Grease Evaluation Under Oxidative Conditions and Resulting Friction and Pressure Analysis

Authors:
William VanBergen
Savant Inc

Canika Owen-Robinson
Savant Inc

Greg Miiller
Savant Inc

Richard Wurzbach
MRG Corp

Sloan Healy
MRG Corp.

1. Introduction

Lubricating grease usage covers a wide range of applications, which include wind turbines over salt water to modern electric vehicles. In recent years, not only has the utilization of grease increased, but the number of applications has also grown. This has contributed to fluctuations in the lubricating grease supply chain. Due to the disruptions in the supply chain many end-users have found it difficult to obtain the recommended grease for their application. The supply chain issues do not stop with the end-product but have also created disruptions with packaging and additive availability. Often it is tempting to have additional stock of recommended lubricating grease on hand, however many greases have finite storage intervals which discourage such practices.

In many applications, to meet equipment maintenance requirements, it has become necessary for end-users to replace or refill existing grease that has a long history of reliability, with new grease of similar composition. As in lubricating oils, grease additives are designed to impart or enhance specific properties. Among these are oxidation and rust inhibitors, extreme pressure (EP) additives, and anti-wear additives. Within the oil industry, it has been observed that mixtures of EP additives and corrosion inhibitors can have both synergistic and antagonistic effects on the tribological properties of the fluid [1,2]. Therefore, when combining greases that have different additive packages in the base oil, it is advantageous to examine the wear properties of the mixed lubricant compared to the lubricant being replaced.

2. Experimental

2.1 Grease Composition and Physical Properties

Greases that utilize similar thickeners are often believed to be compatible and therefore used to replace or blend with the original lubricant. For this reason, two pairs of greases were chosen to be blended in 3 ratios: 10:90, 50:50, and 90:10.

	Grease 1	Grease 2	Grease 3	Grease 4
Thickener Type	*Polyurea*	*Polyurea*	*Lithium complex*	*Lithium complex*
Penetration Worked ASTM D217 (0.1mm)	*285*	*280*	*305*	*315*
Viscosity of Oil ASTM D445 cSt @ 40C	*115*	*116*	*460*	*302*

Figure 1 Physical Properties and Thickener Type

2.2 Grease Working and Thermal Aging

Traditional shear stability is tested by ASTM D6185 at 100,000 double strokes in a grease worker [4]. To better emulate in-service conditions, an extended number of mixing events was utilized using a Dayton Industrial ¾ HP motor. Each grease blend along with and each neat grease were subjected to 18 million mixing events over 72 hours in the grease worker. At this point 6.5 grams were transferred to a glass dish and conditioned using an isothermal pressure vessel at 120 C for 50 hours, using a head pressure of 90 psi at room temperature. After 50 hours of conditioning, each sample was then transferred to a glass vial until tribological testing was performed.

2.3 Tribological Testing by SRV

One of the main functions of lubricating grease is to prevent metal machine components from getting into rubbing contact with one another. Two test methods validated by the ASTM for the evaluation of the tribological properties of greases by SRV are ASTM D5706 and ASTM D5707. ASTM D5706 is used to measure the EP capacity of a grease using incremental loading in two minutes intervals until the lubricant fails, indicated by a large spike in the coefficient of friction [5]. ASTM D5707 is an endurance test that examines the coefficient of friction and wear scar data using a static, moderate load [6].

For this study the following test parameters were used for all SRV testing:

ASTM D5707 Run Conditions	
Path Length	1.5 Millimeters
Frequency	50 Hertz
Temperature	50 Centigrade
Break-in Load	50 Newtons for 30 Seconds
Test Load	250 Newtons for 1 Hour 59 Minutes and 30 Seconds

ASTM D5706	
Path Length	1.0 Millimeters
Frequency	50 Hertz
Temperature	50 Centigrade
Break-in Load	50 Newtons for 30 Seconds
Run-in Load	100 Newtons for 15 Minutes
Step Load	100 Newtons every 2 Minutes

Figure 2 SRV Experimental Conditions D5707 & D5707

For both test protocols, a steel ball (10 mm diameter) is used as an upper specimen and a disk (24mm diameter, 7.9mm height) is used as the lower specimen. Both materials are made of tempered steel 100 Cr6 (AISI 52100). A small amount (approximately 0.2 grams) of test grease is placed on the interface between the two specimens and the initial load applied. Prior to starting the test program, the instrument is allowed to come to the equilibrium test temperature of 50 C. For the step test (ASTM D5706), a maximum coefficient of friction was established of 0.3 to prevent possible damage to the equipment and would indicate the failure load. For future comparative analysis, we will be using the last passing load (LPL). In the sample results below, the LPL would be 1000 Newtons.

