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In This Issue

On Our Cover

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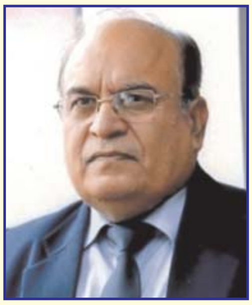
Add IOCL

From President's Desk



52nd issue of GREASETECH INDIA (Vol. XIII, No.4) is in your hands. It is an amazing feat achieved by NLGI-India Chapter. For this I thank the Editorial board and advertisement sponsors. NLGI – India chapter remains the single most interactive platform for discussions on new developments in Indian grease industry and related areas of equipment manufacturing, analytical techniques, additives development, etc.

This issue includes technical papers on biodegradable grease, development of Inorganic Fullerene like WS_2, EP/AW additives and development of synthetic grease for multipurpose applications. Research article on analytical techniques to study cleanliness properties of the commercially available greases is also included in this issue.

Any suggestion for betterment of GREASETECH INDIA technically or appearance wise is most welcome. I earnestly request members and readers to patronize GREASETECH INDIA and give suggestions to increase its circulation.

Dr. R. K. Malhotra
President,
NLGI-India Chapter

Driveline Lubricants : Off Highway — Indian OEMs Challenges

A. Surender

Preface:

The paper mainly deals in Driveline fluids trends at Global level and trends taking place at Indian OEMs in particular off highway application. Specific emphasis is on customer demand and in turn influence on OEM in selection of suitable Driveline Technology. This in turn firms up the supply Standards of Driveline lubricants

Background:

India is fast becoming the global hub for Automotive Industry. The world's best OEMs are operating in great way at Indian Shores. European auto Industry show moderate growth, China & India continue to show strong growth. U.S. and Japan has shown significant recovery.

The various segments of Indian Automotive Industry are booming e.g. Two wheeler @ 26 %, passenger cars @ 26%, commercial vehicles @ 38 %. The commercial vehicle industry is expected to grow by 23-25 % in 2010-11 on the back of sustained growth in economic activity and rise in consumption expenditure. The LCV good segment is expected to grow strongly by 24-26 % on new models, growth in organised detail and increase in consumption expenditure in urban areas. The expected investment in Construction and Mining Industry would touch US$ 4 Bn.

Growth – imperatives:

Growth in LCV is sustained with hub and spoke model proliferation. Tippers have shown significant growth due to increased infrastructure activity. OEMs are constantly driving to reduce the cost of ownership to sustain the buoyant growth trajectories. Launch of newer models at competitive pricing to enable the Market expansion.

Driveline by application:

DCT (Double clutch transmission), CVT (Continuously variable Transmission) to take the space of Automatic Transmissions in Passenger car and commercial vehicle Industry. The selection of Technology is governed by advantages in terms of fuel economy, ride comfort compared against the disadvantages of limited experience in Indian conditions coupled with additional facilities and investment. Universal Tractor and Transmission oil (UTTOs) and TO-4 fluids will cut larger pie of Driveline application in Tractor and off-highway segment by 2012.

Drivers – OEMs way forward

The broad trends for OEMs way forward include emissions, Fuel economy, use of alternate fuels, Hybrid Technology, Globalisation Etc. Each of the segments has its own priorities such as Passenger car is moving more towards higher Power, increased percentage of DI diesel engines and adoption of tarns axles for better drive. SUV segment is the most severe application and is a challenge for development of Transmission fluid. Volume load ratios are going up, Power density is high and the suitability/ selection of fluid is a big task for OEM. Transmissions are one of the key attributes in Marketing of its products by OEMs.

Types of Transmission

DCTs are similar to Manual Transmissions, Paired helical gear sets on twin input shafts to provide the range of gear ratios. CVT uses belt or chain link two pulleys, to manage the gear ratio in response to driving conditions. Nissan is one of the big users of CVT. DCT and CVT are expected to garner 15% of Market share by 2020 in the World.

Drivelinefluid – demands at Indian OEMs:

The Driveline Segment of Indian Automotive Industry is poised for a big change as the Industry is concentrating to yield the much talked about subject of fuel economy from the Drive Line Technology.

The Retail Market for Drive Line Lubricants has gradually shifted to multi grades and today SAE 80 recommended segment has shifted to 80 W 90 and for transmission applications and SAE 40 to 85 W 140 for axle applications.

Extended drain Transmissions by OEMs such as ZF, FORD etc. demand for fluids made from Group II/ III and PAO coupled with proven additive Technology to rely on durability, no shudder and no squawk. Fuel economy is calling for thinner fluids with high shear stability.

Synchronising materials in the transmission calls for different additive technology and to facilitate the customer for a smooth drive. Passenger car OEMs such as Tata Motors, Suzuki, FIAT etc. have started recommending synthetic fluids for transmission and transaxle applications for drain intervals of one lakh kilometres. To-days design of driveline fluids are primarily concentrating on synch compatibility, balancing of frictional characteristics and long oil drain intervals.

