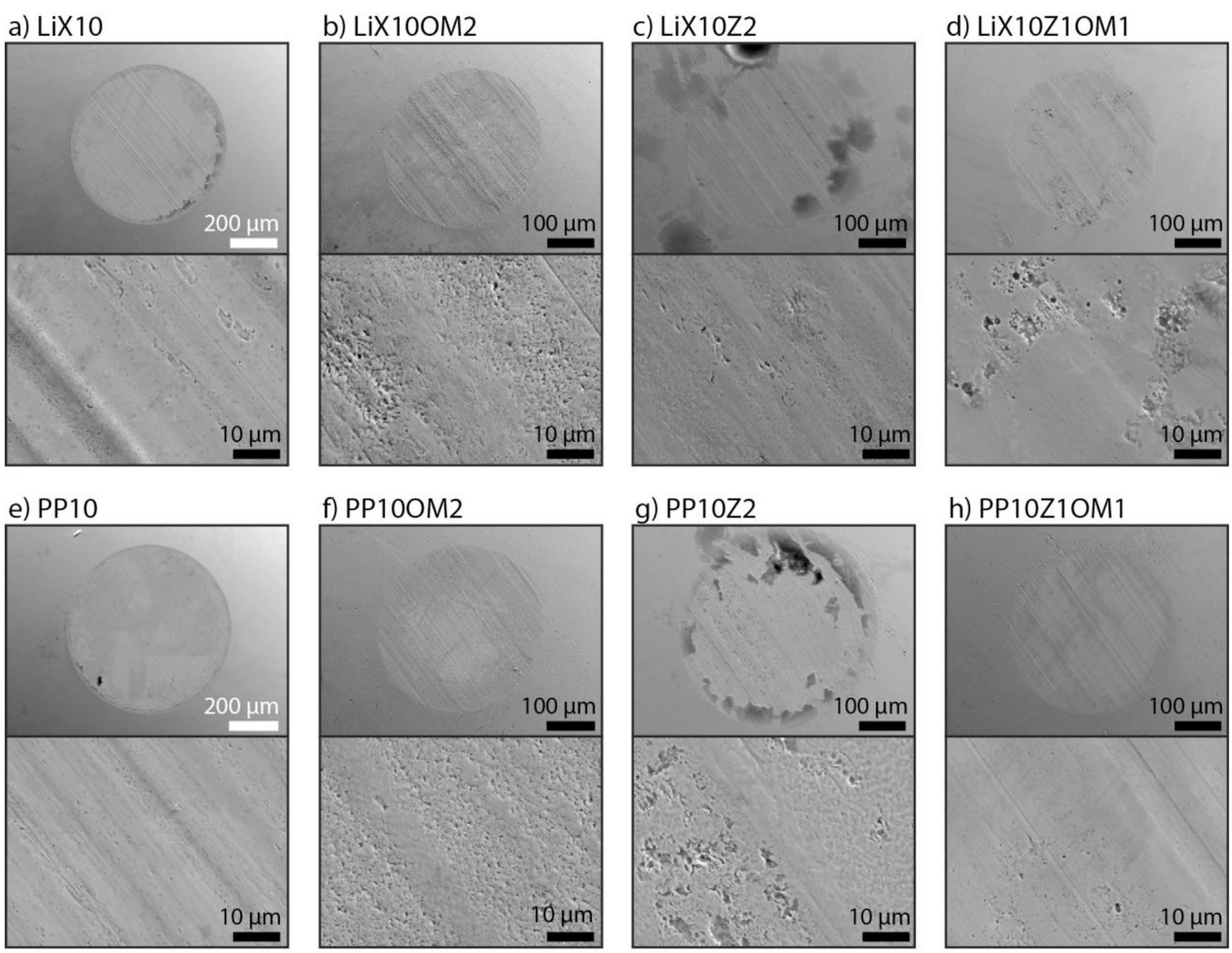

Fig. 4. SEM images of wear scars on balls tested in each of eight grease formulations after 6 h of continuous sliding on a steel counter surface at 50 mm/s and 60 N at 80 °C. (**a-d**) Li-complex greases (**e-f**) polypropylene greases.

