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An Investigation into the Use of Boron Esters to Improve the High-Temperature Capability of Lithium 12-Hydroxystearate Soap Thickened Grease

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Abstract

Lithium complex greases are commonly specified for use in high-temperature applications, such as automotive wheel bearings. The usual thickener system in lithium complex grease consists of the lithium salts of 12-hydroxystearic acid plus a complexing agent. The complexing agent is usually a di-acid such as azelaic or sebacic acid that together with the 12-hydroxystearic acid yields a conventional complex soap. The complex soap increases the high-temperature capabilities of the grease, expanding the upper temperature limit for application. The higher value of the increased high-temperature application range is balanced against higher complex soap content, increased batch manufacturing cycle time and reduced pumpability compared to simple lithium grease.

Boron esters are known to have the effect of raising the dropping point of grease containing lithium 12-hydroxystearic acid based soap. The difference between this technique and the use of conventional complexing agents is that the boron esters do not need to be present in the cooking stage; instead they are added along with other additives, at the finishing stage of a typical batch manufacturing process. For the grease manufacturer, this adds significant flexibility as well as cost savings over conventional methods of obtaining high-temperature soaps.

In this paper, we compare the characteristics of lithium 12-hydroxystearate grease additized with boron ester to conventional lithium complex grease based on azelaic acid. Manufacturing cycle time, high-temperature rheology and low-temperature grease flow properties are compared for the two types of high-temperature lithium grease. Finally, grease characteristics of both types of fully formulated lithium complex greases are compared to the GC-LB requirements of the ASTM D4950 standard classification.

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Introduction

Lithium complex grease is marketed as a superior tier of performance versus lithium simple soap grease. This superiority is a result of increased operating temperature limits, which can be more than 100°F (56°C) higher than the simple lithium soap.[1] Lithium complex thickened grease has maximum temperature limits superior to that of simple lithium grease, because the thickener offers higher thermal degradation limits.[2] According to the NLGI Lubricating Grease Guide, the two types of lithium grease "are quite similar, except in dropping point and in the heat resistance which this indicates."[1] When differentiating lithium greases in the NLGI Annual Production Survey, a dropping point of 210°C (410°F) or above signifies the grease production volume be attributed to the "complex soap" category.

A complex soap is formally defined by the NLGI as "a soap wherein the soap crystal or fiber is formed usually by co-crystallization of two or more compounds: (1) the normal soap, and (2) the complexing agent."[1] In further explanation, the complexing agent is said to "bring about a change in grease characteristics usually recognized by an increase in dropping point."[1] While co-crystallization requires the two compounds to be present during the high temperature heating phase of the grease cooking process and for them to crystallize in close contact together during a transition from fluid to semi-solid form, the caveat "usually" indicates that this is not the only method of complex formation. It has been concluded that during the "complexing" process there is not a formation of new chemical compounds, but rather strong adsorption interactions develop between the soap and complexing agent during preparation of the grease.[3]

Boron esters are known to increase the dropping point of conventional metallic soap derived from 12-hydroxystearic acid.[3] In demonstrating the effect of improving the dropping point of the conventional soap grease, boron ester additized lithium greases meet the NLGI definition for lithium complex grease.[1] While the mechanism for the improvement of dropping point to the level of complex soaps is not well understood, the explanation presented by Siegart and Henry[3] theorized that a stable, coordinated compound is formed by electron sharing between the boron atom of the borate ester compound and the hydroxyl group of the hydroxy fatty acid soap. In doing so, the interactions between soap and complexing agent produce a change in the high-temperature properties of the finished grease, allowing it to maintain its consistency far beyond the melt point of simple soap grease.

Simple soap greases are widely used for general purpose applications where temperatures do not exceed 130°C (266°F). Simple lithium soap greases based upon 12-hydroxystearic acid have been regarded as one of the best performing low-temperature greases, while the complex lithium soaps have been regarded as one of the best performing high-temperature greases.[2] Greases that claim operating temperature ranges exceeding 130°C (266°F) are typically formulated with complex soaps. In this regard, complex greases are commonly referred to as high-temperature greases because of the increase in dropping point that comes from the two-part thickener system. The additive modification by incorporation of a boron ester bridges the gap between these two types of grease. The lower soap content of simple soap grease offers less resistance to flow at low temperature combined with the heat resistance of complex grease.

The high temperature properties of grease are commonly tested by the ASTM method D2265 dropping point test, which measures the temperature at which the thickener can no longer hold the base oil, under the static conditions of the test. As shown in *table 1*, the effect that boron esters have on the high temperature dropping point of lithium soaps of 12-hydroxystearic acid is the equivalent of conventional methods of obtaining high dropping point soaps, such as by formulating with azelaic acid.

