Serving the grease industry since 1933

NLGI

India Chapter

ISSN : 0972-2742

GREASETECH INDIA

A Quarterly Journal of NLGI-India Chapter

Vol. XXII, No. 3, Jan - Mar 20

GREASETECH INDIA

A Quartely Journal of NLGI-India Chapter

Vol. XXII, No. 3, Jan - Mar 20

In This Issue | Page No

Rust for the Record: Significant Factors Affecting Corrosion Protection in Grease

Joseph P. Kaperick Afton Chemical Corporation Richmond, VA

Abstract

Experiments were performed with standard static corrosion tests to investigate effects of lithium grease formulation on corrosion resistance. The alkalinity of three unadditized base greases due to excess unreacted lithium hydroxide did not correlate with visual estimates of corrosion surface area. There was some evidence that alkalinity affected performance of two rust inhibitors, a neutral polyester succinimide and an acidic succinate ester. Other evidence showedthat components of an additive package enhanced corrosion inhibition by these two rust inhibitors while the lithium thickener system interfered with these rust inhibitors. Two different borate amides and a boronated dispersant improved the performance of the rust inhibitors formulated in a lithium base grease. A visual rating system for evaluating the percent corroded surface area on bearing races was used in this study.

Introduction

This paper continues work previously presented by the author on the subject of the alkalinity of lithium greases and the impact of alkalinity on additive effectiveness.[1] As noted in that publication, "The direct cost of metallic corrosion is $276 billion *on an annual basis,* which represents 3.1% of the U.S. Gross Domestic Product (GDP)" [2]. This subject is of great interestto grease formulators and grease consumers who face the challenge of preventing rust with their products.

As pointed out in the previous study, Hunter and Baker [3,4] evaluated different rust inhibitors and greases using two static rust tests (ASTM D1743 and D5969) and the dynamic EMCOR corrosion test (ASTM D6138). They reported good correlation between results from the tapered bearing static test and the EMCOR dynamic test, both in 5% synthetic sea water. Hunter et al. also tested ashless rust inhibitors in the presence of different grease thickeners using these methods in 2000.[5]

The present author's original study used a variety of performance tests to measure differences in effectiveness of two additive packages (containing rust inhibitors) in acidic and alkaline base greases. Results from D1743 showed that the basic grease was inherently corrosive to steel, while the acidic base grease was not (Figure 1). The D1743 test was run in triplicate with each bearing being rated as a "Pass" or a "Fail". A rating of "Fail" was assigned when an area of corrosion greater than 1.0 mm in diameter was observed on the bearing. Pack 1 did not improve corrosion resistance of the alkaline grease. Pack 2 improved the performance of the alkaline grease and may have had a positive effect on the acidic grease.

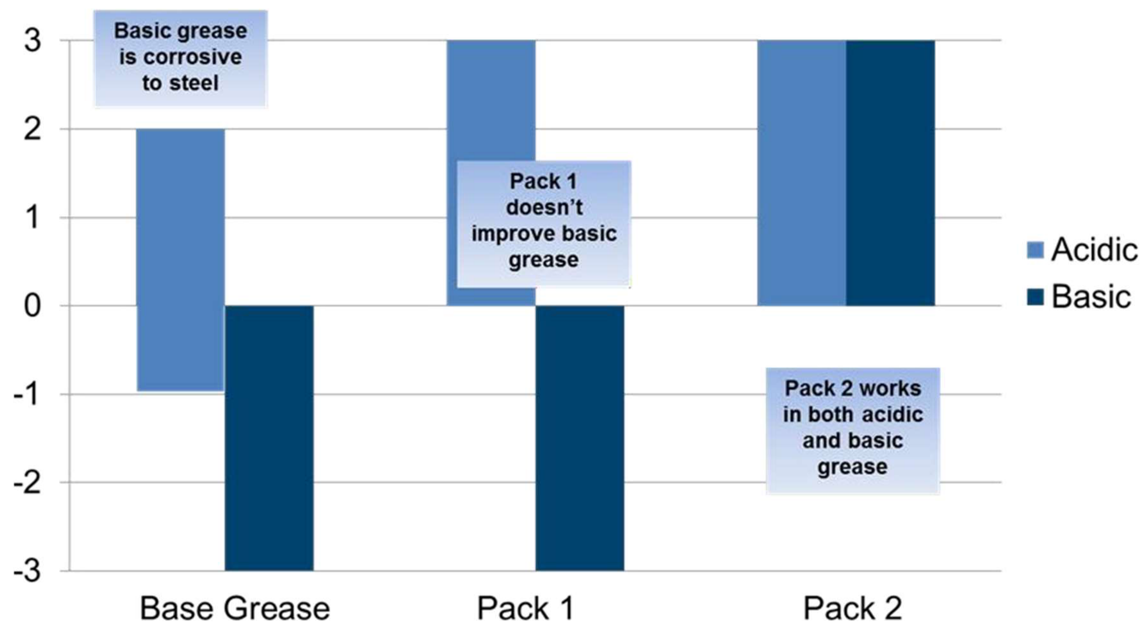


Figure 1- Bearing Rust (ASTM D1743) 52 °C, 48h- Response of Additive Packages in Acidic and Alkaline Base Greases. This graph shows the number of bearings that passed or failed from triplicate runs.

A case study showed the difficulty of using rust inhibitors to prevent corrosion in a highly alkaline grease. An additional study investigated the impact of lithium hydroxide (LiOH) and 12-hydroxystearic acid (12-HSA) on corrosion in the absence of grease thickener (Table 1 and Figure2).

Base Grease: **Rust Test:**	**Straight lithium** **Emcor, Distilled water (ASTM D6138)**							
Additive Package (EP/AW/RI) (Wt %)	**4**	**2**	**2**	**2**	**2**	**2**	**2**	**2**
DDSA Derivative (Wt %)		**0.50**						
Overbased Ca-sulfonate (Wt %)			**0.50**	**1**				
Ca-DNNS (Wt %)					**0.50**	**1**		
Zn-DNNS (Wt %)							**0.50**	**1**
Emcor Rating	**4**	**3**	**4**	**3**	**3**	**2**	**4**	**3**
Li Content of Water After Test, ppm	147	246	206	245	181	266	195	267
As %LiOH leached from Grease	0.102	0.171	0.143	0.171	0.126	0.185	0.136	0.186

Table 1 – Case study - Effect of highly alkaline base grease (0.17% LiOH) on rust inhibitor ability to prevent corrosion. Li content was determined by aqueous ICP of post-test water, and % LiOH was calculated from this determination.

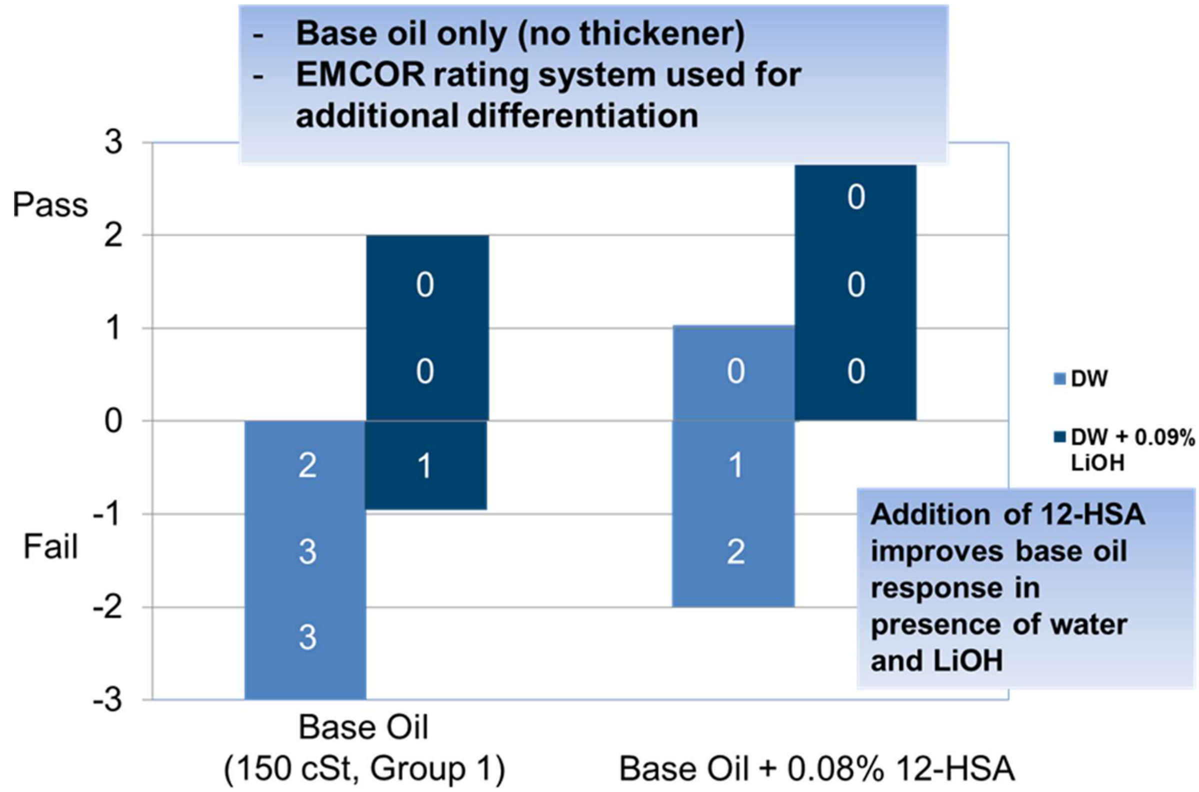


Figure 2 - Effect of LiOH and 12-HSA on corrosion protection in base oil. Ratings are shown on the bar chart for individual bearings of triplicate runs and are based on the D6138 rating scale from 0 [no rust] to5 [>10% surface coverage].

This study seemed to indicate that LiOH (added to the test water) did not directly attack steel, and that increased ionic activity in the water did not cause more corrosion. This led to a hypothesis that there was a likely interaction between the alkali grease thickener and the rust inhibitors that prevented them from working together as well as expected.

Additional results from a separate study by the present author [6] also indicated that the lithium grease thickener by itself did not prevent corrosion (Table 2). It was hypothesized that this thickener might prevent water from physically getting to the metal surface. However, that earlier study was done with the EMCOR corrosion test, a more dynamic test than the static corrosion tests used in the other work discussed here.

Test	Component	#1 (% wt)	#2 (% wt)	#3 (% wt)
	Pack C	3.5	3.5	3.5
	Lithium Base Grease #1	96.5		
	Lithium Base Grease #2		96.5	
	Base oil (in Base Grease #2)			96.5
	Total	100	100	100
EMCOR (50% SSW)	ASTM D6138	3/2	3/2	2/2

Table 2 - No significant difference seen in rust protection between base grease and base oil

Previous work did point to an interaction between rust inhibitor additives and the lithium thickener as the likely explanation for variations seen in corrosion prevention effectiveness.

Experiments

The present study examines the interaction of base greases with corrosion inhibitors in more detail and seeks to better understand the impact of alkalinity. The first part of this study focuses on the standard static bearing corrosion test for grease, which is described by two ASTM standards, one using only distilled water (ASTM D1743) and one using various salt water solutions (ASTM D5969).

The second part of this study looks at the impact of borates on grease thickener systems and their constituent components in terms of their ability to prevent rust and enhance other performance requirements of lubricating greases.

Test Methods

ASTM D1743 "Standard Test Method for Determining Corrosion Preventive Properties of Lubricating Greases" [7]

This test utilizes a tapered roller bearing with a Timken bearing cone/roller assembly and cup which is packed with grease, run in, exposed to distilled water and let sit for 48 ± 0.5 hours at 52 ±1˚C in an atmosphere of 100% humidity.

ASTM D5969 "Standard Test Method for Corrosion-Preventive Properties of Lubricating Greases in Presence of Dilute Synthetic Sea Water Environments" [8]

This test is identical to ASTM D1743 with two exceptions: it runs for only 24 hours, and it uses diluted synthetic sea water (SSW) prepared as specified in ASTM D665. In this study, dilutions of 5, 50 and 100% SSW were used.

According to ASTM D1743 and D5969, a bearing with no spots larger than 1.0 mm in diameter is considered a pass, and two out of three bearings must pass for the grease to be considered acceptable, as shown in Table 3.

Rating	EMCOR (D6138)	D1743/D5969	Mod. rating for study
Pass		No spot >1.0mm (2/3 bearings)	
Fail		Any spot >1.0mm; (2/3 bearings)	
0	No rust visible		Pass
1	≤3 spots; all smaller than 1 mm dia.		Pass
2	Up to 1% surface coverage		1%
3	1 - 5% surface coverage		5%
4	5 – 10% surface coverage		10%
5	>10% surface coverage		10-100%

Table 3 - Rust ratings from ASTM D6138, ASTM D1743/D5969 and modifications for this study

In contrast, the EMCOR corrosion test (ASTM D6138) uses a numerical rating system with "0" indicating no visible rust while "5" indicates more than 10% surface coverage by corrosion.
In the present study, a modified system of rating bearings from D1743/D5969 tests was employed in order to better estimate the impact of each variable on the level of corrosion present. The raceway on the inside of each bearing cup was rated on the basis of a visual estimate (without the use of magnification) of the percent surface area covered by rust. This rating method is illustrated in Figure 3. To minimize variability in this modification, a single technician did all of the evaluations used in this study. Although no formal statistical analysis was possible, a single fully formulated grease sample (**#1 Base A**) was evaluated by this modified method three separate times; seven of the nine bearings rated gave results that were within 10% (surface area corrosion) of the mean, which was 20% (Figure 7). For further discussion on variability of this modification in fully formulated greases, see Additive Packages and Figure 6.