The raw ECR signal was conditioned by using a moving average filter (10 points). For the neat greases, presence of a thickener in the contact is detected by the ECR system (high resistance). Li-complex thickener appears to enter the contact in the very beginning of the test, while it takes quite a long time for the polymer thickener. This may explain the slightly higher coefficient of friction for the PP10 grease. The polar nature of the Li-complex thickener facilitates its adsorption on the disk surface. The ECR system reveals a gradual increase in contact resistance reliably detecting a boundary layer formation by ZDDP as was also observed by Ref. [19]. The onset of the tribofilm build-up is clearly visible for the PP10Z2 grease but masked for the LiX10Z2 grease by the thickener entrainment. Contrary to the ZDDP induced tribofilm, the boundary layer formed by MoDTC is conductive. A possible decomposition pathway, and the reaction kinetics of MoDTC in Group III mineral oil were proposed by Khaemba, Neville, and Morina in 2016 [20]. They propose that shear stresses in the contact lead to scission of the C-S bonds and subsequently the formation of MoS_x ($x > 2$) that is then converted to MoS_2. The durability of the low friction MoS_2 boundary layer is clearly affected by the thickener type, as shown in Figs. 1 and 2. Four hours into the PP10OM2 test, the balance between MoS_2 formation and removal processes for the polymer grease is suddenly lost leading to the breakdown of the low friction boundary film. This transition is evident in both the friction and ECR measurements. In contrast, the "off-balancing" process for the Li-complex grease is gradual.

A combination of ZDDP and MoDTC in oils enhances durability of the low friction tribofilms. Such films are composed of a mixture of glassy zinc/molybdenum phosphates, molybdenum, carbon rich zones, and dispersed MoS_2 single sheets [16]. The upper layer of the tribofilm is composed mainly of MoS_2 sheets, some of which are aligned parallel to the shear plane [21]. The low friction is induced by these favourably oriented MoS_2 sheets. Li-complex thickener may interfere with the formation of such sheets, leading to higher friction compared to the polymer grease.

WSDs and optical images of the worn surfaces on the balls are shown in Fig. 3. A significant reduction in WSD is achieved by adding ZDDP to greases, with all other test parameters held constant. The anti-wear performance of the Li-complex grease is slightly better than that of the polymer grease, a similar finding to that reported in Ref. [10]. It has also been reported that Li-complex thickener can interact with ZDDP [22]. Therefore, it may follow that the Li-complex thickener brings ZDDP to the surfaces promoting quicker formation of anti-wear boundary films.

MoDTC in the PP10 grease further reduces WSD, but in the Li-complex grease performs comparably to ZDDP. The best anti-wear performance is achieved when both additives are used in the greases. As compared to the anti-wear performance of the individual additives, there is only a slight improvement for the Li-complex grease when the two are added in conjunction. For the polymer grease, the improvement is significant. A closer look at the wear scars using SEM images, Fig. 4, does not reveal further differences between the greases.

The EDS analysis is summarized in Fig. 5. All spectra are normalized to the Iron Kα peak at 6.398 keV. A comparison of the spectra over the 0–10 keV range is provided in Fig. 5a. Evidence exists for the presence of iron, zinc (in all but the –OM2 samples), phosphorous (in the PP10Z2, LiX10Z2, PP10Z1OM1 and LiX10Z1OM1 samples), sulfur (in all samples), and molybdenum (in all but the –Z2 samples) within the tribofilms, though accurate quantitative comparison of film composition is not possible due to the assumed thickness of the film being much less than the excitation depth of the electron beam, and due to the assumed variation in tribofilm thickness between samples. In cases where the tribofilm is extremely thin, elements may be present but fall below the detection