	Grease A	Grease B	Grease C
Description	Base Lithium 12-hydroxystearate	Base Lithium 12-hydroxystearate + 3% wt Boron Ester	Lithium Azelate Complex
Dropping Point	207°C (405°F)	283°C (541°F)	274°C (525°F)

Table 1. ASTM D2265 Dropping Point Comparison

While offering comparable effectiveness to azelaic acid in the elevation of the grease dropping point, there are differences with boron esters that may prove useful to a grease manufacturer. Unlike the most widely used di-acid type complexing agents in a typical lithium complex manufacturing scheme, boron esters are unique in that the complexing effect they produce does not require the presence of the component during the cooking stage of soap manufacture. Instead, the boron ester is added along with other performance enhancing additives, such as extreme pressure, antiwear, antioxidant and corrosion inhibitors, near the end of the manufacturing cycle, just prior to packaging.

In this paper, we compare the characteristics of lithium 12-hydroxystearate grease additized with boron ester to conventional lithium complex grease based on azelaic acid. Manufacturing cycle time, high-temperature rheology and low-temperature grease flow properties are compared for the two types of lithium complex grease. Finally, grease characteristics of both types of fully formulated lithium complex greases are compared to the GC-LB requirements of the ASTM D4950 standard classification.[5] The work performed and presented includes selection of materials to prepare samples, grease preparation, and comparative performance testing.

Experimental

In the present study, three greases were prepared. The first lithium complex grease was prepared using 12-hydroxystearic acid and azelaic acid. The second (experimental) lithium complex grease was prepared using 12-hydroxystearic acid and a boron ester added post-manufacture. The baseline lithium 12-hydroxystearate served as a control.

Materials

The first step was to select materials used to make greases for comparison. ISO 220 VG oil blend was selected as being representative of the majority of industrial and automotive applications. To achieve this viscosity, a shear stable polymer was used to increase viscosity from the baseline value. *Table 2* shows viscosity and viscosity index (VI) as determined by ASTM D2270 for the base oil blend. The composition of the experimental greases was held as close as possible to remove external influences. This ensured a valid comparison among tests results among all three greases.

Component	**Grease A** Lithium Soap		**Grease B** Lithium Soap + Boron Ester		**Grease C** Lithium Azelate Complex Soap	
	Grams	**Percent Wt**	**Grams**	**Percent Wt**	**Grams**	**Percent Wt**
Base oil blend 88% Group II 600N 12% Polymer	6800	87.25	6566	84.25	7500	85.68
12-hydroxystearic acid	800	10.26	800	10.26	700	8.00
Lithium hydroxide dispersion	194	2.49	194	2.49	330	3.77
Azelaic acid					223.36	2.55
Boron ester			234	3.00		
Total	7794	100.00	7794	100.00	8753.36	100.00
NLGI Grade	2		2		2	
Dropping Point, °C	207		283		274	
% Free LiOH	0.07		0.07		0.07	
% Soap	10.56		10.56		11.00	
Base Oil Properties						
cSt @ 40°C	220		220		220	
cSt @ 100°C	20.5		20.5		20.5	
Viscosity Index	109		109		109	

Table 2. Experimental Grease Composition and Characteristics

Grease preparation - open kettle process

Both simple lithium and lithium complex greases were prepared in a pilot-size open kettle. *Figures 1 and 2* show the general manufacturing procedures used.

Lithium 12-hydroxystearate grease (using anhydrous LiOH dispersion)

1. Charge oil and 12-hydroxystearic acid are added to the open kettle.
2. Heat is applied to reach 80°C (176°F) to melt the acid.
3. At 90°C (194°F), the anhydrous lithium hydroxide dispersion is added slowly to the kettle over 20 minutes.
4. After all the lithium hydroxide dispersion is added, 15 minutes further mix time is completed at a higher rpm mixing speed (if possible).
5. Following the 15 minutes mixing time, the mixing speed is reduced and full heat is then applied to initiate the temperature ramp to the 195-200°C (383-392°F) process maximum.
6. Once the 195-200°C temperature stage is reached, heat is shut off and 15 minutes max additional mix time is allowed before cooling oil is applied at a slow, controlled rate.
7. The batch is cooled to below 170°C (338°F) and preferably to 150°C (302°F) then transferred to the finishing mixer.
8. Below 90°C (194°F) the grease is milled and the penetration and dropping point are tested.
9. Final adjustment oil can be added to achieve desired grade.