Figure 3 SRV Output ASTM D5706, Grease 3&4

Analysis conditions for the SRV Endurance test were run under the conditions in Figure 2 for ASTM D5707. When comparing the Coefficient of Friction between samples, the mean and minimum coefficient friction calculated using the baseline results from 1 to 120 minutes. The first minute was ignored to allow the test apparatus to complete the initial break-in for the ball and disk and establish a consistent boundary film.

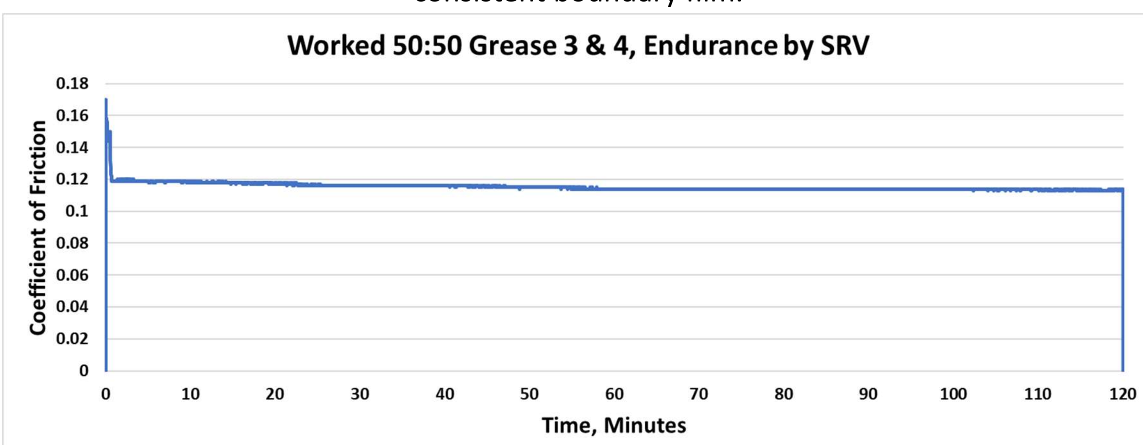

Figure 4 SRV Output ASTM D5707, Grease 3 & 4

3. Results and Discussion

3.1 Step Test Analysis

3.1.1 Neat Greases

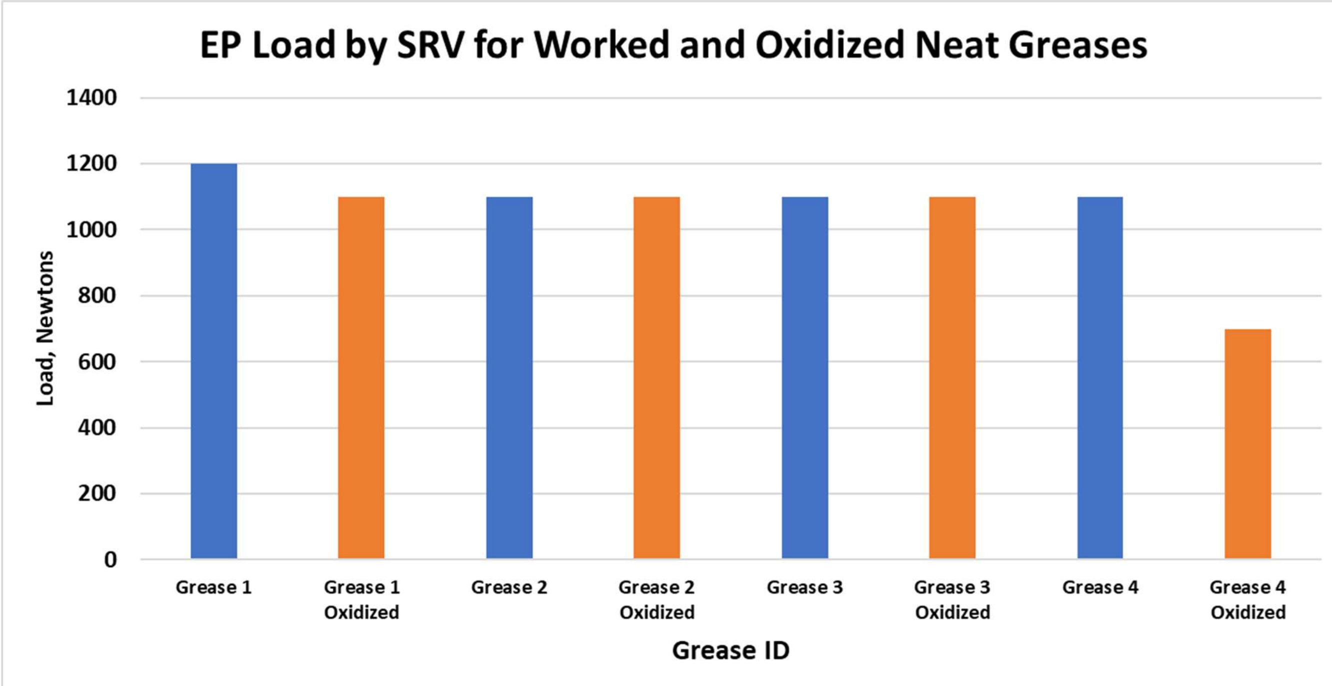

Figure 5 EP Load of Neat Greases by SRV

Three of the four neat greases showed little change between the pre and post conditioned samples. Only Grease 4 showed a significant decrease in EP load carrying ability between the worked and worked/oxidized samples. This is most likely due to thermal decomposition