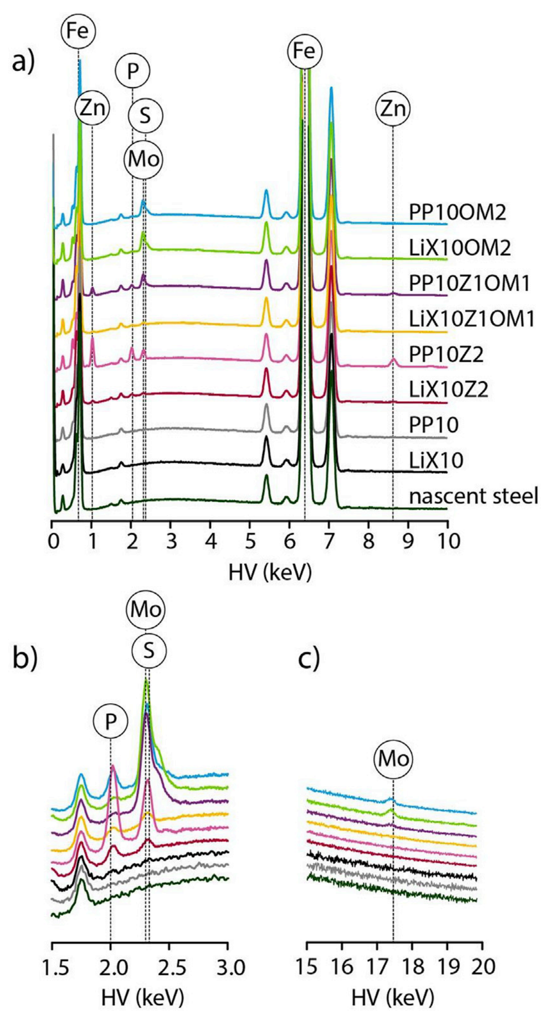

Fig. 5. EDS spectra taken at 30 keV over areas approximately 20 × 40 μm in dimension in the center of each of the wear scars in Fig. 4.

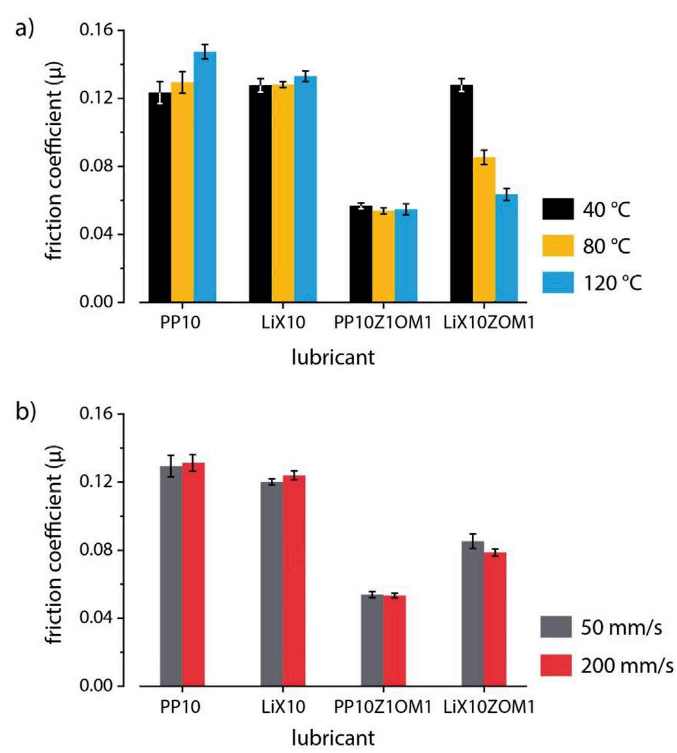

Fig. 6. Friction coefficients averaged over the last five minutes of three one hour long tests for neat Li-complex and propylene greases, and for those same greases with the addition of 1 wt% each ZDDP and MoDTC, are reported at (**a**) three temperatures (v = 50 mm/s) and (**b**) two speeds (T = 80 °C).

limit of the instrument within the excitation volume. As illustrated in Fig. 5b, a closer inspection of the 1.5–3.0 keV range of the spectra reveals overlap of the sulfur Kα and molybdenum Lα peaks. Deconvolution of these peaks is possible using software. In addition, spectra were acquired up to 30 keV in order to capture the molybdenum Kα peak at 17.441 keV (Fig. 5c) to avoid ambiguity. This peak is quite weak, and is not visible at this scale in the case of LiX10Z1OM1. However, software deconvolution of the overlapping peaks around 2.3 keV, combined with the acquired intensities at higher voltage did suggest that molybdenum is present at low atomic percentages (~1%) within the excitation volume on the worn –OM2 and –OM1 samples.