Figure 1. Grease Making Process – Simple Soap

Lithium Complex Grease (using anhydrous LiOH dispersion)

1. Charge oil and azelaic acid are added to the open kettle.
2. Heat is applied to reach 100°C (212°F) to melt the azelaic acid.
3. Upon confirmation of the azelaic acid melting, 12-hydroxystearic acid and cooling oil are added to reduce temperature down to about 90°C (194°F).
4. At 85-90°C (185-194°F), the anhydrous lithium hydroxide dispersion is added slowly to the kettle over 20 minutes.
5. After all the lithium hydroxide dispersion is added, 15 minutes further mix time is completed at a higher rpm mixing speed (if possible).
6. Following the 15 minutes mixing time, the mixing speed is reduced and full heat is then applied to initiate the temperature ramp to the 190-195°C (383-392°F) process maximum.
7. Once the 195°C temperature stage is reached, heat is shut off and 15 minutes max additional mix time is allowed before cooling oil is applied at a slow, controlled rate.
8. The batch is cooled to below 170°C (338°F) and preferably to 150°C (302°F) then transferred to the finishing mixer.
9. Below 90°C (194°F) the grease is milled and the penetration and dropping point are tested.
10. Final adjustment oil can be added to achieve desired grade.

Figure 2. Grease Making Process – Complex Soap

Grease manufacturing comparison

The time from start to finish of a grease batch is closely studied, as a significant savings in energy can be obtained by reducing the batch process cycle time. An increase in the grease plants production capacity can also be achieved by shortening the process cycle time, as an increased number of grease batches can be produced in the same equipment. This is one method of increasing the output of a grease plant without investing in additional capital equipment.

According to published literature examples, simple soap lithium greases can be prepared in a much shorter time than complex soap lithium greases. One example that is well cited in many NLGI papers shows a contactor process finished in five hours for simple lithium soap, compared to a seven hour process for the complex lithium counterpart. When less efficient open kettles are used, the process length is further extended, with the simple soap completed in seven hours versus nine hours for the complex.[6] The result of faster batch cycle times is improved throughput, higher efficiency and lower costs.

The soap content of typical complex lithium grease is generally higher then the same consistency simple lithium grease[6] because of the reduced thickening power of the low molecular weight salts used to form the complex soap. High soap levels can negatively influence the pumpability and low temperature flow characteristics of the finished grease. The higher soap requirement also results in greater lithium hydroxide consumption to neutralize the complexing acids. As *figure 3* indicates, lithium hydroxide monohydrate has undergone a significant increase in price over the last decade and is expected to further increase with strong demand from growing non-grease sources.

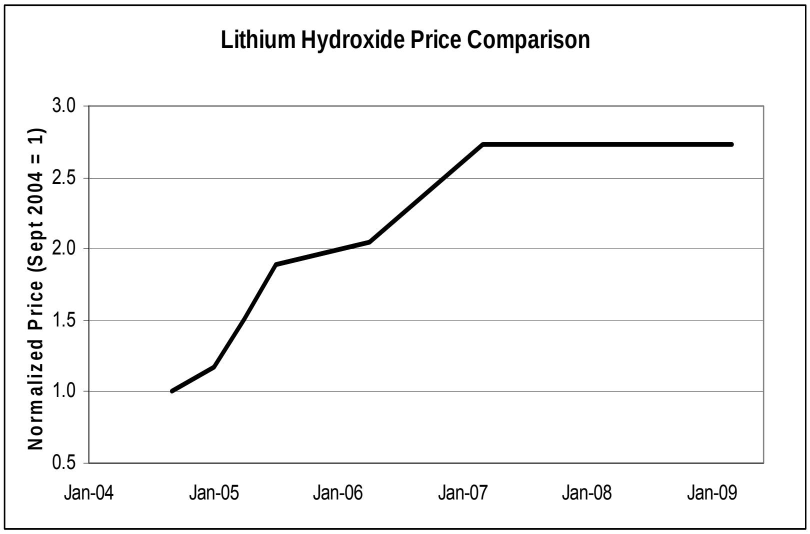

Figure 3. Normalized LiOH Monohydrate Prices 2004 to 2009

Grease prepared using the boron ester additive as the method of complexing is advantageous because it follows the shorter production cycle of simple lithium soap. In this regard, simple soaps additized with boron esters are faster to manufacture than conventional complex lithium soaps. Manufacturers are also able to simplify the production of lithium grease by utilizing only one cooking procedure for both simple and complex greases. The process of filling part of a batch as simple lithium grease, and then converting the remaining portion of the grease with boron ester additive to obtain complex grease allows great flexibility in manufacturing. Unlike acidic complexing agents, boron esters do not require neutralization with lithium hydroxide. The reduction in processing time and lower lithium requirement for the production of lithium complex grease by using boron esters creates an economic advantage to the grease manufacturer using this technology.