of the additive package during the thermal conditioning step. Grease 4 also showed the most dramatic visual change between the pre and post conditioned grease (Figure 6):

Figure 6 Grease 4 Pre and Post Conditioning

We also observed a greated degree of oxidation at the contact area when examining the wear scar at the end of test. This is due to the large amount of heat generated at the point of contact when the boundry layer fails at the end of test. Below is an example of this behaviour on one of the Polyurea greases (Grease 2)

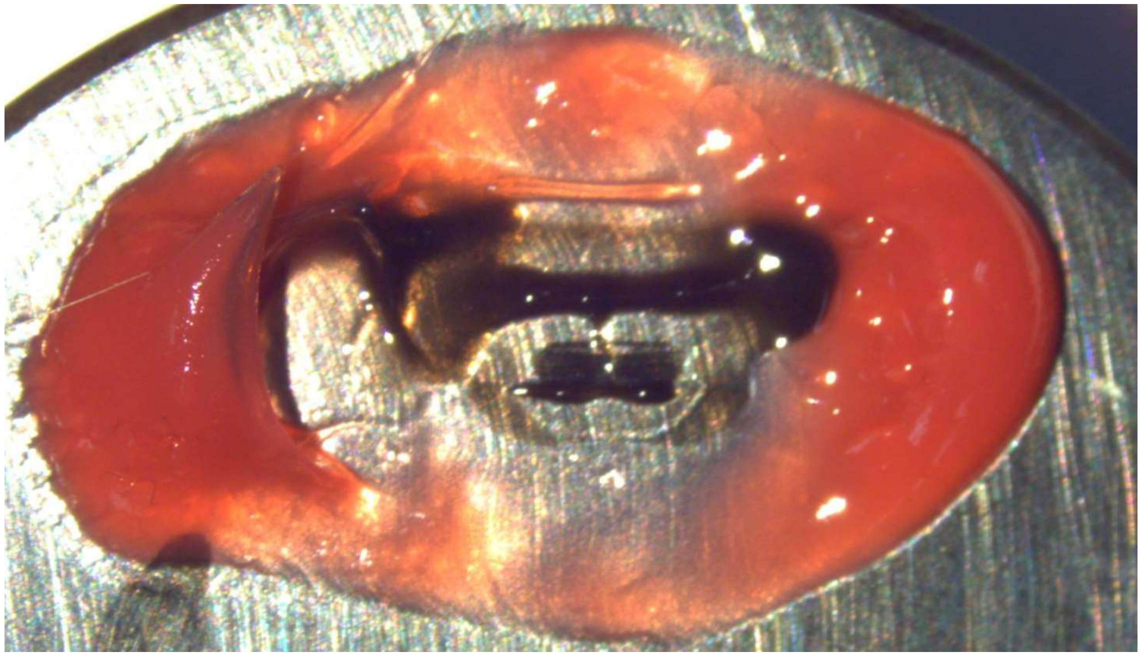

Figure 7 EP contact with Oxidized grease

This can also be seen on the thermal readout of the temperature controller. As the mechanical contact begins to fail, increases of several degrees celsius are seen on the thermal controler. In all cases, this temperature increase was a precursor to the EP failure for the grease in question. Below is a example of this behavior on one of our test samples.