Figure 3 - Modified rust rating used in this study - percent surface coverage of corrosion

Grease Samples

The base greases used for the work done in this study (**Base A, Base B, Base C**) were made inthe author's facility using a lab-scale, covered and jacketed 5-gallon kettle operated at atmospheric pressure with a single-motion, anchor-style agitator with scraper blades and fixed vertical baffle attached to the bottom of the lid cover. Heating and cooling were achieved by the circulation of heat transfer oil through the kettle's jacket using a loop consisting of an oil reservoir,pump, heater and heat exchanger. The kettle was connected to a second pump used to circulatethe contents of the kettle through a colloid mill, to provide additional agitation, and to discharge the final product. The operation of the entire unit was computer controlled. All greases were degassed before testing. Degassing was done in shallow pans placed in a vacuum oven held at 29 inches Hg. For the base greases, the oven temperature was held at 60 °C and the greases were kept in the vacuum oven for 1 day. For the formulated greases, the oven temperature washeld at 80 °C and the greases were kept in the vacuum oven for 30 minutes.

Lithium 12-hydroxy stearate greases were prepared with a blend of ISO 150 paraffinic Group I oils and adjusted to NLGI #2 grade. Different levels of alkalinity were obtained by adjusting the amount of LiOH used in making the base greases (Table 4). The alkalinities are reported in %LiOH (not $LiOH\bullet H_2O$) as calculated by ASTM D128, Section 21 – Free Alkali [9].

Grease	Alkalinity (% LiOH)
Base A	0.066
Base B	0.057
Base C	0.031

Table 4- Base grease alkalinity

One fully formulated grease (**Fully Formulated #1**) was prepared for this study using a commercial grease additive package that contained ZDDP, a sulfur source, two rust inhibitors anda synergistic antioxidant combination. To further study the impact of separate components, the two individual rust inhibitors were used in the same ratio and concentration as in the package. The inhibitors are a neutral polyester succinimide and an acidic succinate ester.

Finally, the impact of borate chemistry was investigated using three borates, two borate amides and a boronated dispersant.

Alkalinity

To establish baseline performance, the three base greases were tested by ASTM D1743 and showed an average of about 30% surface rust. Some variation was seen in the evaluations of percent surface area corrosion with a notable anomaly for Base A, where one of the bearings showed no corrosion at all while the other two had 40 and 50%. The need for triplicate analysis is obvious from this example. There didn't appear to be a correlation between the alkalinity and the amount of rust present (Figure 4).

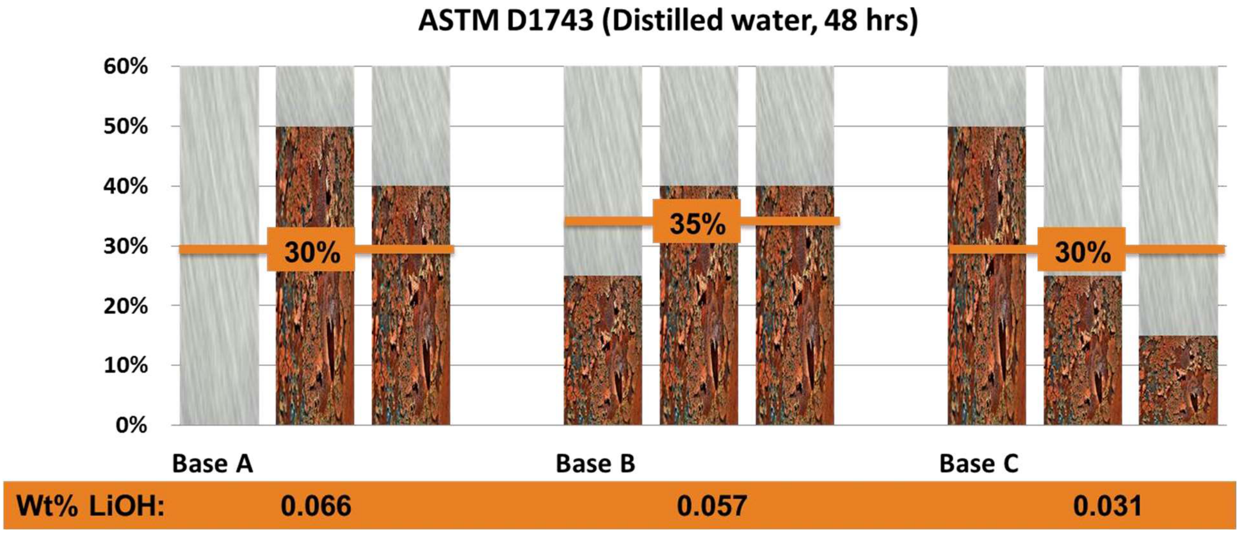


Figure 4 - Base grease rust response – percent area of corrosion on bearing race

Base C (with an alkalinity of 0.031 %LiOH) was then used to make **Fully Formulated #1** and study the effect of salt water concentration and immersion time on corrosion. **Base C** and the additive package were chosen because their performance in a fully formulated grease was borderline in previous ASTM D1743 testing, so that positive or negative effects were seen.

Test Parameters

As shown in Figure 5, the additive package significantly reduced the amount of corrosion in standard testing with distilled water over 48 hours. However, when either the time or salt water concentration was increased, the amount of corrosion also increased. Raising the salt water concentration from 5 to 100% resulted in an increase in rust coverage from 23 to 42% for 24 hour tests. Extending the time from 24 to 48 hours increased coverage from 23 to 30% (in 5% SSW solution) and from 42 to 63% (in 100% SSW solution). The amount of corrosion appeared to plateau around 70% in 50 and 100% SWW.

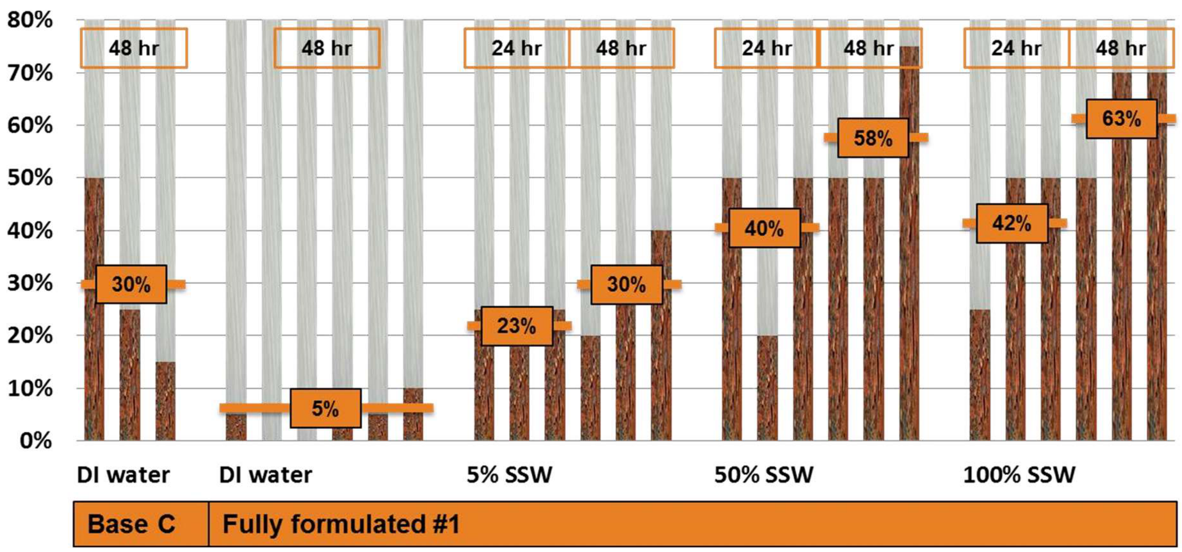


Figure 5 - Increasing test severity - time and salt water concentration.

Additive Packages

To investigate the repeatability of the rating method used in the this study, **Formulation #1** was made up four times in **Base A (#1A, #1B, #1C, #1D**) and tested by ASTM D1743 (Figure 6). These results showed an overall reduction in corrosion due to the use of the additive package. Results ranging from 13 to 23% corrosion surface coverage with the additive package versus 30% for the base grease (**Base A**).

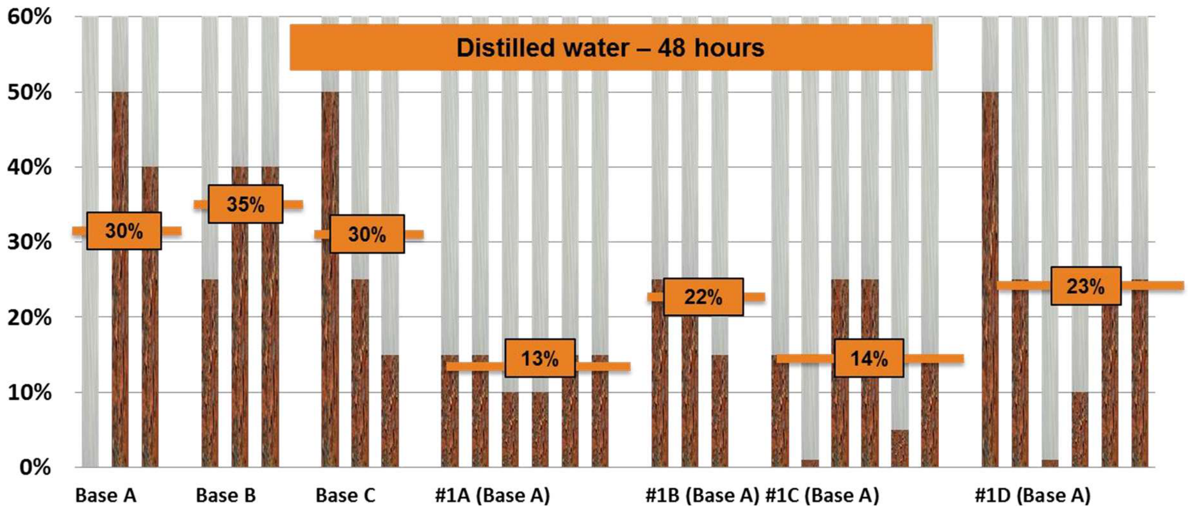


Figure 6 - Repeatability of surface coverage rust rating

Next, the effect of the alkalinity of the base grease on fully formulated greases was studied. Each of the three base greases was blended with the additive package #1 [**#1 (Base A), #1 (Base B),#1 (Base C)]** and tested with ASTM D1743. As seen in Figure 7, it appears that the lowest alkalinity base grease (**Base C**) responded more favorably to the additive package than the other two base greases.

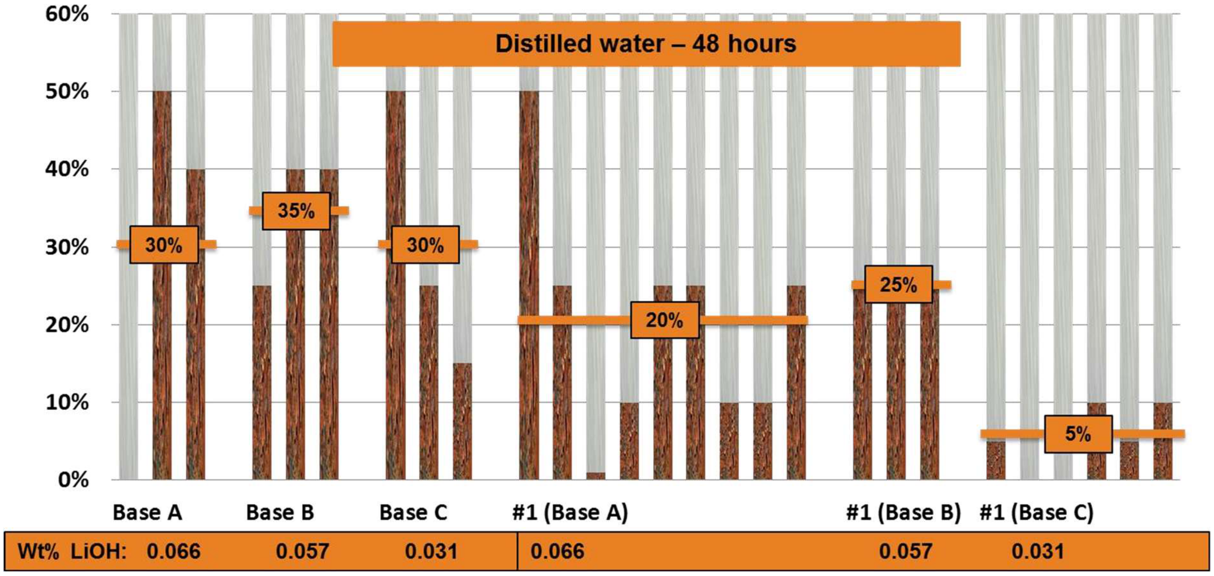


Figure 7- Effect of alkalinity on fully formulated greases

To further explore this possible influence of alkalinity on corrosion protection, each base grease was treated with only the rust inhibitors from the additive packages (Figure 8). For **Base A**, results were comparable – 20% corrosion for Additive 1 (package) versus 18% for the two additives.