Performance testing

Comparative performance testing of the three types of unadditized greases included grease characteristics, high-temperature and low-temperature testing. Grease is expected to perform over a range of both high and low temperatures. High-temperature greases are expected to perform in equipment operating at sustained temperatures of 120-150°C (248-302°F). In the case of automotive wheel bearings, greases meeting the performance requirements of ASTM D4950 category GC must be able to satisfactorily lubricate wheel bearings over a wide temperature range. The standard states that “The bearing temperatures may range down to -40°C (-40°F), with frequent excursions to 160°C (320°F) and occasional excursions to 200°C (392°F).”[5] Finally, differences and similarities of fully additized greases showed how complex grease made with boron ester compared to traditional lithium complex grease in order to properly evaluate the high temperature properties.

Grease characteristics

The simple lithium grease, complex grease made from the original simple lithium grease combined with boron ester, and the complex grease made with azelaic acid all were NLGI#2 grade grease (*Table 2*). Grease B and grease C both gave dropping points greater than 260 ºC (500°F) showing that a good complex had formed. Grease B made from the boron ester had a higher dropping point at a lower soap level than grease C made with azelaic acid.

High temperature testing

High temperature testing was done in two parts. First, controlled stress rheometry was employed using an AR 500 rheometer with an environmental test chamber (ETC) accessory (*figure 4*). The testing was run using an oscillation procedure that provided information on the elastic modulus (G') of the grease as the temperature was increased. Second, pressure differential scanning calorimetry (PDSC) was run using the ASTM D5483 test method to look for any differences in oxidative stability of the two types of complex grease.

Procedure for High-Temperature Rheology Oscillation Test

- **Step 1:** Conditioning Step: 160°C (320°F), 1 minute equilibrium time
- **Step 2:** Temperature Ramp: 160-260°C (320-500°F), 5°C.per minute, 100 sample points, elastic modulus (G') is measured at 0.1% strain, 1 Hz.
- **Step 3:** Post-Experiment Step: cool to 80°C (176°F).

The elastic modulus (G') is measured at 0.1% strain, 1 Hz. It is also recommended that a fume extraction duct be used for tests over 200°C (392°F).

Figure 4. Rheometer equipped with environmental test chamber

Figure 5 shows a comparison of G' versus temperature for conventional greases A and C. The simple lithium soap (grease A) shows a decrease in consistency, exhibiting considerable softening when the temperature approaches its dropping point. The conventional azelaic acid complexed lithium soap (Grease C) shows a decrease in consistency as the temperature approaches the melt point of the simple soap constituent, however it regains its consistency as the complexing agent shows its influence.

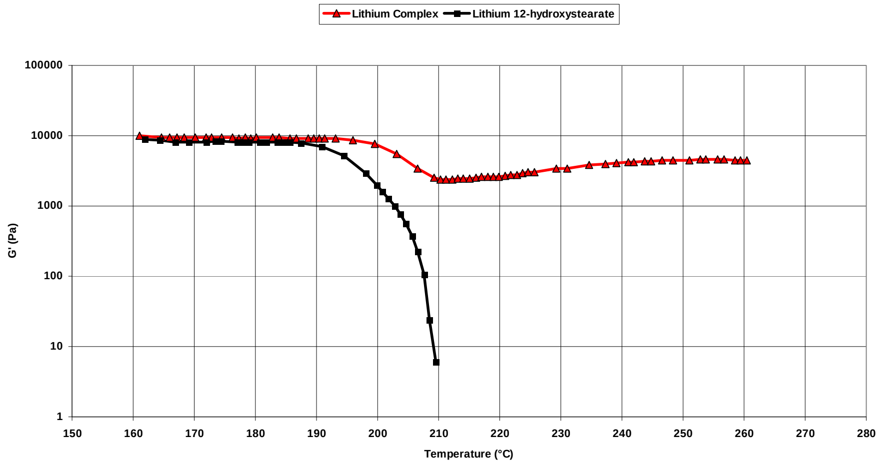

Figure 5. Rheology Plot: Grease A vs. Grease C

In contrast, the G' of the simple lithium grease additized with boron (grease B) ester does not decrease as much as the grease C when comparing *Figures 5 and 6*. Instead, it exhibits excellent stability of G' that has been shown to relate to consistency. This translates to a more uniform consistency over the entire temperature range.