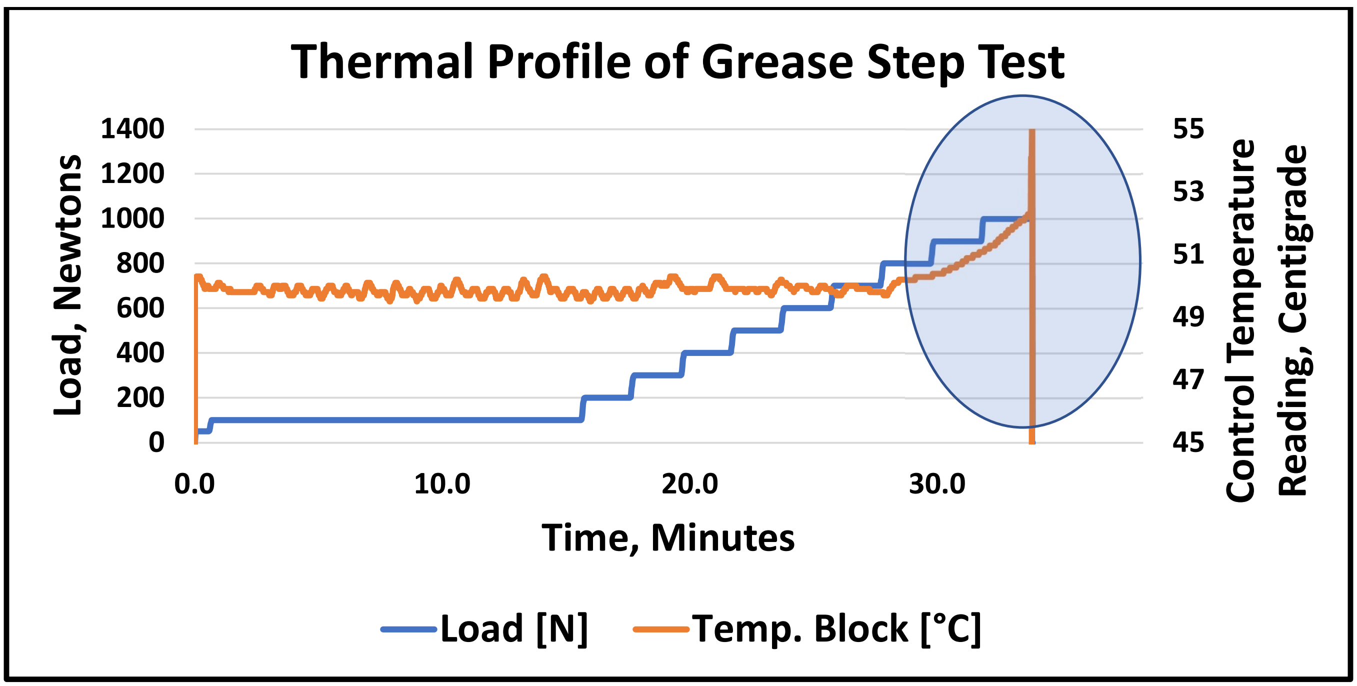

Figure 8 Thermal control loss due to friction

3.1.2 Blended Greases

Two Sets of Greases selected by thickener type were blended in 10:90, 50:50 and 90:10 ratios and worked and approximately seven grams of the worked grease was thermally conditioned. Portions of both the worked and conditioned grease were analyzed with the SRV Step test.