In **Base B** and especially **Base C**, the rust inhibitors gave less protection from corrosion than Additive 1. In **Base A,** the results were almost identical (20 vs 18%) but in both **Base B** and **Base C**, the rust inhibitors on their own gave worse results than when the rest of the package was present in the formulated grease (25 vs 43% for **Base B** and 5 vs 17% for **Base C**).

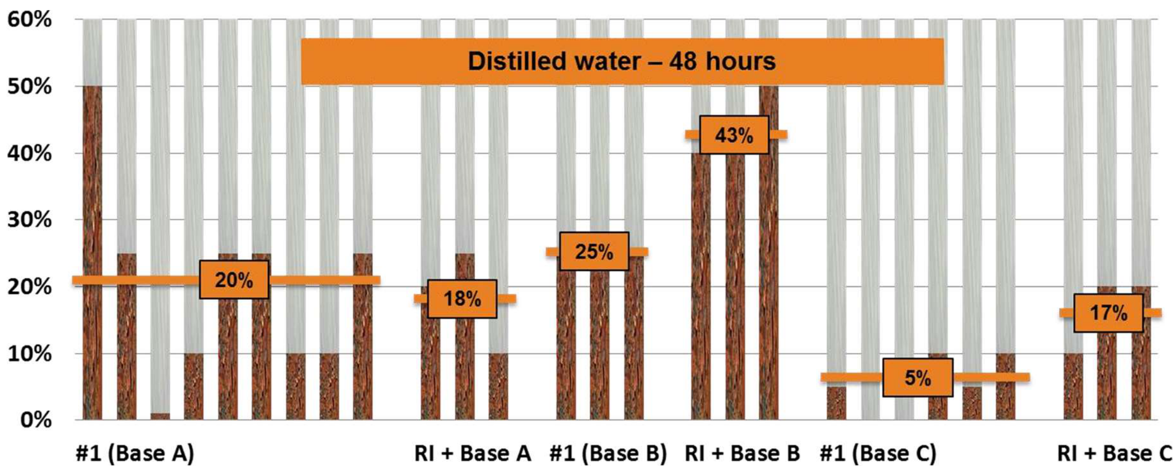


Figure 8 - Rust inhibitors as components of a package and as individual additives

At this point, two possible explanations for the behavior seen in this experiment were considered. First, the ZDDP and sulfur components in Additive 1 could provide additional rust protection or act synergistically to improve the performance of the rust inhibitors. Or second, ZDDP or sulfur compounds could react with excess LiOH and thereby allow the rust inhibitors to be more active. This latter explanation would mean that excess LiOH reacted with the rust inhibitors or otherwiseinterfered with corrosion inhibition.

Lithium Hydroxide Interactions

To further study possible interactions between LiOH and rust inhibitors, this experiment was repeated without the grease thickener. As can be seen in Figure 9, in tests in distilled water, results for corrosion were similar for base oil by itself (without lithium stearate soap) and two of the rust inhibitor/base grease combinations (**RI + Base A, RI + Base C**), although more corrosion was observed for the rust inhibitors with **Base B**. However, when the same rust inhibitor combination was added to the base oil (**RI + Oil**), it was very effective and prevented rust.

To investigate the effect of excess lithium hydroxide in the absence of the grease thickener system, 0.06 wt% LiOH was added to the distilled water used in the ASTM D1743 testing. This amount of LiOH is approximately the same amount present in the most alkaline of the base greases in this study. The addition of LiOH to the water used in testing the base oil (no thickener) significantly reduced the amount of rust.

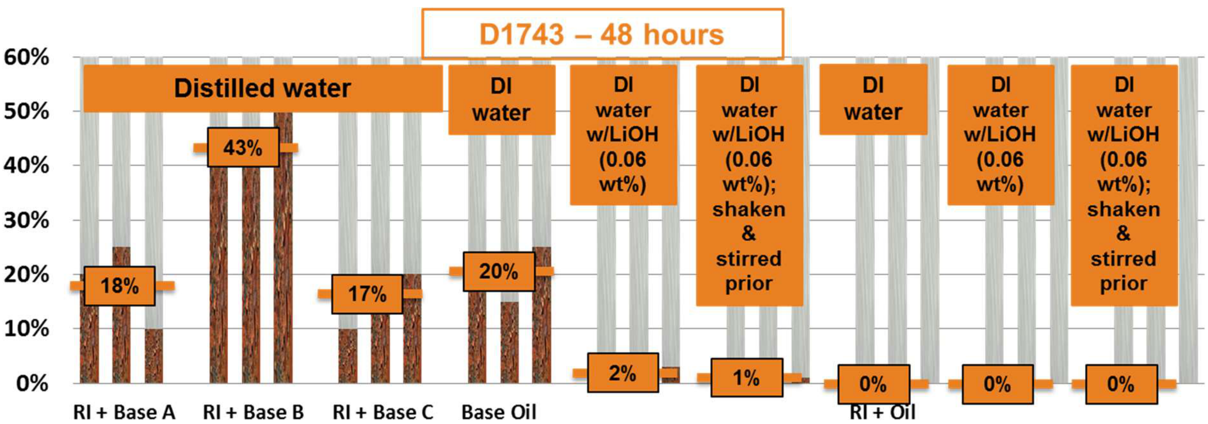


Figure 9 - Effect of excess LiOH on rust inhibitors

These results showed that some property of the grease thickener, and not excess LiOH present in the grease, reduced the effectiveness of the rust inhibitors. However, the distilled water containing LiOH did not come into contact with the metal surfaces until after the rust inhibitors were in contact with the bearing surfaces and may have formed a rust preventive film.

A follow up study was done in which the two solutions were first shaken and then stirred with DI water containing 0.06 wt% LiOH for ten minutes prior to testing to ensure that the rust inhibitors were given sufficient opportunity to interact with the LiOH. This additional mixing had no significanteffect on the results and confirmed that LiOH by itself did not have a negative impact on rust protection, either by reacting with the rust inhibitors or by causing corrosion on its own.

In fact, it appeared that LiOH helped prevent corrosion when present in base oil that contained no additional rust inhibitors. It is likely that the benefits of distilled water spiked with LiOH were due to raising the pH of the water.

The main conclusion of this part of the study is that LiOH did not directly reduce the effectiveness of rust inhibitors in lithium grease. This implies that negative effects seen in alkaline greases are likely due to interactions of corrosion inhibitors with the grease thickener system.

Borate Additives

The final step of this study was to observe the effect of borates on these thickener systems and their ability to prevent rust as well as to carry out other performance requirements of fully formulated lubricating greases. The fully formulated grease [**Formulation #1 (Grease A)**], using the more alkaline base grease (**Base A**) and the performance additive package from earlier work (**Fully Formulated #1**) was prepared and then top treated separately with two different borate amides and a boronated dispersant. Performance testing was carried out including standard rust, dropping point, fretting wear and four ball weld and wear testing as shown in Table 5.

Formulation #1 (Grease A)	100	99.33	98.5	98
Borate amide #1		0.67		
Borate amide #2			1.5	
Boronated dispersant				2
Testing:				
Rust (ASTM D1743)	F/F/F, F/F/F	P/P/P	P/P/P	P/P/P
Drop point, °C (ASTM D2265)	203	299	305	304
Fretting wear, mg (ASTM D4170)	13.1	7.1	6.3	8.4
4 ball weld, kg (ASTM D2596)	210	230	250	260
4 ball wear, mm (ASTM D2266)	0.45	0.40	0.49	0.44

Table 5- Impact of borates on fully formulated grease performance

While the effect of borates on raising the dropping point is well known in the grease industry, it is less expected to see such an improvement in rust performance, fretting wear and extreme pressure performance. Individually, each improvement could possibly be explained by changes in consistency or other phenomena. But it is harder to explain their collective improvement. Was this improvement a direct result of the borate chemistry? Or did these borates have an indirect effect, i.e., complex the grease thickener and thus allow other additives to interact with the metal surface instead of the thickener system?

An additional grease mixture (**Base Grease A + RIs + ZDDP**) was prepared using the same alkaline base grease (**Base A**) along with the same rust inhibitors and ZDDP used in the previous work. Samples of this grease were then top-treated individually with the three borates. The high dropping point results (Table 6) showed that these borates achieved a high degree of complexation of the base grease thickener (with likely synergistic interaction of ZDDP). The passing rust test results may have been due to the presence of ZDDP and borates, or to more effective complexation of the thickener system.

Base Grease A + RI's + ZDDP	99.33	98.5	98
Borate amide #1	0.67		
Borate amide #2		1.5	
Boronated dispersant			2
Testing:			
Drop point, °C (ASTM D2265)	302	290	271
Rust (ASTM D1743)	P/P/P	P/P/P	P/P/P

Table 6- Impact of borates on base grease with RI and ZDDP

Next, a grease mixture (**Base Grease A + RIs**) was made with just the rust inhibitors at the same level as the previous formulations. This mixture was then treated separately with the three borates and tested for dropping point and corrosion (Table 7). The degree of complexation, as indicated by the dropping points, was lower than that of the greases containing ZDDP. However, the greases still passed the rust test, although the previous tests showed a significant amount of rust (18% surface coverage) for the **RI + Base A** formulation without borates (Figure 8). It is possible that a sufficient amount of complexation allowed rust inhibitors to get to the metal surface. Sinceno rust was present in either case, it was impossible to know if the effect depended on complexation.

Base Grease A + RI's	99.33	98.5	98
Borate amide #1	0.67		
Borate amide #2		1.5	
Boronated dispersant			2
Testing:			
Drop point, °C (ASTM D2265)	253	249	256
Rust (ASTM D1743)	P/P/P	P/P/P	P/P/P

Table 7- Impact of borates on base grease with only RI

Finally, the experiment was repeated with only the individual borates added to the base grease (**Base Grease A**). Similar dropping points were obtained (Table 8) as the previous formulations with the rust inhibitors included. However, these greases also gave passing rust results without the addition of rust inhibitors. While borate amides are used as rust inhibitors, boronated dispersants are not used typically to provide protection against corrosion. These results may provide additional evidence that something in the base grease itself helps to cause corrosion.
As seen previously, there was 30% surface corrosion with **Base A** (Figure 7), and only 20% with the **Base Oil** (Figure 9). However, in this study, there were possible issues with repeatability of visual evaluations of surface corrosion and differentiating effectiveness of rust inhibition among greases that passed. However, these results certainly indicate that additional testing in this area could be worthwhile.

Base Grease A	99.33	98.5	98
Borate amide #1	0.67		
Borate amide #2		1.5	
Boronated dispersant			2
Testing:			
Drop point, °C (ASTM D2265)	256	257	255
Rust (ASTM D1743)	P/P/P	P/P/P	P/P/P

Table 8- Impact of borates on base grease alone

Summary and Conclusions

Baseline studies using base greases with different alkaline contents seemed to show that the alkalinity of the base grease by itself had little or no impact on the amount of corrosion seen in standard static corrosion testing (ASTM D1743). There seemed to be a plateau effect where the amount of rust was limited by immersion time and surface coverage. Increasing the immersion time or the concentration of the salt solution increased the amount of rust formed, but not in a linear manner (Figures 4 and 5).
In the presence of performance additives, the alkalinity of the base grease may have had an impact (Figure 7) on the ability of the grease to prevent rust. But the relatively narrow range of alkalinities and the limited repeatability of visual measurements in this study call for more work inthis area in order to confirm these findings.
The presence of lithium hydroxide was ruled out as a primary factor in reducing the corrosion protection ability of these greases (Figure 9). There was no evidence to suggest that LiOH reacted with the performance additives or caused corrosion by its presence. In fact, its presence seemedto have a mitigating effect on corrosion.

There was evidence to suggest that something in the thickener system contributed to corrosion or, at the very least, interfered with the corrosion inhibitors. The base oil by itself caused less corrosion than when the thickener system was present (Figures 7 and 9). The addition of rust inhibitors to the base oil eliminated corrosion, while those same rust inhibitors in grease provided only a minor reduction in corrosion (Figures 7 and 8).

Further work with borates in these base greases gave additional evidence that the lithium grease thickener system caused corrosion. One hypothesis is that the complexing effect of the borates in lithium greases improved the performance of the corrosion inhibitors in the base grease (Table7, Figure 8). But the fact that improvement was also seen without the corrosion inhibitors present (Table 8) may be further indication that something in the base grease itself contributed to corrosion. Another hypothesis is that there was less corrosion because there was less water present due to interactions with the borates or with the more complex grease thickener system. Further study is needed to clarify these points.

Additional study is needed to further explore interactions of base grease thickener systems and performance additives. New experimental techniques may be useful, especially with regard to more repeatable and precise measurements of corrosion. Average Gray Value and other optical measurement techniques to more precisely determine surface coverage by corrosion are becoming more mainstream. Contact angle evaluation and Quartz Crystal Microbalances (QCM) allow further exploration of rust inhibitor action and effectiveness. Further work with multivariateanalysis is needed to explore the interactions that appear to improve rust inhibition including borates, rust inhibitor combinations, ZDDP and other additives. These evaluations should includeadditional base greases as well as different types of corrosion testing such as the dynamic EMCOR bearing corrosion test (ASTM D6138).