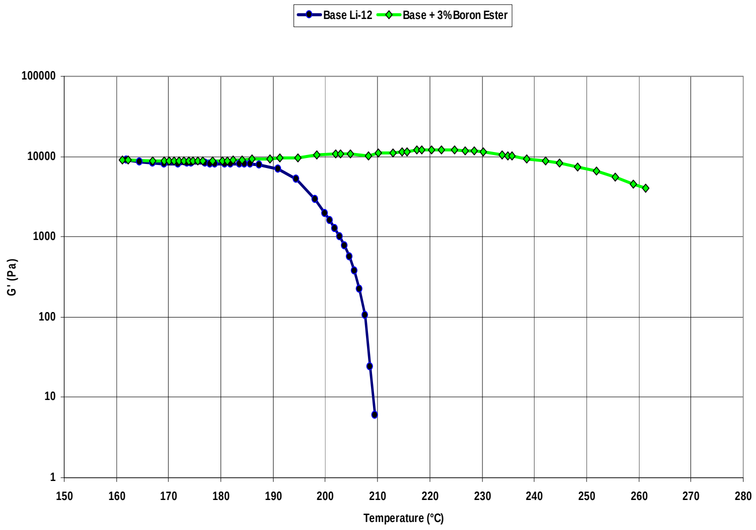

Figure 6. Rheology Plot: Grease A vs. Grease B

Figure 7 shows a photographic comparison of Grease A and Grease B. At the completion of the high temperature rheology tests, the appearance of the simple soap exhibits a complete separation of oil and soap. In contrast, the boron ester additized soap (Grease B) retains oil indicting that the grease structure is stable after exposure to high temperature.

Grease A

Grease B

Figure 7. Rheology Post Test photos

Oxidation resistance

Greases A, B and C were tested by ASTM D5483 run at 155°C (311°F), and the results plotted in *figure 8*. The simple lithium grease (Grease A) and the azelaic acid complexed grease (Grease C) both show similar oxidation induction times. The simple lithium grease additized with the boron ester (Grease B) shows improved oxidation resistance as exhibited by an increased oxidation induction time. The result was unexpected and may indicate a further advantage in using boron ester to make complex grease.

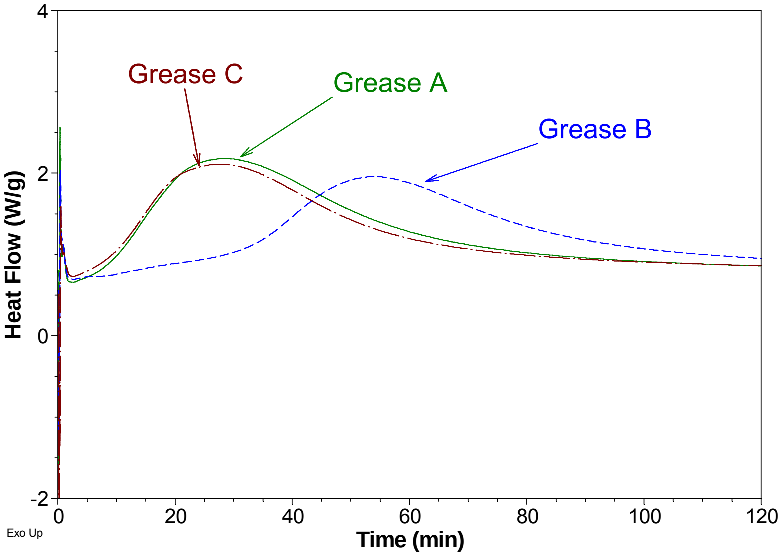

Figure 8. OIT Comparison

Based upon rheology high temperature test results demonstrating the unique heat resistance exhibited by test greases containing boron esters, a scanning electron microscopy comparison was made of the experimental greases *(figure 9)*. The goal of this study was to gain insight into any visual soap fiber differences that, if present, may help to explain the pronounced heat resistance observed in the boron ester containing simple soap greases. The preparation of the example greases prior to SEM included successive hexane washes to isolate the solids from the oil, air drying on glass cover slips, and finally, the application of a gold coating to render them conductive to the electron beam. Once prepared, the samples were run in a Carl Zeiss SMT EVO50 variable pressure scanning electron microscope, used in the high vacuum mode.