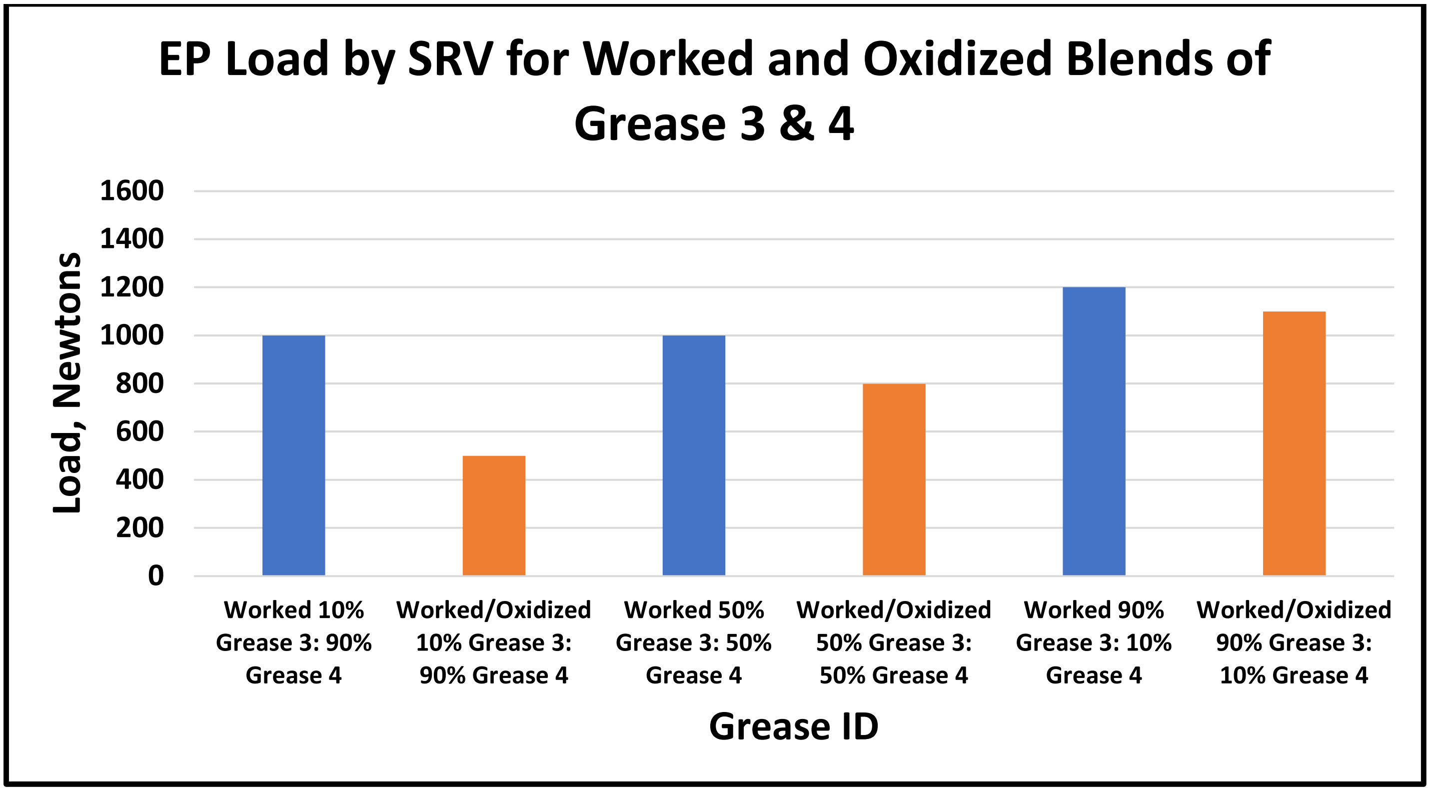

Figure 9 EP Load Results, Grease 3 & 4 Blends

The data above shows the LPL results from the blends of greases 3 & 4. For the conditioned (oxidized) sample, it can be observed that the EP load limit decreases with increasing level of grease 4, which exhibited a decrease in EP performance during the analysis of the neat greases. In fact, the conditioned 10:90 blend performs worse than its neat analog. It is possible that some detrimental effects are amplified withing the grease blends that cannot be wholly subscribed to individual performance of the test grease.