3.2. Influence of operating conditions on friction reduction

The hour long unidirectional sliding tests of contacts lubricated with the neat greases and the greases containing 1 wt% each of ZDDP and MoDTC sought to elucidate the effects of temperature and sliding speed on the performance of the greases. Fig. 6a illustrates the results of temperature variation (40, 80 and 120 °C, at 50 mm/s). The average friction coefficient measured in the neat PP10 lubricated contacts increased around 20% between 40 and 120 °C. In the neat LiX lubricated contacts, a lesser overall frictional increase was observed – around 4%, which lies within the error of the measurement. The polymer grease with both ZDDP and MoDTC added (PP10Z1OM1) performed similarly well at all three temperatures tested and outperformed the other formulations by a fair amount, resulting in a nearly 60% reduction in friction as compared to the neat polymer grease, PP10. At 40 °C, the performance of the lithium complex grease containing both additives (LiX10Z1OM1) was comparable to that of the neat grease, LiX10. However, a sharp decrease in average friction coefficient was observed with increasing temperature. At 120 °C, the average measured friction in the LiX10Z1OM1 lubricated contact dropped to half its value at 40 °C. Higher friction at lower temperature maybe due to the formation of MoS_x and $FeMoO_4$ as reported by Refs. [20,23]. Increasing temperature helps in converting MoS_x to MoS_2. In case of the PP10Z1OM1, the polymer thickener may prevent formation of $FeMoO_4$ and facilitate growth of MoS_2 layers. Fig. 6b compares the frictional results at two different speeds (50 and 200 mm/s, at 80 °C). Neither the neat greases nor the greases containing both additives demonstrated a significant change in friction at the two speeds tested.

The load ramp experiments (Fig. 7) did not show a significant dependence of friction on contact pressure over the range of pressures tested for either the neat greases or for the greases containing both additives. A small drop in friction was observed in the LiX10Z1OM1 lubricated contact after the first load increase, but this lower value was not maintained throughout the duration of the test. Both of the greases containing both ZDDP and MoDTC outperformed the neat greases at all loads tested.

The performance of the neat greases and the greases containing both ZDDP and MoDTC under reciprocating motion is summarized in Fig. 8. Each friction trace is an average of three friction curves. In general, the friction levels (Fig. 8a) are higher (except for PP10Z1OM1) than those obtained from the continuous sliding. The friction performance of the polymer grease is improved dramatically by the inclusion of ZDDP and MoDTC in conjunction; at the same time, the average steady state friction value for the PP10Z1OM1 lubricated contact (μ~0.055) is 50% lower than that for the LiX10Z1OM1 lubricated contact (μ~0.108) - a marked difference as compared to the relatively identical performance of these samples in the unidirectional sliding tests. Despite a relatively large disparity in friction coefficient, the WSDs on the balls from the modified greases were comparable, Fig. 8b.

The difference in friction results for the continuous and reciprocating sliding tests may be due to the different contact replenishment conditions. The grease scoop used in the continuous sliding tests assured fully flooded conditions at the entrance to the contact while in the reciprocating tests lubrication was provided by the oil bleeding. Higher temperatures increase oil bleeding. In the SRV tests the PP10Z1OM1 grease

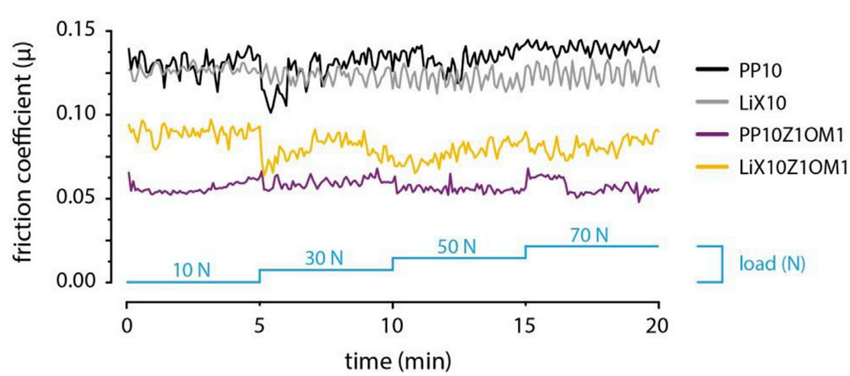

Fig. 7. Representative friction traces at increasing contact loads (80 °C, 50 mm/s) for neat Li-complex and propylene greases, and those same greases with the addition of 1 wt% each ZDDP and MoDTC.