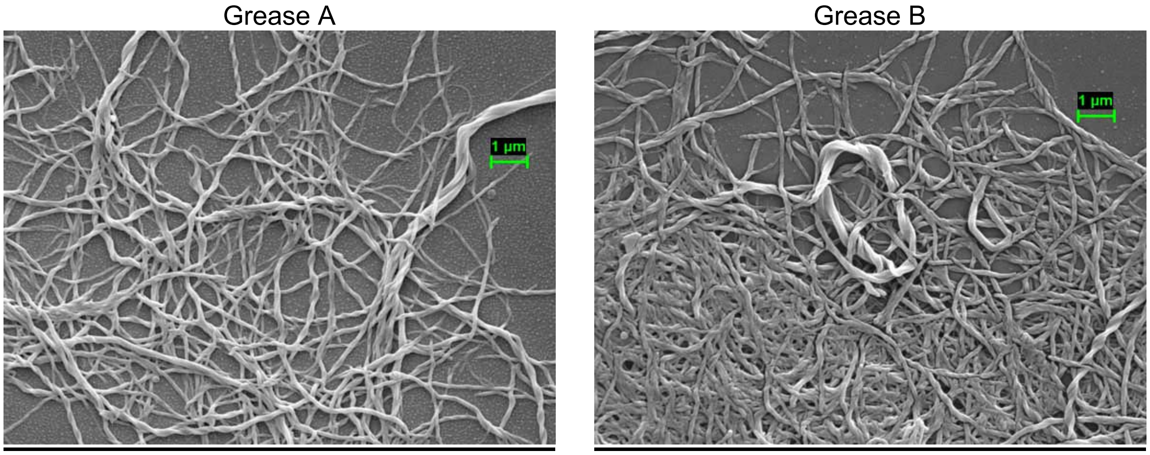

Figure 9. Scanning Electron Microscopy Photos

The appearance of the simple lithium soap was of rather long, mostly twisted fibers having a diameter of ≤100 nm. There were random fibers that were ~350 nm in diameter, but they may be composed of several ca. 100 nm fibers twisted around each other. In general, they were also rather free from each other, and there was not much in the way of remaining matrix material surrounding them.

By contrast, the boron ester additized lithium 12-hydroxystearate grease showed fibers that appeared to be somewhat thicker than the unadditized sample. The most striking difference was the high level of association between the fibers, producing a much more dense fiber network. Some agglomerates were observed to be present, which appeared to be made up of concentrations of intertwined fibers. This can most likely be explained by the higher degree of associative forces between the fibers resulting from the presence of the boron ester.

Low temperature testing

Low-temperature torque and pumpability are important properties of greases expected to be used at sub-ambient temperatures. Lithium soaps of 12-hydroxystearate have previously been shown to have some of the best low temperature properties when compared to other thickener types.[4] Typically, the low-temperature properties of lubricating greases are evaluated by standardized test methods, including ASTM D4693 low temperature torque, grease mobility and the Lincoln Ventmeter.

ASTM D4693

Significant for the design and specification of greases for low temperature service, the low temperature torque test measures the extent to which a grease sample retards rotation of a bearing assembly at the test temperature.

A comparison was made between the azelaic acid complexed lithium grease and the boron ester complexed simple lithium greases in the ASTM D4693 low temperature torque test. The results are plotted in *figure 10*. As shown in the graph, the boron ester complexed simple lithium soap grease shows improved low temperature properties as shown by the lower torque required to rotate the bearing at -40 ºC (-40°F).

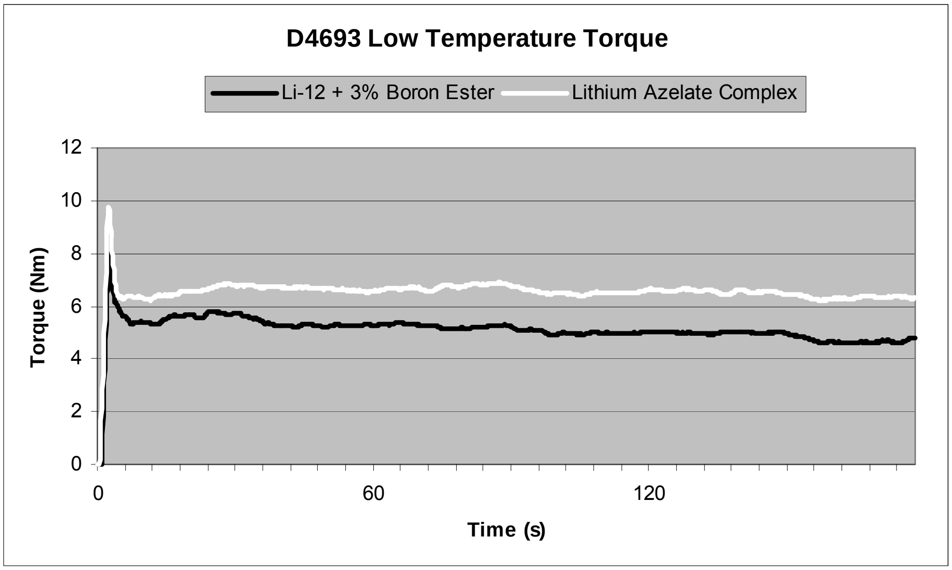

Figure 10. Torque Trace

Grease mobility

The US Steel grease mobility test apparatus determines the resistance of lubricating grease to flow under prescribed conditions. Mobility is measured in grams per second by pumping the sample through a standardized SOD pressure viscometer at controlled temperature and pressure. Grease mobility tests at low temperatures are used to predict pumpability characteristics and are commonly used to determine the suitability of greases for application in centralized lubrication dispensing systems.