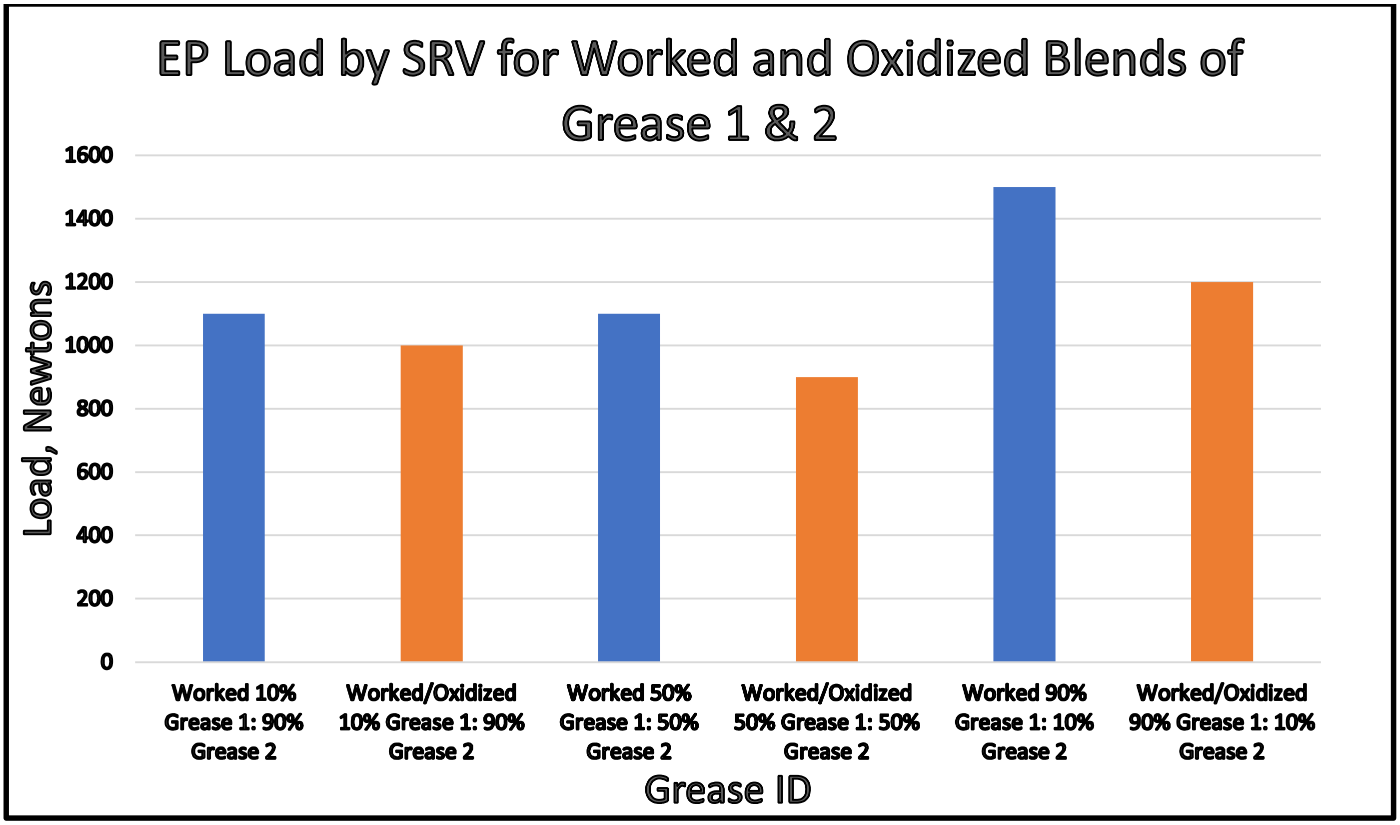

Figure 10 EP Load Results, Grease 1 & 2 Blends

The blends of greases 1 & 2 exhibit a small decrease in EP load capacity for the 10:90 and 50:50 blends, with a slightly larger loss in the conditioned samples. The 90:10 blend is the most interesting as we see an increase in EP load capacity. An additional replicate run of the worked 90:10 grease sample yielded a result of 1400 Newtons. This type of synergistic response is not unheard of in boundary film analysis [6] and could be an example of the synergistic effect being concentration dependent.

Examination of the EP characteristics of blended greases can be difficult to predict, and both the level of additive package fatigue and composition ratios can have a pronounced effect on the ability of EP additives to protect contact surfaces at higher load requirements.

Additionally, exothermic response from metal-metal abrasion can be predictive indicator of potential EP load failures when examined on the SRV tribological tester.

3.2 Endurance Testing by SRV

3.2.1 Neat Greases

Endurance testing was run at a moderate static load for 120 minutes. Both the mean and minimum coefficient of friction were calculated over the interval from one minute until the end of test at 120 minutes.

Grease ID	Average Coefficient of Friction	Minimum Coefficent of Friction
Grease 1	0.12	0.119
Grease 1 Oxidized	0.122	0.119
Grease 2	0.122	0.119
Grease 2 Oxidized	0.115	0.113
Grease 3	0.117	0.114
Grease 3 Oxidized	0.126	0.116
Grease 4	0.118	0.115
Grease 4 Oxidized	0.112	0.098
Worked 10% Grease 1: 90% Grease 2	0.118	0.116
Worked/Oxidized 10% Grease 1: 90% Grease 2	0.115	0.109
Worked 50% Grease 1: 50% Grease 2	0.115	0.113
Worked/Oxidized 50% Grease 1: 50% Grease 2	0.119	0.114
Worked 90% Grease 1: 10% Grease 2	0.117	0.114
Worked/Oxidized 90% Grease 1: 10% Grease 2	0.115	0.113
Worked 10% Grease 3: 90% Grease 4	0.119	0.109
Worked Oxidized 10% Grease 3: 90% Grease 4	0.111	0.104
Worked 50% Grease 3: 50% Grease 4	0.115	0.113
Worked/Oxidized 50% Grease 3: 50% Grease 4	0.111	0.095
Worked 90% Grease 3: 10% Grease 4	0.125	0.115
Worked/Oxidized 90% Grease 3: 10% Grease 4	0.123	0.116

Figure 11 Average and Minimum CoeF by SRV

Examination of the raw numerical results shows little difference between the individual formulations and their oxidized counter parts, with a slight downward trend being observed. On closer examination of the full tribological profile between samples, some interesting information is obtained.