References

1. Aguilar, G., Kaperick, J.P., Keisler, M., Lennon, M., "A Cursory Look at the Effects of Free Acid and Alkali on 12-Hydroxy Stearate Grease", Presented at NLGI India Chapter 18th Lubricating Grease Conference, Aamby Valley City, Lonavala, Maharashtra, India, February 5-7, 2016.
2. Koch, G.H., Brongers, M.P.H., Thompson, N.G., Virmani, Y.P., Payer, J.H. "Corrosion Costs and Preventive Strategies in the United States" Publication No. FHWA-RD-01-156.
3. Hunter, M. E. and Baker, R. F., "Corrosion-Rust and Beyond", NLGI Spokesman, Vol. 63, No.1, March 1999.
4. Hunter, M. E. and Baker, R. F., "The Effects of Rust Inhibitors on Grease Properties", NLGI Spokesman, Vol. 63, No. 12, 2000.
5. Hunter, M. E., Rizvi, S.Q.A., and Baker, R. F., "Ashless Rust Inhibitors for Greases", Presented at 67th NLGI Annual Meeting, Asheville, NC, October 2000.
6. Kaperick, J.P., Aguilar, G., Lennon, M., "Rust Never Sleeps – An Investigation of Corrosion inGrease Lubrication", NLGI Spokesman, Vol. 79, No. 6, Jan/Feb 2016.
7. ASTM D1743-13 "Standard Test Method for Determining Corrosion-Preventive Properties of Lubricating Greases", (2013) ASTM International, West Conshohocken, PA.
8. ASTM D5969-11(2016) "Standard Test Method for Corrosion-Preventive Properties of Lubricating Greases in Presence of Dilute Synthetic Sea Water Environments", (2016) ASTM International, West Conshohocken, PA.
9. ASTM D128-98(2014)e1 "Standard Test Methods for Analysis of Lubricating Grease", (2014)ASTM International, West Conshohocken, PA.

The Impact of the Raw materials on the Characteristics of Lithium Complex Greases

Mehdi Fathi-Najafi (Nynas)* and Yanqing Gao & Lanying Zhang (Sinopec)

Abstract

It is well known that higher solvency of the base oil has a significant impact on the several properties of the lubricating greases. It is also known that Lithium complex greases can usually be prepared by using different complexing agents with low molecular weight dicarboxylic acid.

The purpose of this study was to focus on the impact of the degree of solvency power of three naphthenic oils on two different Lithium complex greases. To reduce the source of errors, parameters such as crude type, viscosity of the selected oils and the manufacturing process of the greases have been kept constant.

Within this framework, it has been found that naphthenic oil with medium viscosity performs very well in different types of lithium complex grease formulations and the solubility of base oil affects the structure of grease in a positive direction. Furthermore, the formulation of a lithium complex grease can be easily optimized by using suitable base oil and complexing agent.

Key words: Naphthenic oil, Solvency, Lithium Complex Grease, Flow Pressure, Elastomer Computability, Tribology, Rheology*) corresponding Author

Introduction

A typical complex grease consists of 12-hydroxide Stearic Acid or/and Castor Oil, Lithium Hydroxide, a complexing agent and at least one mineral oil. It is well known, however, that the quality of the fatty acid, purity and particle size of the lithium hydroxide have some impacts on both characteristics and manufacturing time of the greases. In this paper, the focus will be on the mineral oil and the complexing agent.

Mineral oil should fulfill numerous requirements in many different applications. The characteristics of a mineral oil are determined by number of parameters such as type of crude and refining process. The degree of refining is equivalent to the degree of solvency of the oil which, to some extent, can be quantified by Aniline point (AP) and Viscosity Gravity Constant (VGC).

A mineral base oil consists mainly of carbon and hydrogen bound in molecules with different structures that are either paraffinic, naphthenic or aromatic. The type of hydrocarbons varies between different type of mineral oils depending e.g. on crude type and degree of purification (refining). Base oils and crudes are classified in Paraffinic or Naphthenic, however, there is no sharp distinction between the two type of Base oils. Hence, the characterization is based on the paraffinic content, measured by e.g. IR

1. Paraffinic content (CP) = 42-50% then it's considered as naphthenic oil
2. Paraffinic content (CP) = 50-56% then it's considered as intermediate oil
3. Paraffinic content (CP) = 56-67% then it's considered as paraffinic oil

AP is defined as the lowest temperature at which a mineral oil is completely miscible with an equal volume of aniline (ASTM D 611). The lower the AP, the better the solubility. While VGC is a calculated dimensionless constant that is processed from viscosity and the density values of the oil (ASTM D 2501). This constant varies from 0.760 to 1.000, the higher VGC indicates higher degree of solvency.

The aim of this study however was to investigate how some naphthenic oils with different degree of solvency (corresponding to degree of refining and polarity) and different type dicarboxylic acids affect the characteristic of lithium complex greases with a low thickener content (9.5 wt%).

The Base Oils

Three naphthenic base oils with different degree of refining have been chosen for this study. The base oils (BO1, BO2 and BO3) have been manufactured by using the same crude and process (hydrotreatment). Table 1 highlights some of the typical characteristics of these base oils.

Characteristics	Unit	Method / ASTM	BO1	BO2	BO3
Density @ 15°C	kg/dm^3	D 4052	0.915	0.905	0.895
Viscosity @ 40°C	mm^2/s	D 445	109	98	100
Viscosity @ 100°C	mm^2/s	D 445	8.8	8.6	8.9
Viscosity Index (VI)		D 2270	19.11	33.53	41.01
Flash Point, PM	°C	D 93	212	216	214
Flash Point, COC	°C	D 92	228	228	220
Pour Point	°C	D 97	-30	-33	-30
Aniline Point (AP)	°C	D 611	87	96	104
Viscosity Gravity Constant		D 2501	0.855	0.843	0.830
Reflective Index @ 20°C		D 1747	1.501	1.495	1.487
Copper Corrosion		D 130	1	1	1
Sulphur content	wt.%	D 2622	0.11	0.03	0.01
Color		D 1500	<1.0	<1.0	<0.5
Total Acid Number	mgKOH/g	D 974	<0.01	<0.01	<0.01
Carbon Type Composition		D 2140			
CA (Aromatic content)	%		11	7	<1
CN (Naphthenic content)	%		39	40	44
CP (Paraffinic content)	%		50	53	55

Table 1. Typical characteristics of the three naphthenic oils.

Table 1 reveals some interesting information such as:

a) Higher value on density, VGC, Sulphur content and C_A as well as a lower AP and VI suggest that BO1 has the highest solvency power and lowest degree of refining followed by BO 2 and BO3. With other words, BO 3 is the most refined oil among these three.
b) The pour point for all three oils is regarded to be the same and very low due to the use of an almost wax free crude.

Tribological measurements of the six lithium complex greases were conducted by using two different tribological systems; a tribo-cell and film thickness of the greases at EHL by using a ….

Tribo-cell is a device attached to a rheometer. The theory behind the tribometer has been described previously [Ref]. In principle, the "tribo-cell", which is an accessory to the rheometer, consists of a steel ball on three plates (substrates). When a rotational speed is applied to the shaft, the ball slides with respect to the contact points on the three plates, see Figure 1. This instrument allows measurements of friction curves during controlled sliding speeds from to 10^{-6} to 1.4 m/s.

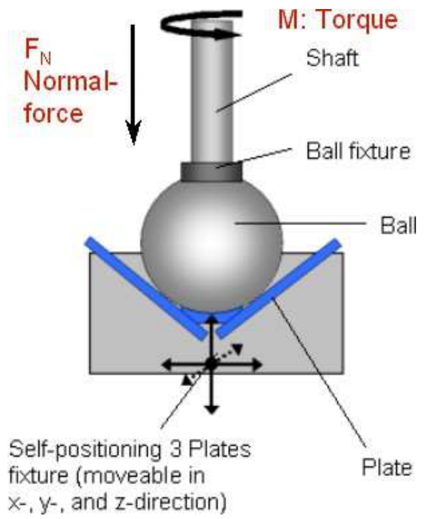


Figure 1. Tribo-cell

Valid Parameters for all measurements	
Sliding speed range	$1x10^{-6}$ to 1.4 m/s
Radius of steel ball	6.35 mm (1/2 inch)
Normal force	10 N

Table 2. Test conditions in the tribo-cell.

The tribological measurements performed by the tribo-cell consist of the measurements of the friction coefficient (μ) for the three base oils at ambient temperature.

The measured friction coefficient as a function of sliding speed for the three base oils at ambient temperature can be seen in Figure 2 where BO1 shows lowest friction coefficient (most probably due to the higher viscosity at 25 °C) at low speed followed by BO2.

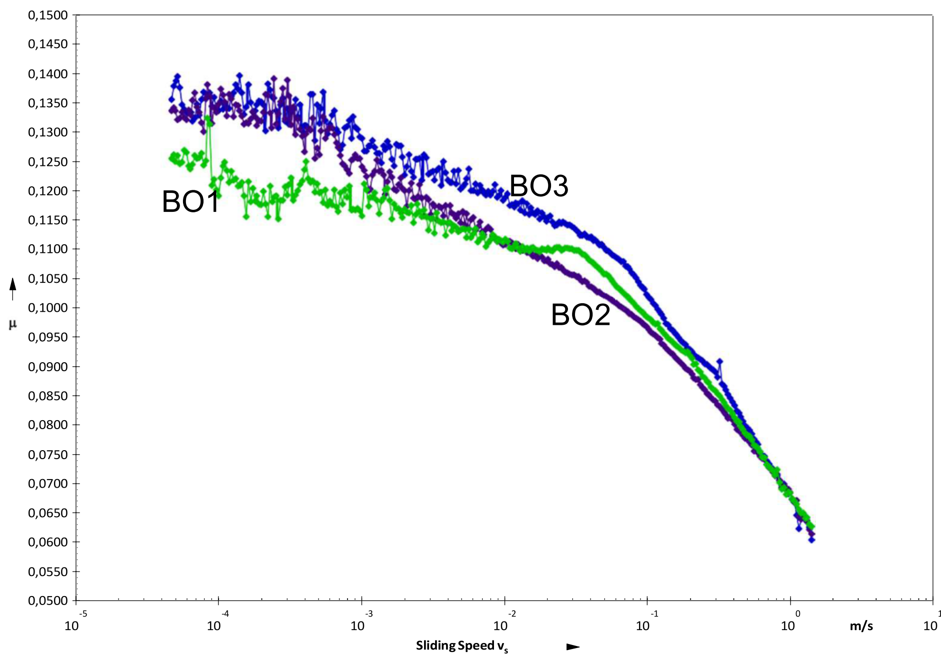


Figure 2. the measured friction coefficient for the base oils.

The Greases

In total, six lithium complex greases (A, B, C, D, E and F) have been prepared in a pilot plant by using the three naphthenic oils (BO 1, 2 and 3) and two dicarboxylic acids (Acid R and Acid G). Acid R has a shorter carbon chain than Acid G and the chosen fatty acid was 12-Hydroxy Stearic Acid (12HSA).

To reduce the source of errors, the soap content, the ratio between the 12HSA and the Dicarboxylic acids, and production parameters have been kept constant. Table 3 demonstrates some of the properties of the greases. Notable that the greases contain an antioxidant package composed of amine and phenolic derivate.

Properties	Grease A	Grease B	Grease C	Grease D	Grease E	Grease F
Base Oil (Naphthenic)	BO1	BO2	BO3	BO1	BO2	BO3
Dicarboxylic Acid	Acid R	Acid R	Acid R	Acid G	Acid G	Acid G
Soap Content; wt.%	9.5	9.5	9.5	9.5	9.5	9.5
Dropping Pont; °C	305	297	304	307	270	303
Pen (unworked); mm^{-1}	275	280	282	253	258	258
Pen (after 60 str.); mm^{-1}	276	280	285	253	266	261
Pen (after 10^5 str.); mm^{-1}	326	326	332	312	307	328
Diff after 10^5 str.; mm^{-1}	+50	+46	+47	+59	+41	+57
Oil Separation; wt.%	5.24	6.06	6.10	2.57	3.47	2.59
Oxidation stability, PDSC OIT@180°C/3.5MPa; min	11	9	20	6	5	13
Flow Pressure@-20°C; mbar	341	328	300	498	373	400
Flow Pressure@-30°C; mbar	546	519	512	759	569	604
Flow Pressure@-40°C; mbar	1170	963	914	1414	1017	1124

Table 3. The measured characteristics of the lithium complex greases A review of Table 3 suggests that

a) All the Dropping points are above 260 °C which is regarded to be the minimum value for a complex grease. However, Grease E shows the lowest dropping point and the reason behind this is not clear to the Authors.

b) The greases based on dicarboxylic acid G show higher consistency, about 0.5 NLGI grade higher than dicarboxylic acid R based greases.

c) The worked penetration number of all the greases after 100,000 strokes indicates good and similar shear stability.

d) The bleeding tendency measured according to IP 121 (40°C/168hours) for the greases based on dicarboxylic acid R show almost twice as high as for the dicarboxylic acid G based greases which can be attributed to the higher consistency which in turn may be depended on the structure of the greases that is going to be discussed later in this paper.

e) Oxidation stability of the greases were measured as oxidative induction time (OIT) using a pressurized differential scanning calorimeter (PDSC). The results emphasize that BO3 based greases are significantly better than BO1 and BO2 based greases. Furthermore, lithium complex greases based on dicarboxylic acid R show twice as high OIT than dicarboxylic acid G based greases. The common denominator is a factor two in oxidation stability if a neat lithium complex grease based on BO3 and dicarboxylic acid R the selected formulation.

f) Pumpability of the lubricating greases can be simulated by different methods e.g. measurement of the flow pressure according to DIN 51805. Parameters such as soap content, consistency of the grease, polymer content, kinematic viscosity of the oil, pour point as well as the degree of the wax content in the base oil are the main parameters that can affect the mobility of the greases. Although, the flow pressure measured for all lithium complex greases down to – 40 °C show excellent results, it is inevitable not to note that BO1 based greases show slightly higher flow pressure within the temperature interval (-20 °C to -40 °C).