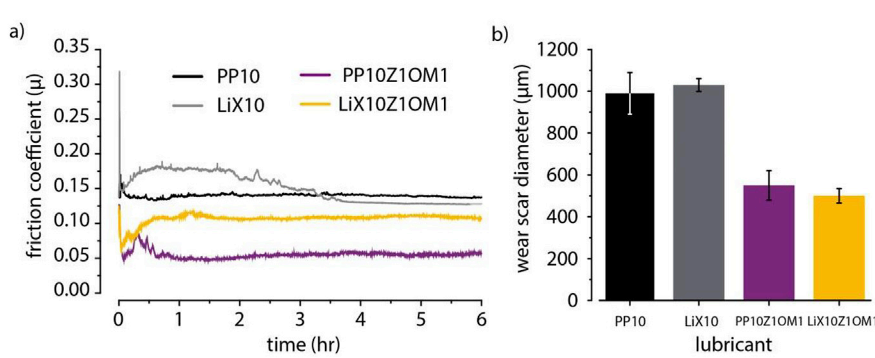

Fig. 8. (**a**) Friction traces in reciprocating sliding (1 mm stroke, 50 Hz, maximum 2.74 GPa, 80 °C) and (**b**) accompanying wear scar diameters/wear rates of the balls for contacts lubricated with neat Li-complex and propylene greases, and for contacts lubricated with those same greases with the addition of 1 wt% each ZDDP and MoDTC.

provided a consistent reduction in friction, from 0.08 at 40 °C to 0.05 at 120 °C, similar to the results reported for an engine oil [24]. However, the LiX10Z1OM1 grease showed an opposite trend: an increase in friction, from 0.08 at 40 °C to 0.12 at 120 °C. If these results are compared with the results obtained in the continuous sliding tests it becomes apparent that significant interactions exist between the Li-complex thickener and the additives. The nature of these interactions is complex [22] but they lead to the lower additive concentration in the bleed oil and render the Li-complex thickener less efficient compared to the polypropylene thickener.

4. Conclusions

Additive response in the Li-complex and polymer greases in terms of friction and wear reduction was compared for a range of loads, speeds, and temperatures as well as for two types of sliding: continuous and reciprocating. The greases, based on PAO10, were tested both as neat greases and with a 2 wt% addition of ZDDP and/or MoDTC. The results show that thickener plays an important role in the tribofilm formation process and its subsequent durability. Depending on the grease sample type, EDS revealed the presence of iron, zinc, phosphorous, sulfur, and molybdenum within the tribofilms.

The general conclusion of this study is that a combination of ZDDP and MoDTC in polypropylene grease provided a synergistic effect in terms of the highest friction reduction independent of operating conditions or type of sliding.

Specific conclusions for the continuous sliding are as follows.

1. Polypropylene grease with ZDDP and MoDTC demonstrated much lower friction with greater longevity compared to the Li-complex grease, independent of speed, load and temperature.
2. Li-complex grease with added ZDDP and MoDTC showed a reduction of the friction coefficient with increasing temperature. At 40 °C the friction coefficient was similar to the neat grease, whereas at 120 °C the friction coefficient was comparable to PP grease with these additives.
3. The additive combination of ZDDP and MoDTC provided the best anti-wear performance in both greases. There was an essential improvement in the anti-wear performance of polypropelene grease over the individual additives but only a marginal improvement for the Li-complex grease.

Specific conclusions for the reciprocating sliding are as follows.

1. The friction reducing performance of the ZDDP and MoDTC additives is much less efficient in the Li-complex grease compared to the polypropylene grease.
2. The wear trends for both greases are similar to the continuous sliding tests despite a relatively large disparity in friction coefficients.

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