When examining the profile of the worst performing neat grease in out step-test analysis, we see a very intriguing tribological profile compared to the typical endurance profile observed for all the samples that do not contain Grease 4 as part of the blend:

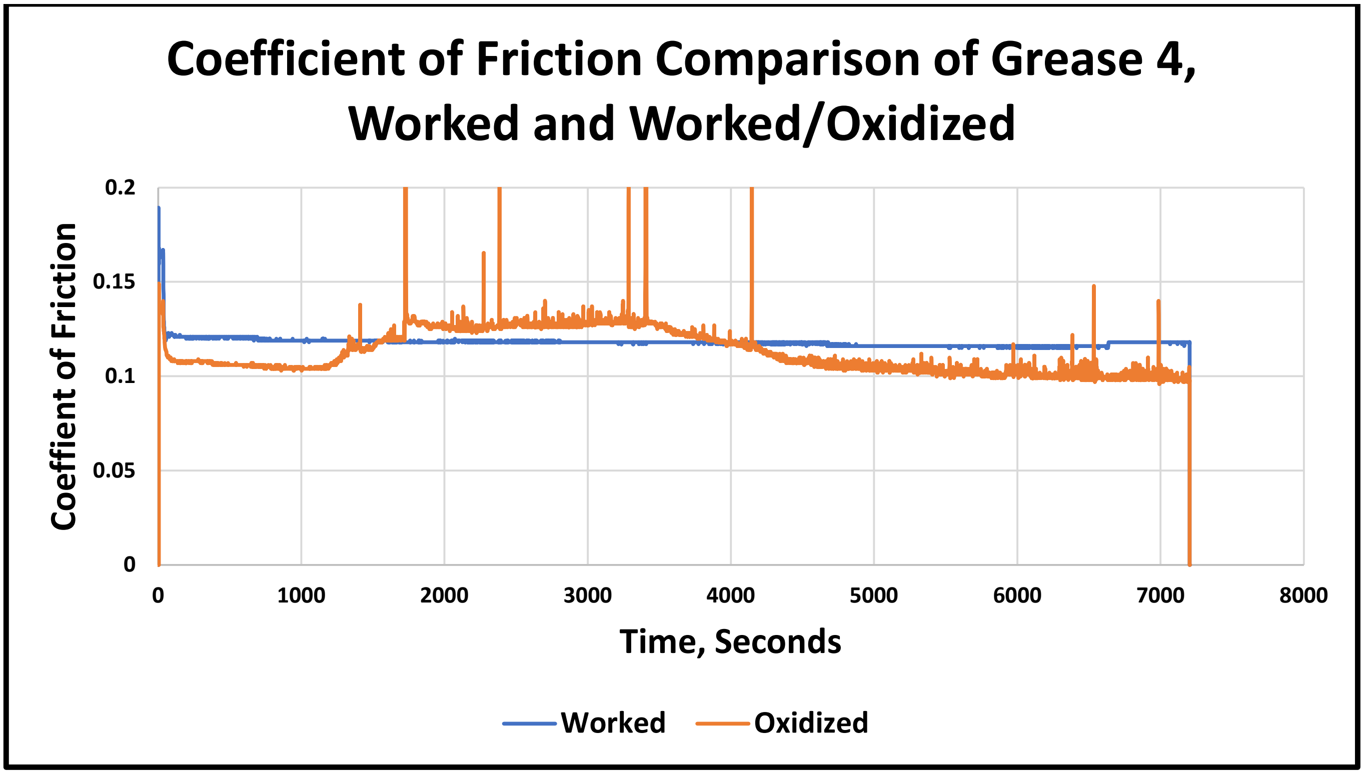

Figure 12 Endurance Test Profile Grease 4

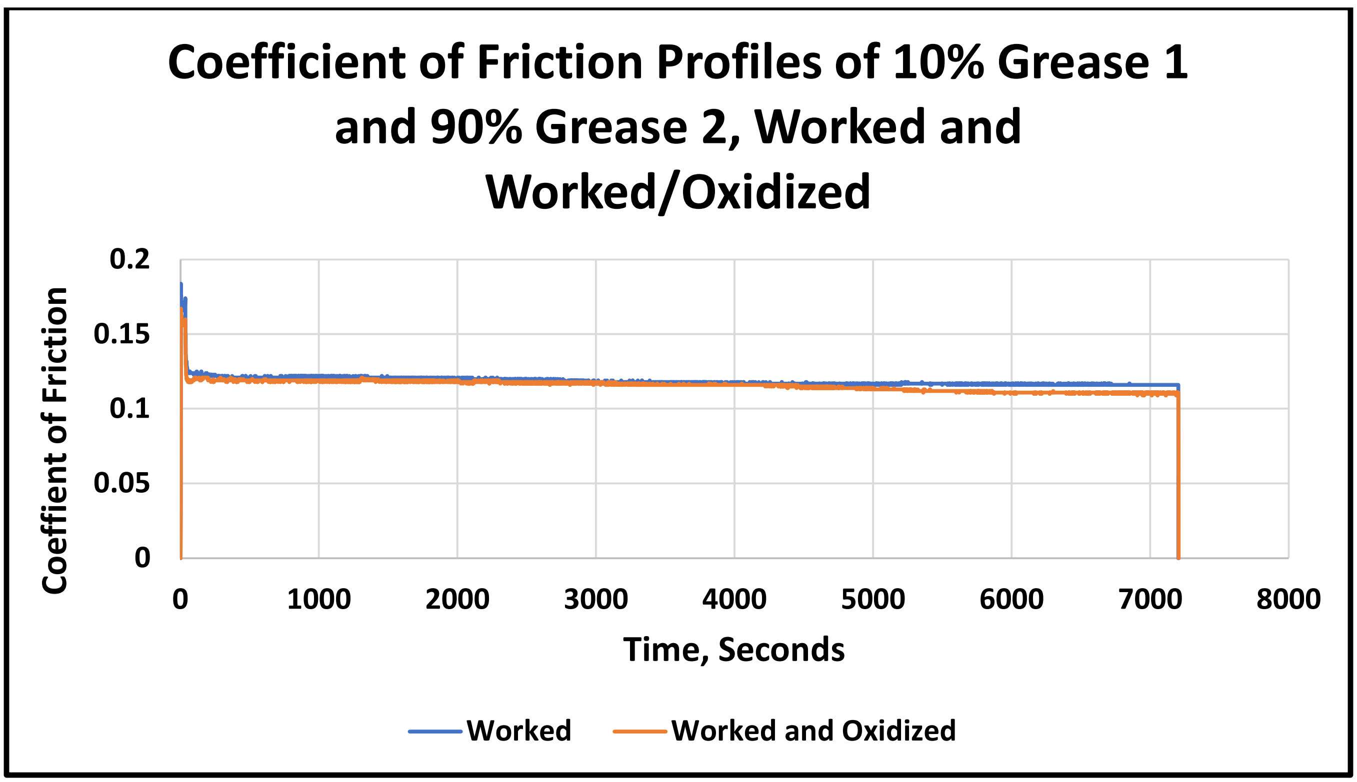

Figure 13 Typical Endurance Profile

Looking at the Coefficient of Friction profile of the oxidized sample of grease 4, we see a much more erratic response compared to that worked grease. This response volatility is indicative of inconsistent boundary layer lubrication with the oxidized grease and is likely caused by oxidative stress from the conditioning step. Further investigation into the relative level of oxidation achieved during the thermal conditioning is worthy of initial investigation.

4 Conclusions and Next Steps

Lubricating greases are a complex mixture of components formulated to achieve the lubrication requirements across a vast array of applications. Examination of the load bearing characteristics of both neat and blended greases is valuable in determining whether a lubricant meets the operational parameters for a given application.

EP performance comparisons indicate that thermal decomposition can adversely affect the maximum load capacity of the grease. Utilization of tribological testing of an in-service lubricant may prove beneficial in detecting EP reduction, minimizing the likelihood of damage to critical contact surfaces. Further examination