What is reported above is often enough for the formulators to move on with one or another base oil and/or thickener system, however, the authors would like to expand the scope of this work by investigating further a second layer of this investigation by considering the following areas:

- softening degree of the greases as function of temperature.
- the rate of the oil bleed over a longer period.
- the structure of the greases.
- elastomer compatibility on Chloroprene rubber
- the rheological properties

Softening degree of the greases as function of temperature can be measured e.g. the modified ISO 2137-1985 test method which simply means that the unworked penetration number is measured at various temperatures. The idea here is to simulate the condition of the greases in a static condition at evaluated temperatures. A high degree of softening a grease may indicate higher tendency to leakage of the grease e.g. out of the bearing which may in turn reduce the performance and/or the re- lubrication time of the grease.

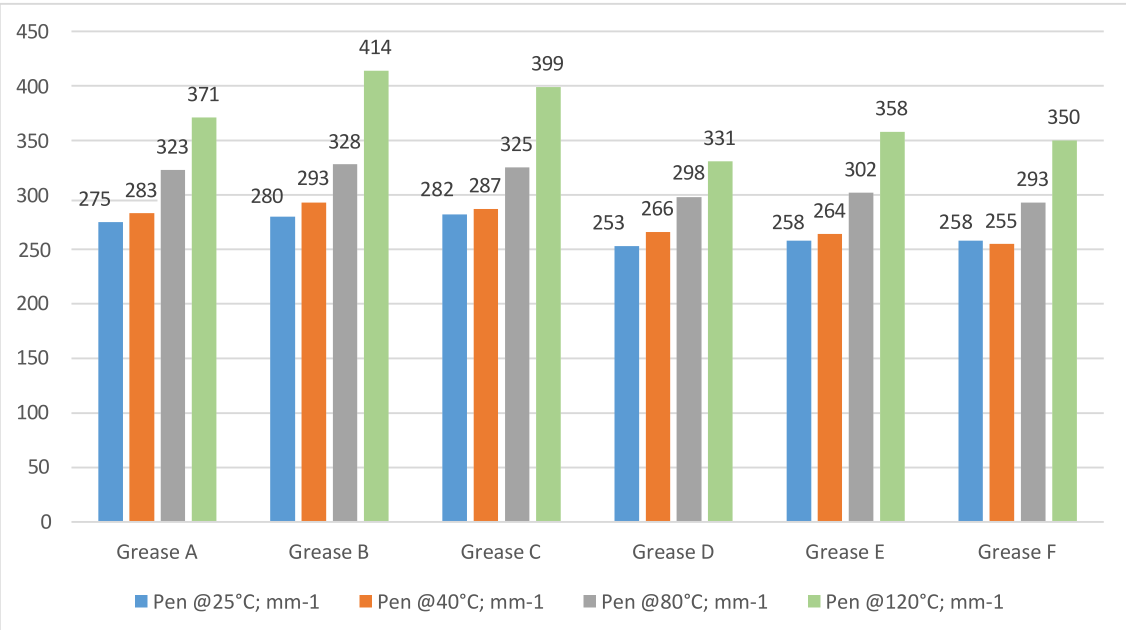


Figure 3. Unworked penetration of the six lithium complex greases at different temperatures.

Results summarized in Figure 3, indicates that dicarboxylic acid G based greases (Grease D, E and F) have less tendency to soften than the dicarboxylic acid R based. On the base oil side, there is also a tendency that BO1 based greases resist the thinning effect better than other base oils (BO2 and BO3). It seems that the higher polarity of BO1 interacts better with the thickener system regardless of the type of the complexing agent and it overcomes the lower viscosity index.

The rate of the oil bleed of a lubricating grease is an important parameter which can have both a positive and negative impact on the storage and performance of a grease.

The bleed oil can appear both in the static condition and the dynamic condition. Heat, shear, gravity, centrifugal forces and vibration are among the parameters that can release the oil from the thickener system matrix.

In principal, the oil separation during the storage time should be as low as possible, however in the application, a controlled amount of bleed oil is desirable, in order to maintain smooth lubrication. This is one of the typical challenges when formulating lubricating greases that supposed to operate optimally both at high and at low temperature.

Complex greases contain often high percentage of thickener which will for example ensure better resistance to the heat and the collapse of the film in the contact area, but then as a spin off effect it may also prevent the grease from bleeding too much at the operating condition which in turn may contribute to a longer relubrication time. However, at the ambient and low temperature, too high a soap content may block the oil from bleeding out into the contact surfaces e.g. of rolling elements and raceways which may lead to poor lubrication or so-called starvation and consequently to wear.

For instance, Calcium sulfonate complex with 25 up to 40 percent of thickener can fall into this category of low bleed products. After all it is well known that a high thickener content does not guarantee good lubricity since the thickener system has very limited ability to lubricate but excellent capability to separate two surfaces from each other. Therefore, a "controlled" bleed oil rate, may be regarded as one of the important parameters, that should be considered when longer service life is targeted.

Thus, it's a challenge for the formulators to optimize their formulation in a way that it can work well over a wide range of temperature e.g. -40 °C to about + 170 °C for the mineral oil based greases.

IP121 is a static oil separation test method that results to a certain weight percent of the bleed oil after 168 hours at 40 °C. The result shows the volume not the rate of the separated oil. It has been stated that when about 50 percent of the oil is leaked out from a grease then the grease should be replaced with a fresh one. Therefore "controlling" the rate may extend the life of the grease.

The degree of the bleed oil in a grease can be optimized by using a modified version of IP121. For instance, conducting the test at lower and higher temperatures than 40 °C and logging the rate of the separated oil frequently. In this study, however only the rate of the bleed oil at 40 °C has been measured, Figure 4.

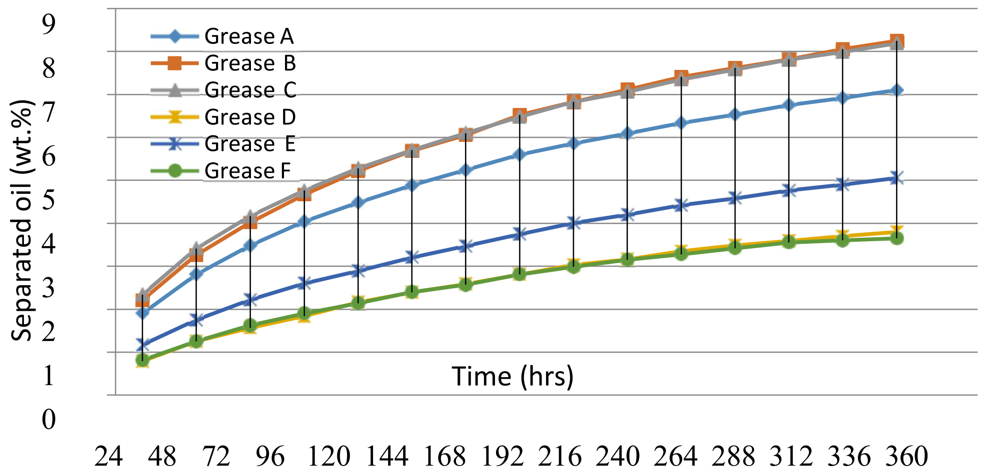


Figure 4. Bleed oil as a function of time (modified IP121)

As it can be seen in Figure 4, there are four different profiles for the six greases with different bleeding rates over a longer period of time. Greases A and D contain BO which has lowest degree of refining, exhibit lowest degree of oil separation. This is in-line with the expectation that BO1 has highest polarity compared with other two oils and subsequently enjoying stronger interaction with the fibrous matrix.

T**he structure of the lithium complex greases** has been captured by using a Scanning Electron Microscope (SEM) which is an impressively and powerful equipment.

Figure 5a. Grease A (BO1/ Acid R)

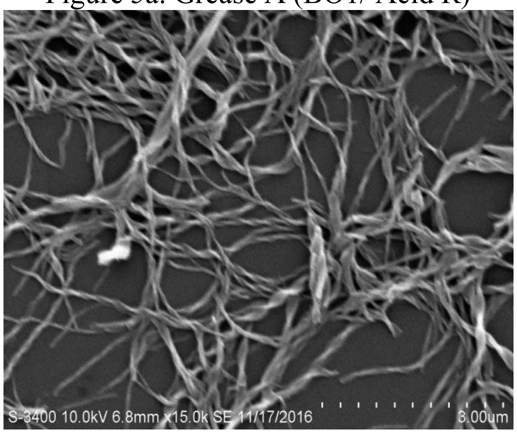


Figure 5b. Grease B (BO2/ Acid R)

Figure 5d. Grease D (BO1/ Acid G)

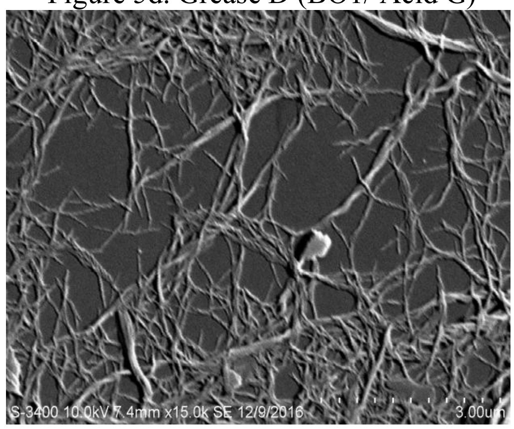
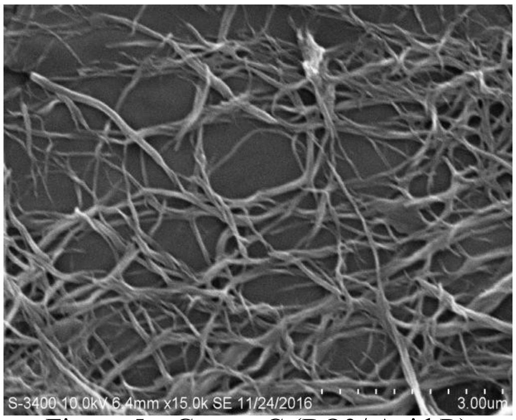


Figure 5e. Grease E (BO2/ Acid G)



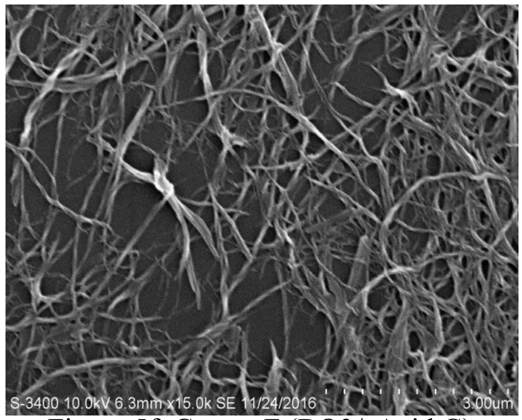
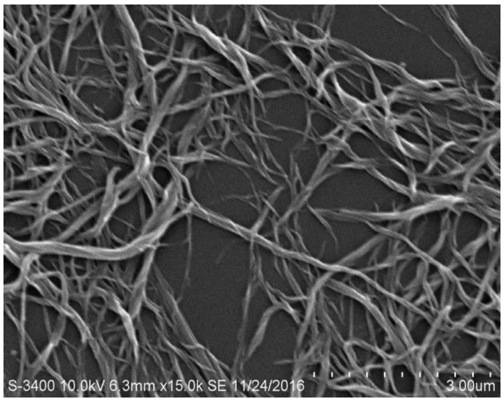


Figure 5c. Grease C (BO3/ Acid R) Figure 5f. Grease F (BO3/ Acid G)



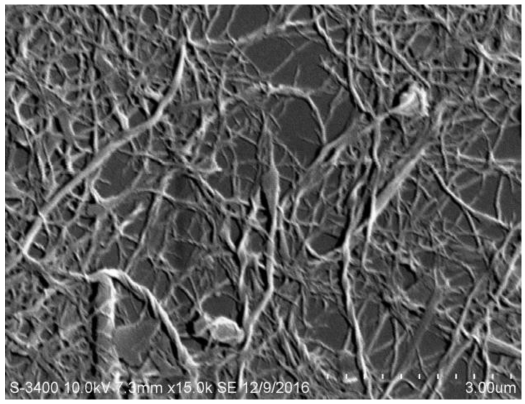


Figure 5. SEM picture of the structure of the six lithium complex greases

Using electron microscopy technique for studying the thickener system is relevant and accurate if we take also the source of errors into the consideration. In other words, it's essential that to take the SEM pictures as a piece of evidence for completion of a multi-disciplinary study but not relying only on that. All the pictures in Figure 5 has same magnitude (300 nm) which means that the thickness of the fibers can be compared with each other.

Figure 5 suggests some possible explanations concerning the consistency of the greases that was shown in Table 1. There are two different variables involved in this work; complexing agent and degree of solvency, hence, the interpretation of the SEM pictures may be divided into two different parts:

Firstly, the impact of dicarboxylic acids on the formation of the thickener; to analyzing the impact of using different complexing agents on the formation of the fibers (thickeners), it requires that primarily to study the impact of the solvency of the oils is excluded, hence, these pictures should be compared one to one; 5a vs. 5d, 5b vs. 5e and 5c vs. 5f. These comparisons suggest that the lithium complex greases based on dicarboxylic acid G shows a denser and smaller fibrous structure than the dicarboxylic acid R based grease.