A comparison was made between the azelaic acid complexed lithium grease and the boron ester complexed simple lithium greases in the LT37 grease mobility test. The results are plotted in *figure 11*. As shown in the graph, the boron ester complexed simple lithium soap grease shows improved low temperature flow.

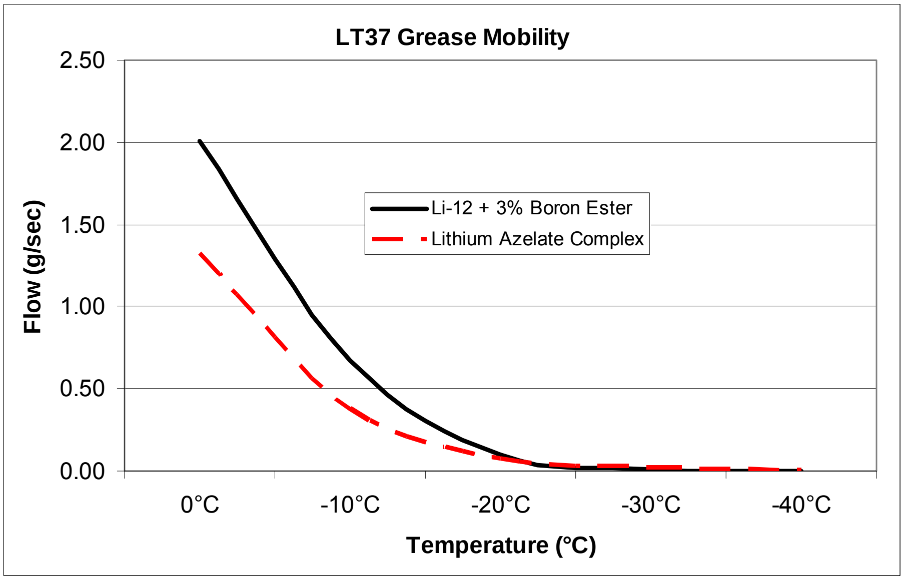

Figure 11. Grease mobility testing

Lincoln Ventmeter

The Lincoln Ventmeter is a tool commonly utilized to determine the suitability of grease for use in a centralized lubrication delivery system. Specifically, the Lincoln Ventmeter is a device that measures grease ventability. Ventability is defined as a measure of residual pressure where the grease ceases to flow through a length of coiled tubing after a pressure of 1800 psig (122.5 atm) is applied and then vented. The pressure that is measured in the coil drops from its initial pressure to a residual value after allowing the grease to flow out of the tube. By evaluating the residual pressure values, any two greases can be directly compared for flow properties as long as the reported values are taken at the same test temperature. When a direct comparison is made, the grease that has a lower residual pressure after the 30 second venting is concluded to have a reduced resistance to flow at the test temperature.

A direct comparison was made between the azelaic acid complexed lithium grease and the boron ester additized lithium 12-hydroxystearate. As *figure 12* shows, for each respective temperature, the boron ester complexed lithium grease demonstrated improved ventability.