of the exothermic response exhibited prior to the seizure load may provide a useful testing parameter for analysis of boundary films. Discreet test values do not always give a complete picture of a grease's performance. Comparative analysis of the friction profile can provide indicators to the overall health of the grease. A detailed analysis of the oxidative state of each grease will be forthcoming to provide a more complete analysis of oxidation on wear characteristics.

5 References

1. KK Mistry, A Neville, A Morina, MN Webster. "Lubricant/surface interactions under extreme pressure conditions: Corrosion inhibitor interacting extreme pressure/anti-wear additives on steel surface."

2. Wong VW, Tung SC. "Overview of automotive engine friction and reduction trends—effects of surface, material, and lubricant-additive technologies." Friction. 2016;4:1-28. doi: 10.1007/s40544-016- 0107-9

3. ASTM D6185: Standard Practice for Evaluating Compatibility of Binary Mixtures of Lubricating Greases. ASTM Digital Volume D6185

4. ASTM D5706: Standard Test Method for Determining Extreme Pressure Properties of Lubricating Greases Using a High-Frequency, Linear-Oscillation (SRV) Test Machine. ASTM Digital Volume D5706 Section

5. ASTM D5707: Standard Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine. ASTM Digital Volume D5707 Section

6. A Suresh. "Synergistic and Antagonistic Effect of Sulfur-Based Additives on the Performance of Greases with ZDDP and PTFE", Master of Science, University of Texas at Arlington, 3-30-2010, http://hdl.handle.net/10106/2042