Secondly, the impact of degree of solvency of the base oils on the formation of the thickeners; it implies a comparison of dicarboxylic acid R lithium complex greases (Figure 5a, 35b and 5c) with dicarboxylic acid G based lithium complex greases (Figure 5d, 5e and 5f). Here, it seems to be difficult to distinguish a clear trend within these two blocks of Figures

Elastomer Compatibility of the Greases has been conducted on chloroprene rubber (CR), according to GB/T1690-2010 (ISO 1817-2005, modified method).

The weight and hardness are measured with Chloroprene rubber type I (25×50mm), the tensile properties were also measured by type II dumbbell shaped specimen (specimen length was 20mm and the width of narrow part was 4mm).
The tests were conducted as follow: the rubber samples were immersed totally in a container filled with the grease which then was placed in an oven at 100 °C. After 70 hours, the samples were cooled down to ambient temperature, then the greases were removed and the changes were measured and demonstrated in Figure 6.
It is relevant here to pay attention to the relationship between the solvency of the base oil and its compatibility with different rubber types. It has been shown in the literature that base oils with a higher solvency, corresponding to a lower aniline point, have a higher impact on more polar rubbers such as CR. The higher impact is commonly shown as a higher oil absorption rate in the rubber material. This in turn leads often to a higher softening effect on the rubber, e. g. a decrease in hardness and tensile properties.
This behavior also explains the results seen in Figure. 4: The solvency of the oils in this study increases as BO1> BO2 > BO3. Hence, the impact on the CR is higher for the greases made with BO1 followed by those made with BO2 and BO3 respectively.

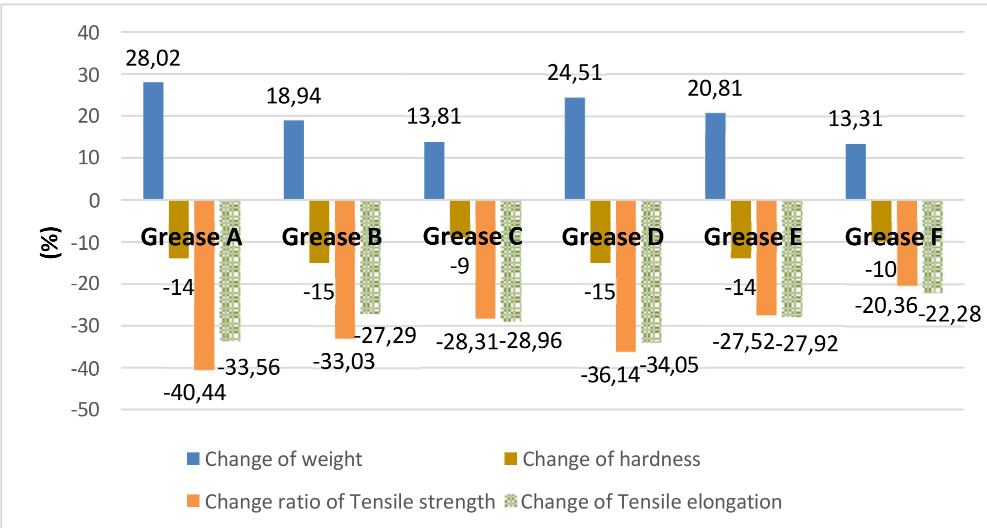


Figure 6. the impact of the lithium complex greases on the chloroprene

Rheological behavior of the greases has been studied by using a rotational rheometer in oscillating condition. The flow point of a lubricating grease is determined in a strain sweep at the cross over point G'=G''. The test is carried out at an angular frequency of w=10 rad/s and at the 6 different temperatures, from -30 °C to +120 °C, Figure 7.
The measured shear stress at the flow point decreases with the increased temperature for all greases which is in-line with the expectation since lubricating grease is a viscoelastic material. However, Figure 7 emphasis that the shear stress has low degree of fluctuation between 0 and 40 °C for all greases, however, it becomes to be more pronounced when approaching -30 °C. This is apparently a consequence of an increased result of the change in elasticity (storage modulus) of the greases.
Figure 7 implies further that grease A, B and C are less sensitive to the applied temperature while grease D, E and F (dicarboxylic acid G based), which may be related to the structure stability and denser fibrous matrix. It is obvious that all six greases behave similar to each other from 120 °C to 80 °C, but as soon as the temperature drops, the degree of the elasticity of the greases based on Dicarboxylic acid G) is increasing rapidly.

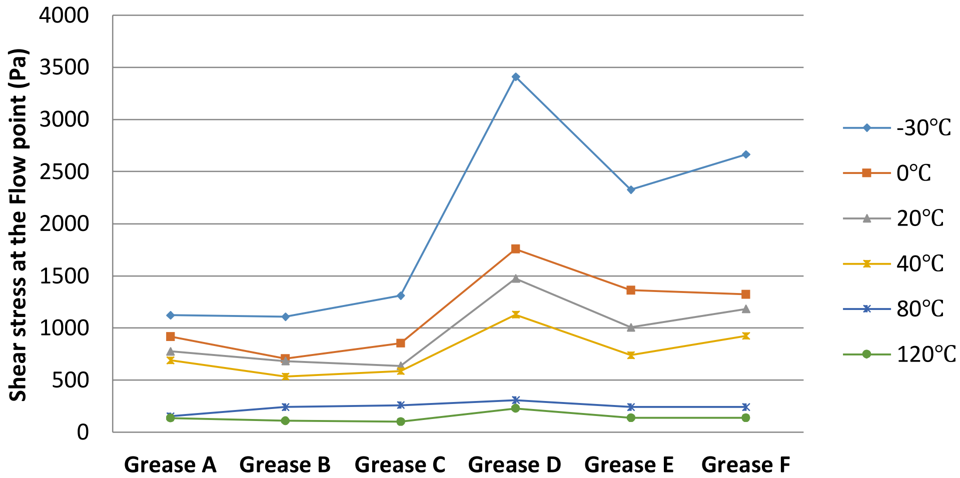


Figure 7. Shear stress at the flow point of the lithium complex greases.

The damping factor (or loss factor) is expressed by tanδ (=G"/G'; Viscous modulus/Elastic modulus). This factor varies from 0 to infinite, if "zero" then it means that grease behaves like an ideal solid and if " infinite", it means that it behaves like an ideal liquid. Figure 6. shows that all six greases behaved as viscoelastic material where the elastic behavior became to be more pronounced as the applied temperature drops and reaches a maximum at -30 °C which is equal to the pour point of the base oils.

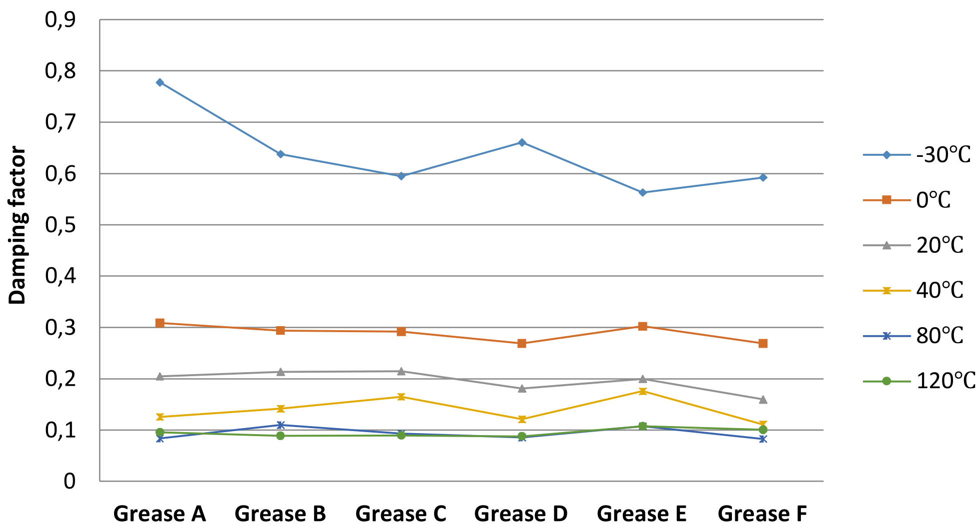


Figure 8. Loss factor for the greases at various temperatures.

Summary

This multi-disciplinary study has resulting to the following conclusions:

I. Three wax free naphthenic base oils with different degrees of refining have been selected for the preparation of lithium complex greases. The characterization of the base oils showed that BO1 has the highest solvency power followed by BO2 when compared with BO3.

II. Tribological properties measured by using a the tribo-cell, indicate that BO1 shows lowest friction coefficient followed by BO2.

III. Six lithium complex greases have been produced, based on the three base oils and two different complexing agents (dicarboxylic acid G and dicarboxylic acid R), showing good characteristics such as shear stability and high dropping point. Notable that although dicarboxylic acid G has longer carbon chain than dicarboxylic acid R.

IV. A review of the main characteristics of the lithium complex greases show:

a) higher consistency, about 0.5 NLGI grade higher for greases based on acid G than acid R based.

b) the oil bleed for the greases based on dicarboxylic acid R show almost twice as high as for the dicarboxylic acid G based greases most probably due to the lower consistency (softer grease).

c) BO3 based greases are significantly better than BO1 and BO2 based greases with respect to the oxidation stability, most probably due to the higher degree of refining and subsequently better response to the antioxidant.

d) Lithium complex greases based on dicarboxylic acid R show almost twice as high OIT (oxidative induction time) than dicarboxylic acid G based greases.

e) The mobility of the lubricating greases, based on the measure flow pressure test, show that all lithium complex greases down to an extreme low temperature (– 40 °C) offer excellent results.

V. The SEM pictures indicate that the lithium complex greases based on acid G contributes to a denser and smaller fibrous thickener matrix than acid R based greases.

VI. The comparative study of the elastomer compatibility of chloroprene rubber (CR), GB/T1690-2010 (ISO 1817-2005, modified method) confirms the previous finding that the degree of solvency of the oils has a significant impact on the obtained results. Hence, the impact on the CR is higher for the greases made with BO1 followed by those made with BO2 and BO3 respectively.

VII. Rheological behavior of the greases shows that loss factor for all the greases displays viscoelastic, which rapidly transformed to a more elastic at an applied temperature of -30 °C which is equal to the pour point of the base oils. However, it seems that grease A, B and C (complex greases based on acid R) are less sensitive to the applied temperature compared with grease D, E and F (based on acid G) which may be related to the differences in the fibrous networks.

Reference

1) M. Fathi-Najafi et.al; Low temperature tribology; Eurogrease 201204
2) www.nynas.com; Base oil handbook
3) P.M. Lugt; Grease Lubrication in Rolling Bearings; January 2013

Acknowledgement

The authors would like to thank

1) Mrs. Zhiying Liu and Mr. Baojie Wu from Sinopec for their contribution during this study.
2) Nynas Petroleum Co., Ltd. (Shanghai) for suppling the naphthenic base oils.

Fuel Efficient and High Performance Multigrade Gear Oil, its Feature, Advantage and Benefit in automotive applications- a case study

S.K.Haldar, IndianOil, Bhopal, India

Historically, monograde Gear Oils have been used in cars and commercial vehicles. Recently, demands for improvements in the low temperature properties of gear oils and the realization that gear oil performance can make a contribution to fuel economy, less vehicle downtime and lower maintenance have led to the development of multigrade gear oils.
Multigrade Automotive Gear Oils in SAE viscosity grades 75W80, 75W90, 80W90, 80W140 85W140 have more attracted interest in the context of high efficiency and fuel economy. Targets of gear transmission design today are higher efficiency, higher torque capacity and reduced size. Increasingly smaller transmissions with higher torque lead to increasing operating temperatures. This trend is further intensified by the use of noise abatement devices and improved aerodynamic body styling that reduces the airflow around transmissions. The friction in the transmission is responsible for temperature increase and efficiency losses, and thus the reduction of friction is the main measure in order to improve the efficiency and to keep the operating temperature low. The lubricant influences and is subjected to all changes of operating conditions. Higher operating temperatures result in a higher consumption of friction modifiers; extreme pressure and anti wear additives, higher corrosion and oxidation rates, and a thinner oil film separating the various components. On the other hand, the torque transmission losses and, as a consequence, the operating temperature can be reduced by the right choice of base oil and additives.
Most lubricants for manual gearboxes and differentials contain extreme pressure (EP) additives and antiwear additives to cope with the sliding action of hypoid bevel gears. The essential functions of an automotive gear lubricant viscosity modifier (VM) are to maintain fluid film protection of gears and bearings as the lubricant warms to operating temperature, to improve cold temperature flow for efficient lubrication in winter and to minimise viscosity loss in a high shear, high load environment. Although a number of different VM technologies can be considered appropriately resistant to permanent shear for automotive gear oils use, their effect on fluid efficiency can vary widely. Some modern automatic transaxles (integrated transmission and differential) do not use heavy oil at all but lubricate with the lower viscosity hydraulic fluid, which is available at pressure within the automatic transmission.
This paper outlines the study of Multigrade Gear Oil for assessing relationship of operating temperature, operating viscosity and axle efficiency under different load and speed regimes and setup of a light duty axle efficiency test in evaluating gear lubricants for their fuel economy performance. The fluids presented were formulated to equal kinematic viscosity at 100 °C but vary widely in viscosity index (VI), elastohydrodynamic (EHD) traction and EHD film thickness. The differences observed during efficiency testing were qualitatively related to the rheological properties of the technology present. A light-duty axle efficiency test for evaluating gear lubricants for their fuel economy performance is described. Multigrade gear lubricants (mainly 75 W 90, 80W90 LL, 80W 140 LL & 85 W 140) were

made under a variety of pinion torques and speeds to simulate highway and city driving conditions. Lubricant rheology and its importance in maintaining film strength for adequate bearing and gear lubrication for optimum torque efficiency and axle temperature with major OEM field trial data are depicted in this study.