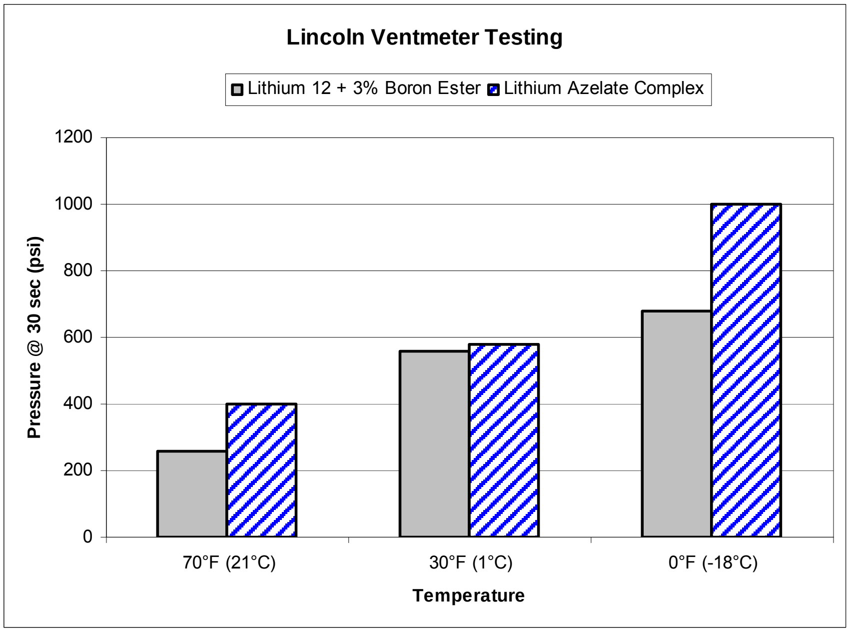

Figure 12. Lincoln Ventmeter Comparison

Finished grease performance

The purpose of this work has been to determine if the greases prepared using boron esters as the complexing agent actually produce grease that performs at the same level as lithium complex greases formulated with conventional complexing acids, such as azelaic acid. To make this determination, a comparison was initiated to subject experimental greases utilizing both types of complexing agent in a variety of tests in an end use application that has typically been attainable only by true high temperature grease. The ASTM D4950 category GC testing was selected for the high temperature requirements in ASTM D2265 dropping point, ASTM D4290 bearing leakage and ASTM D3527 wheel bearing life testing typically requiring the heat resistance of complex greases.

Evaluation by ASTM D4950

Fully formulated lithium-based greases were compared to the standard classification for automotive service greases, ASTM D4950 [5] The NLGI classification system for automotive service greases is the most common specification in the United States, and the GC classification for high-temperature, severe-duty wheel bearing service is usually only achieved by complex greases. As previously discussed, the three main tests where simple lithium soaps would not typically pass include dropping point, bearing leakage and wheel bearing life. The results of the evaluation as shown in *table 3* demonstrate that boron ester complexed greases offer comparable performance to azelaic acid complexed lithium greases and are able to meet the complete requirements of ASTM D4950 performance category GC and LB, offering true multipurpose performance.

ASTM Standard	Description	GC requirement	LB requirement	Boron Ester LiX + 4% Additive Pkg	Azelaic Acid LiX + 4% Additive Pkg
D217	Consistency, worked penetration, mm/10	220-340	220-340	254/254	293/293
D2265	Dropping point, °C, min	220°C	150°C	311°C	285°C
D4693	Low temperature performance, torque at -40°C, Nm, max	15.5	15.5	5.47 Nm	6.86 Nm
D4170	Fretting protection, mass loss, mg, max	--	10	6.85	7.15
D1264	Water resistance at 80°C, %, max	15%	--	4.0%	5.1%
D1742	Oil separation, mass %, max	6%	10%	2.1	1.9
D1743	Rust protection, rating, max	Pass	Pass	Pass	Pass
D2266	Wear protection, scar diameter, mm, max	0.9	0.6	0.53	0.55
D3527	High temperature life, hours, min	80	--	80	164
D4289	Elastomer SAE AMS 3217/2B compatibility [NBR]				
	Volume change, %	-5 to +30%		9.8	9.7
	Hardness change, durometer-A points	-15 to +2		-1	-2
D4289	Elastomer SAE AMS 3217/3B compatibility [CR]				
	Volume change, %		0 to 40%	15	12.9
	Hardness change, durometer-A points		-15 to 0	-10	-9
D4290	Leakage tendencies, g, max	10		3.64	2.21
D2596	EP Performance:				
	Weld point, kgf, min	200	200	315	315
	LWI, kgf, min	30	30	49.8	47.5

Table 3. Comparison against ASTM D4950 Specification

Conclusions

The use of boron esters as the method of complexing lithium 12-hydroxystearate grease provides a simpler, faster, more economical method of producing lithium complex greases. Lithium complex greases produced by adding boron esters as the complexing agent have shorter batch cycle times versus greases of conventional manufacture, which results in energy savings and improved production efficiency.

Experimental work conducted to compare the operating temperature range of grease additized with boron ester to that of the conventional complex grease showed similar performance in high temperature rheology testing. The greases containing complexing agents were much more resistant to consistency change as temperatures were increased. Additionally, lithium complex greases prepared from boron esters were shown to offer expanded low-temperature service range as demonstrated in the ASTM D4693 low temperature torque, LT37 grease mobility and Lincoln Ventmeter tests.

Boron esters were shown to contribute additional beneficial properties to the finished grease, including improving oxidation induction times (OIT) as measured by the ASTM D5483 PDSC test.

Finally, when fully formulated greases containing performance enhancing additive packages were compared against the ASTM D4950 standard classification for automotive service grease, both experimental greases showed similar performance and were able to meet the full requirements of the most severe service classifications for use in wheel bearing and chassis service, GC and LB.

Acknowledgements

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