The major targets of transmission design today are higher efficiency, higher torque capacity and reduced size. Increasingly smaller transmissions with higher torque lead to increasing operating temperatures. This trend is further intensified by the use of noise abatement devices and improved aerodynamic body styling that reduces the airflow around transmissions. The friction in the transmission is responsible for temperature increase and efficiency losses, and thus the reduction of friction is the main measure in order to improve the efficiency and to keep the operating temperature low. The lubricant influences and is subjected to all changes of operating conditions. Higher operating temperatures result in a higher consumption of friction modifiers; extreme pressure and anti wear additives, higher corrosion and oxidation rates, and a thinner oil film separating the various components. If the lubricant is not formulated to withstand these more severe conditions the transmission may be damaged. On the other hand, the torque transmission losses and, as a consequence, the operating temperature can be reduced by the right choice of base oil and additives. In vehicle application today, most OEMs start the lubricant development in an early phase of the development of a new transmission in order to get the optimum contribution of the lubricant. These OEMs see the lubricant as a construction element needed for the development of performance optimized transmissions. In order to select the most effective combination of additives and base stocks offered by the lubricant industry, the OEMs have developed screening tests with good correlation to field performance. Such tests range from a simple bearing test to OEM-specific transmission or axle tests. We have used two tests developed by VW, that correlate well with a car installed on a chassis-dynamometer and a transmission rig test, in order to identify and quantify the influences of fluid viscosity, base stock types, and VI Improver chemistry on efficiency and operating temperature. Finally, examples of improved semi- and fully synthetic formulations with significantly reduced operating temperatures and optimum efficiencyare presented.

A light-duty axle efficiency test for evaluating gear lubricants for their fuel economy performance is described. Data collected for internal reference oil highlight the repeatability of the test with different axles. Comparisons between single-grade SAE 90 and multigrade gear lubricants were made under a variety of pinion torques and speeds to simulate highway and city driving conditions. Lubricant rheology and its importance in maintaining film strength for adequate bearing and gear lubrication for optimum torque efficiency and axle temperature are discussed.

The last twenty-five years have seen an increasing importance placed on fuel economy by automobile manufacturers. Today’s light- and heavy-duty trucks, passenger cars and buses have better fuel economy than their recent predecessors. In certain regions of the world, government legislation and increased competition between automobile manufacturers have given the end user fuel- efficient automobiles suited to many needs and applications.

The drive for improved fuel economy is being pursued with increasing vigor and has become more challenging in the light of perceived consequences for the environment. Examples of such global effects include warming from the greenhouse effect, increased acid rain, and ozone depletion. Automobile manufacturers are incorporating improved aerodynamics, lower rpm and higher- torque engines, and drivelines with increased power densities in their current Gear Oil 80W-140 is a fuel economy, API GL-5 and MT-1, multi- grade gear oil for a wide temperature operating range. Gear Oil 80W-140 reduces friction thereby increasing the miles per gallon of the vehicle plus providing excellent gear train protection against wear, corrosion and costly breakdowns.

Gear Oil 80W-140 as wide range multi-grade gear oil, offers good economic improvement in fuel economy. Independent commercial tests conducted over extended periods of time in various fleets under completely different operating conditions showed up to 2.65% improvement in fuel economy over similar units using various straight grade gear oils or 80W-90 gear oils. Depending upon the vehicle, miles of operation per year and cost of fuel, savings can range up to $250.00 per year per vehicle. Multiply this by a number of vehicles and a considerable savings can be achieved.

Gear Oil 80W-140 provides the low initial starting torque when the equipment is cold saving on fuel economy and the extra hard ware strain on the engine and drive chain. At the high running temperatures, Gear Oil 80W-140 provides the needed film cushion on the moving parts in the gear unit. Gear Oil 80W-140 is one gear oil for year-round service; no need to change the gear oil when the weather or seasons change. Gear Oil 80W-140 saves on inventory - one lubricant instead of three; less inventory movement - eliminates possible mix-up in gear oil selections.

First of all, it should be considered that the fuel consumption of a car depends on a set of parameters only partly related to tribology. Their influence is much more pronounced than that of the lubricant.

Driveline Lubricant Types & End Uses:

- AGO –Automotive Gear Oil
  –AGO w/LS –Automotive Gear Oil with Limited Slip
- ATF –Automatic Transmission Fluid
- MTF –Manual Transmission Fluid
- DCTF –Dual Clutch Transmission Fluid
- CVTF –Continuously Variable Transmission Fluid
- UTTO –Universal Tractor Transmission Oil
- ERTTO –Environmentally Responsive Tractor Transmission Oil
  –Like UTTO, but biodegradable
- STOU –Super Tractor Oil Universal –Like UTTO, but can be used as Tractor Engine Oil also
- TDTO –Total Driveline Transmission Oil (CAT)
- FDAO –Final Drive Axle Oil (CAT)

Gear Lubricant Performance Needs

	MTF	Axle Oil
Extreme Pressure	++	+++ Cope with sliding of hypoid gears
Anti-wear	++	++
Dispersancy	+	+
Detergency	++	+
Oxidation Inhibition	++	++
Corrosion Inhibition	+++	++Protect parts of the transmission
		Smooth shift feel
Friction	+++	+

Typical Gear Oil Lubricant Formulation

Performance Package (5-10% weight) Pour

Point Depressant (0.3% weight) Viscosity

Modifier (0-30% weight)

Base Oil (60-90% weight)

Role of Additives

Friction Modifiers –provides smooth shifting and reduces wear

Anti-foam –ensures proper lubrication by reducing foam

Corrosion Inhibitor -Prevents corrosion of critical parts and ensures synchronizercompatibility

EP –prevents metal to metal contact and welding (sulfur)

Anti-wear –prevents metal surface wear (phosphorus)

Dispersant-Keeps parts clean at lower temperatures and protects against oil thickening due to contamination

Automotive Gear Oil Industry Specifications

Specification	Description
SAE J306	Gear lubricant viscosity grades, example SAE 80W-90

API Specifications	ASTM D7405-13 (API categories GL-5 and MT-1)
SAE J2360 MIL-PRF-2105E	Multipurpose gear lubricating military oils, equivalent to

Other considerations are:

1. Only the mechanical losses can be decreased by lubricant-related measures. Therefore, the fuel economy improvement that possibly might be realized is rather limited, especially when taking into account the rather high efficiency of gears.
2. When evaluating the influence of viscosity on fuel consumption, the so-called effective viscosity must be taken into account. This is most important for non-Newtonian oils.
4. Reducing the gear oil viscosity by one SAE viscosity grade will result in fuel consumption reductions of 0.2-1.5 per cent at high temperatures and 0.4-2.5 per cent at lowtemperatures.
5. Using friction modifiers in gear oils, fuel consumption reductions of between 1.0 and 6.0 per cent are realistic.
6. On the basis of a 50 per cent friction reduction maximum fuel consumption reductions between 1.0 and 5.1 per cent by other gear oils are possible, considering different driving programmes.
7. Tests with a real automobile gear resulted in fuel economy improvements of the order of magnitudes of 1 per cent by other gear oils.

Friction Modifiers – provides smooth shifting and reduces wear

Anti-foam – ensures proper lubrication by reducing foam

Automotive Gear Lubricant Viscosity Classification

SAE Viscosity Grade	Maximum Temperature for Viscosity of 150 000 cP, °C	Kinematic Viscosity @ 100 °C, cSt min	Kinematic Viscosity @ 100 °C, cSt max
70W	-55	4.1	—
75W	-40	4.1	—
80W	-26	7	—
85W	-12	11	—
80	—	7	<11.0
85	—	11	<13.5
90	—	13.5	<18.5
110	—	18.5	<24.0
140	—	24	<32.5
190	—	32.5	<41.0
250	—	41	—

API Service Classification :

Class	Application	Formulation/Comment
GL-1	Manual Transmission Operated Under Mild Conditions	Straight Mineral Oil – No Friction Modifiers of EP Additives Permitted
GL-2	Worm-Gear Drives and Industrial Oils	Contain Anti-Wear and/or Very Mild EP Additives
GL-3	Manual Transmissions and Spiral-Bevel Axles Operated Under Mild to Moderate Conditions	Contains Mild EP Additives Not Suitable for Hypoid Gear Applications
GL-4	Manual Transmissions and Spiral Bevel Axles Operated Under Moderate Speeds and Loads	Usually Satisfied by 50% GL-5 Additive Treatment Level
GL-5	Hypoid and Other Gears Operated Under Moderate to Severe Conditions (High Speed and/or Low Speed, High-Torque Applications)	Primary Field Service Recommendation of Most Car/Truck Manufacturers.
GL-6	High-Offset Hypoid Gears Operated Under Severe Conditions	Equivalent to Some OEM Performance Requirements
MT-1	Non-Synchronized Manual Transmissions Used in Buses and Heavy Duty Trucks	Focus is on High Temperature Cleanliness and Oil Seal Performance

Typical Properties of Multigrade Gear Oil:
Type of Gear Oil 80W 140 LL
KINEMATIC VISCOSITY cst @ 100 C - 28-
33VISCOSITY INDEX,MIN 90
FLASH POINT, (COC) C,MIN
190POUR POINT, C , MAX -27
CORROSION COPPER STRIP
AT 121 C FOR 3 HRS MAX.
2POUR POINT, °C, Max (-
42)

OEM Specifications
Gear Oil 80 W 90
LL
KINEMATIC VISCOSITY, CST. @ 100°C 13.5 -24.0
VISCOSITY INDEX, MIN 90
FLASH POINT, (COC), °C, MIN
165POUR POINT, °C, MAX – 21
Gear Oil 75 w 90
KINEMATIC VISCOSITY, CST. @ 100°C 13.5 -
24.0VISCOSITY INDEX, MIN 170
FLASH POINT, (COC), °C, Min
180POUR POINT, °C, Max (- 36)

OEM specifications are needed to ensure performance in specific hardware: Industry standard is used as a base level of performance Factory fill and service fill specifications are common OEMs differ in their lubricant approval processes Specifications are being developed focusing on fuel economy and improved durability

OEM -Specifications and Approvals

OEM	Manual and Automated Manual Transmissions	Axle	Approval Type
Daimler	235.1, 235.4, 235.5, 235.11	235.0, 235.6, 235.8, 235.20	Issue formal approvals
Mack	Mack GO-J,GO-J Plus	Mack GO-J,GO-J Plus	Issue formal approvals. Requires SAE J2360 + seals.
MAN	341, 3343	342, 3343	Issue formal approvals
Volvo	97305, 97307	97310, 97312	No official approved oil list.

Extended Drain

- Higher quality base oil
- Highly shear stable VM

Wear Protection

- Thermal stability
- Anti-wear
- Improved durability

Operating Costs

- Improved fuel economy
- Increased durability

Hardware Designs

Compatibility with new hardware

- Improved Efficiency
- Lower viscosity
- Wide span multi-grades
- Gear Oils
- Gear Oils need to be balanced to meet the above demands of today's market

Extending Oil Drain Interval in the World

- Europe: 90,000 km >200,000 km>500,000 km
- North America: 100,000 miles>250,000miles>500,000 miles
- India: 36,000 km>72,000 km>150,000 km

Thermal Durability (Load + Temperature)

Ensures the oil will work to the correct performance level over an extended period of time, for a range of operating temperatures.

Why Higher Operating Temperatures?

- Greater power densities
- Hardware downsize
- Greater Loads
- Lower oil volumes
- Improved aero-dynamics
- Noise shielding

The Challenge of Escalating Costs Driving the demand for:
Reduced fuel consumption, less vehicle downtime, Lower maintenance costs. 49% of costs relate to repair, maintenance and fuel.

Requirements beyond API GL-5API GL-5 does not define:

- Base oil types employed in the formulation
- SAE viscosity grade (operating temperature)
- Shear stability
- Improved thermal stability
- Fuel efficiency
- Oil drain interval

Multi-grade Gear Oil Plus fuel efficiency:
Since oil must have viscosity at both low and high temperatures, their viscosity index must be sufficient high to achieve this. Multigrade automotive gear oils in SAE viscosity grades 75W80, 75W90, 80W90, 80W140 and 85W140 can be quoted as examples.
Multigrade automotive gear oils have more recently attracted interest in the context of fuel economy. According to one reference , fuel economy with heavily-loaded heavy duty truck gears improves by 1.2- 2.8 % if SAE 80W 140 multigrade gear oil is used. A synthetic 75W90 oil improved fuel economy in city travel by 2.2 % and in highway travel by 1.6% in comparison with 80W90 oil .
Suitable friction modifiers in gear oils can improve gear efficiency and decrease fuel consumption. The effect of friction modifiers increases with increasing temperature since oil viscosity decreases and the proportion of mixed friction increases. The oil temperature in the gearbox (final drive housing) first increase and then become stable for the whole time of operation of the vehicles, while the efficiency of the gears increases with increasing oil temperature in the presence of friction modifier.
Under severe condition of service, where low viscosity gear lubricants formulated with conventional sulphuro- phosphorus EP additives are thought to be questionable in terms of durability, an EP additives system based on a dispersion of potassium triborate has been shown to provide superior bearing and protection. The use of this borate EP additive system in low viscosity lubricant formulation provides, at the same time, the potential for improved fuel efficiency.
Multigrade gear oils, particularly those in the lower viscosity grades 75W, can be suitably prepared from part synthetic oils, e.g. from mixture of mineral oils with polyalpholefins or from fully synthetic oils. In comparison with petroleum-based oil, thee lubricants have low viscosities at low temperature and relatively high viscosities at temperature; they provide lubricant film with satisfactory load-carrying capacity i.e. gear efficiency to be increased, and fuel consumption to be cut.
Since synthetic components have high VI , partially or fully synthetic gear oils need little or no added polymeric viscosity and VI modifier, allowing high shear stabilities to be achieved (however, very high shear stable polymers, such as polyisobutene can be used). On the other hand oils with higher polyolefin content need a higher EP additive dosage to achieve the required performance; the reason for this does not appear to be clear.

In Collaboration with OEM, field trial was conducted at different conditions (both hilly and non-hilly locations) and Fuel Efficiency result of Multi-grade Gear Oil over Mono-grade is shown below:

Sample Size- 500							
Sl No	KM Mono-grade	KM MG GO	Fuel Cons (lit)	Oil Sump	Mono (Avg)	Multi (Avg)	Avg Fuel Efficiency (%)
Truck1	84002	91538	23401	36	3.59	3.91	
Truck12	42823	45926	15799	16	2.71	2.91	
Truck28	79394	85882	27364	11	2.90	3.14	
Truck45	63868	69825	18225	16	3.50	3.83	
Truck66	116559	127001	32805	16	3.55	3.87	
Truck78	52886	57178	14142	7.5	3.74	4.04	
Truck81	76243	84801	25654	16	2.97	3.31	
Truck92	119703	129449	33279	16	3.60	3.89	
Truck101	143950	158116	40543	16	3.55	3.90	
Truck117	65315	71186	25393	16	2.57	2.80	
Truck129	94160	105577	37995	16	2.48	2.78	
Truck136	147833	161164	41432	16	3.57	3.89	
Truck149	150779	160855	50476	16	2.99	3.19	
Truck157	80664	88627	31614	16	2.55	2.80	
Truck168	116288	131444	47304	16	2.46	2.78	
Truck179	160574	200649	51583	16	3.11	3.89	
Truck198	186212	200264	62843	16	2.96	3.19	
Truck219	99620	110341	39360	16	2.53	2.80	
Truck228	143615	163647	58893	16	2.44	2.78	
Truck231	198309	249808	64221	16	3.09	3.89	
Truck242	229972	249329	78239	16	2.94	3.19	
Truck253	123030	137374	49003	16	2.51	2.80	2.38 %
Truck267	177365	203741	73322	16	2.42	2.78	
Truck279	244911	311011	79955	16	3.06	3.89	
Truck282	284015	310415	97408	16	2.92	3.19	
Truck327	151942	160031	61009	16	2.49	2.62	
Truck348	219046	253658	91286	16	2.40	2.78	
Truck369	302466	387209	99544	16	3.04	3.89	
Truck377	350759	386467	121273	16	2.89	3.19	
Truck391	187649	212934	75956	16	2.47	2.80	
Truck399	270521	315804	113651	16	2.38	2.78	
Truck413	373545	482075	123933	16	3.01	3.89	
Truck434	433187	481151	150984	16	2.87	3.19	
Truck455	231746	265102	94565	16	2.45	2.80	
Truck466	334094	393175	141495	16	2.36	2.78	
Truck473	461328	600184	154296	16	2.99	3.89	
Truck488	534986	599033	187976	16	2.85	3.19	
Truck500	286207	330052	117733	16	2.43	2.80	

Field testing is not required for API GL-5 level of performance

OEM	Automated Manual Transmissions	Axle	Approval Type
Daimler	235.1, 235.4, 235.5, 235.11	235.0, 235.6, 235.8, 235.20	Issue formal approvals
Eaton	PS-164 Rev 7	NA	Issue formal approvals
			Issue formal approvals.
Mack	Mack GO-J, GO-J Plus	Mack GO-J, GO-J Plus	Requires SAE J2360 +seals
MAN	341, 3343	342, 3343	Issue formal approvals
			No official approved oil list.
		STO 1:0	Scania gives
Scania	STO 1:0		acknowledgement to
		STO 2:0A	
			products if proof of
			performance is provided
Volvo	97305, 97307	97310, 97312	No official approved oil list.

Changing Demands on Axle and MT Fluids

Market Needs	Impact on Lubricants
Extended Drain	Robust additive technology with performance retention
Reduced used oil disposal	Use of more thermally stable Group III and synthetic stocks
Lower maintenance cost	Highly shear stable VM to stay in grade
Wear protection	Better Low Temperature performance
Increasing loads and power density	
Higher operating temperatures	Thermal durable additive components

Reduce vehicle operating cost	Anti-wear for wider operating temperature coverage
Improved drivability/safety	Improved gear and bearing durability
Increasing complexity in hardware design	Reduce downtime – better durability
Newer hardware materials	Improve fuel economy
Improved efficiency	Compatibility and durability with newer hardware materials:
Downsizing, lighter materials	Synchronizer
More gear ratio in MT	Seal
Reduced fluid sump	Electric component
Global availability and supply reliability	Lower viscosity to reduce churning loss
	Wide span multi-grade GO for efficiency gains
	Need enabling DI and VM technology to deliver efficiency
	whilst not compromising durability
	Global regulatory compliance
	Sustainability

Summary:

Axles and transmissions require unique lubricants designed for the application enhanced fuel economy and emissions performance is a key market driver. Hardware design is changing and many OEMs design specific fluid requirements for their equipment using the wrong lubricant in an application can cause loss of performance and even hardware failure.

The case study shows that the friction in the transmission is responsible for temperature increase and efficiency losses, and thus the reduction of friction is the main measure in order to improve the efficiency and to keep the operating temperature low. GL-5 is not necessarily backward-compatible in synchro-mesh transmissions which are designed for a GL-4 oil: GL-5 has a lower coefficient of friction due to the higher concentration of EP additives over GL-4, and thus synchros can not engage as effectively. API viscosity ratings for gear oils are not directly comparable with those for motor oil, and they are thinner than the figures suggest. For example, many modern gearboxes use a 75W90 gear oil, which is actually of equivalent viscosity to a 10W40 motor oil. Multigrade gear oils are becoming more common; while gear oil does not reach the temperatures of motor oil, it does warmup appreciably as the car is driven, due mostly to shear friction with a small amount of heatconduction through the bell housing from the engine block.

It is also established that fully synthetic gear oils are also used in many vehicles, and have a greater resistance to shear breakdown than mineral oils.
It is also necessary to improve the efficiency by not only using lower viscosity gear oil, but also by making clear the power loss in automotive drive train.
Government legislation & increased competitiveness between automobile manufacturers have given the end user fuel efficient automobiles suited to many applications. Fuel economy drive is passionately pursued currently and has become more challenging in the light of dire consequences to the global environment, which includes global warming from the green house effect, increased acid rain and depletion of ozone layer. Automobile manufacturers are incorporating improved aerodynamics, lower RPM & higher torque drive lines with increased power densities in their current production. This trends will continue well into the next generation. As direct consequences of these trends,gear lubricants are required to perform under wide range in operating temperature and also promote the useful life of the gears and bearings they lubricate. Most automobile manufacturers are extending their drain interval to 100000 kms and in some instances it is closed to 200000 kms. Additive and lubricant manufacturers continue to strive to meet these demands with single and multigrade mineral, partial & full synthetic based gear lubricants.

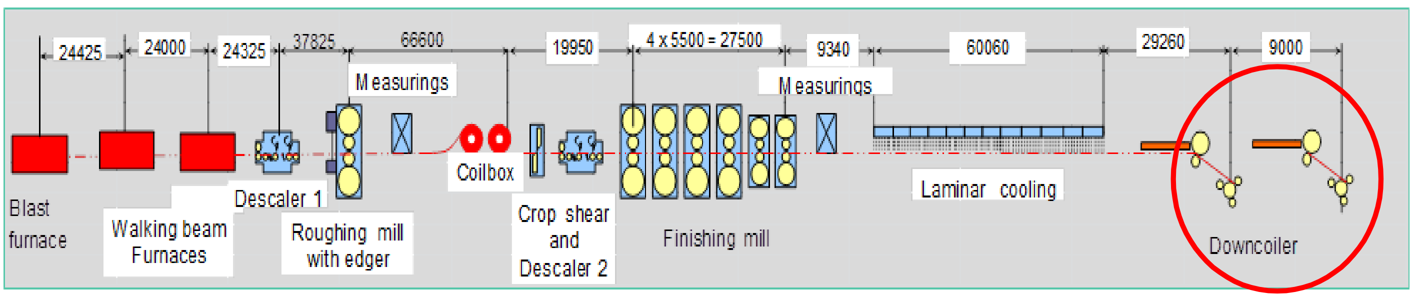
New age lubricant to minimize Man-Machine Interface

Manjunath.S[1], G.R.P.Singh[2], A.K.Jha & A.K.Verma –Tata Steel

In Steel Industry there is a growing demand of heavy duty industrial lubricants to increase the efficiency of the machine. With the technological advancements, the new age greases have been developed with various combination of additives, base oil and thickener to meet stringent & extreme application conditions.
Lubrication in steel industry is very challenging as the operating condition of machineries are very harsh with respect to very high load, temperature, dust and water ingress. Also due to extreme hazard conditionin steel industry, there is an increased demand of reducing man machine interface.
Additives are generally added in small amount to enhance the desired performance in the end application. Base oil of selected groups are picked to increase the lubrication interval and thickener are selected carefully to fulfil the application condition. In this study, there is an attempt to increase the lubrication efficiency as well as lubrication interval to reduce the work hazard during the time of lubrication and eliminate man machine interface. Earlier the re lubrication frequency was once in seven days with normal EP grease. Now its interval has been aligned with the monthly shutdown.

INTRODUCTION

Mandrel Outboard Bearing grease lubrication:
In Tata steel, Hot strip Mill is a vital plant for flat product segment. Maintenance activities of this plant are also vital for us.
A down coiler coils the finished steel strip of a hot strip mill onto a high temperature mandrel. The strip enters the down coiler from a pinch roll and is immediately formed around the mandrel using wrapper rolls. Unlike a traditional coiler, a hot strip mill down coiler utilizes a series of rolls and sensors to maintain tight and well aligned coil wraps. The down coiler eliminates telescoping coil sides and irregular wraps on a coil. This mandrel is cantilever which is supported with outboard bearing. During wrapping of strip, out board bearing give support to the mandrel to bear the strip load with the help of mandrel support arm which is hydraulically operated cylinder.



HOT STRIP MILL LAY OUT

Down coiler Mandrel

The Outboard bearing here at HSM is in a very hazardous location and due to hot coiling, online greasingis not possible because of existing mechanism. To keep outboard bearing healthy, manual greasing was done after every three days, which was very unsafe and time taking job.

ANALYSIS

Bearing Details:-

Bearing type:- **TRB: 93787/93127CD + X3S-93787**
ID:- 200.025 mm
OD :- 317.5 mm
Mean Diameter:- 258.76
Weight:- 40.58 kg.
RPM:- 1000
DN value:- 1000x258.76 = 258762
Working temperature:- 120˚C to 180˚C.

Existing Scenario:- Present greasing interval is 3 days with normal Lithium Grease. Since the area is exposed to Very High radiation, heat hence the manual greasing becomes extremely difficult. The concerned department approached us for a solution in terms of enhancing the re-greasing interval. This greasing activity was carried out during the roll change time which is approximately 10 mins. Hence doing manual greasing in the down coiler area in that specified time becomes very difficult due to presence of water in this area and grease spillage on the floor.

Developmental Study: Firstly the properties of existing grease were tabulated. Then looking at the properties of grease and comparing with the **tribological requirement** of the system, it was concluded that the grease in this area should have the following properties.

- Should have very high Oxidation stability.
- Should have very high Drop point.
- Grease should have the DN factor within range.

Upon looking at the various tribological requirement of the application we have zeroed on grease having the below properties.

- Grease with PAO as the base oil stock has inherent very good thermal stability.
- PAO greases can work in wide temperature range.
- High Viscosity Index compared to normal mineral oil based grease.

The comparison with the normal lithium grease with Polyurea grease is given below.

	Normal EP-2	Poly urea/Synthetic
Base oil	Mineral	Polyalphaollefins
NLGI	2	2
Drop Point in ˚C	180	>240
Thickener	Lithium	Polyurea
Viscosity @40˚C	180 cst	400 cst

Trial Insights

It was observed that after 7 days of application of new Synthetic-Urea grease, there is no any abnormality observed in terms of rise of bearing temperature or abnormal sound. The used grease sample was then collected after another 7 days during the Back-up change of the Mill. Upon analysing no any depletion of properties observed in the used grease sample.

Conclusion:

After the successful trial, it can be concluded that use of high performance synthetic grease in combination of Polyurea thickener has increased the re-greasing frequency in outboard bearing of down coiler mandrel and it has eliminated frequent **man-machine interface** as well as **high risks associated** with it without compromising the equipment healthiness.

At present the greasing is scheduled for 45 days once. The main benefit is in terms of lesser Man Machine Interface. Intangible benefit is mainly the elimination of work hazard as greasing activity was carried out in a very lesser time frame.