Indian OEMs such as Tata Motors, M&M are constrained to increase oil drain intervals as the other aggregates durability is to be considered and synchronize during the downtime of equipment.

In to-days scenario, Off-highway Vehicles have started outsourcing the majority of transmission and axle requirements from the biggest OEMs such as ZF, Carraro Transmissions, Allision, Eaton Corporation etc. These OEMs have very specific requirements on driveline fluids. Specific programs run by the OEMs in order to bring awareness to customers using the right fluids for longevity of the equipment and sustain warrantee claims.

The space of Viscometrics in Driveline Fluids in India have increased from earlier one or two to Viscometrics such as 75W85, 75W90, 75W80, 80W90and 85W140. The increased use of synch materials is calling for driveline fluids having control on sulphur phosphorus chemistry.

Scenarios at certain OEMs

Tata Motors Ltd

India's largest OEM in CV Industry with market share over 62% has its new recommended Driveline fluids include part synthetic base blend stocks for Transmission application in World Truck.

FIAT powertrains are calling for fluids of 75W80 with elaborate OE specific performance specific requirements

ATF fluids have very unique requirements

Fluid/ Feature	Type F	TO-4	THF	Dexron® II	Mercon®	Dexron® III	Mercon® V
Friction	Grabby	Grabby	Most slipper	Slippery	Slippery	Slippery	Slippery
EP/AW	Good	Very Good	Excellent	Good	Good	Good	Very good
Oxidation	Fair	Good	Good	Good	Very Good	Very Good	Very Good
Low temp	Good	Poor	Good	Good	Very Good	Very Good	Excellent
Water Sensitivity	Not tested	Wet antifoam	Very Good	Not tested	Not tested	Not tested	Not tested
Color	Red	—	—	Red	Red	Red	Red
Viscosity	7-8 10W-20	10-11 SAE 30	9.5-10.5 10W-30	7.0-7.5 10W-20	7.0-7.5 0W-20	7.0-7.5 0W-20	7.0-7.5 0W-20

Recommends OE and Service fill same product line

Maruti Suzuki Ltd

The largest Passenger car OEM is soon to touch more than million cars at a single Country. The recommendation is for API GL-4 SAE 80W90 would be switching over to 75W90.

ZF Transmissions:

ZF has commissioned its new facility for CV and off highway Transmissions at Pune. There is an increased presence at OEMs such as Tata Motors Ltd, AMW, and MANFORCE .etc. Its presence is increasing at Off-highway OEMs.

Arvin Meritor and Eaton Corporation, having the best of World's Technologies are having its equipment at Ashok Leyland, Kamaz Vectra etc.

Komatsu:

Commissioned its Plant at Chennai for manufacture of 100T dump trucks.

Off- Highway segment

Tractor Industry

India is the World's third largest Tractor Market. M&M, TAFE, TMTL, John Deere, ITL are major OEMs dominating the Market. The Market with annual sales of 0.4 Million Tractors is moving towards Wet brake Technology due to increased HP and convenience to the customer. The technology is calling for fluids more on J20 C based plus very OEM specific compatible to brake-shoe material.

The current recommendation of fluid for manual transmission is SAE 80, API GL-4 at majority of the OEMs. John Deere has very specific formulated product meeting J 20C for wet brake application. Wet Brake fluids for each of the OEM such as ITL, Eicher Tractors, and Escorts are conforming to J20 C but the fluid characteristic depends upon metallurgy of material employed in Brake shoe. Brakes India is the major supplier of components at these OEMs.

Construction & Earth Moving equipment:

The estimated total Lube potential at off highway segment is 80000 KL and the requirement of transmission fluids is very complex. BEML, Caterpillar, JCB, Telcon, L&T Komatsu etc. are some of the major OEMs in the segment. New OEMs such as Komatsu, Kobelco, Lieugoung. Etc. are bringing in new requirements in Driveline fluid. Backhoes manufactured by Escorts Construction equipment have common AT fluid for Torque convertor and Transmissions. Limited slip application in this segment requires SAE 90 and SAE 80W90 fluids conforming to ZF standards. TO-4 fluids are the demand requirements by BEML and Komatsu in 100T Dumpers. These fluids are recommended for Transfer case, Power take off unit, differential case and Final Drive.

The Power density has increased by 20% in the last one decade in this segment. These OEMs are increasingly recommending for use of genuine oil fluids for After Market requirement. Plant fill and After Market requirement have common oil performance standards.

Conclusion:

The trends taking place in Driveline Lubricants for off highway application is greatly influenced by new Technologies based equipment brought in by Indian OEMs as well as International OEMs. Bigger equipment demanding fluids having better EP and antiwear characteristics. Ownership cost is also adding new dimension in development of high performance fluids.

An Eco Friendly Grease

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Abstract

Mineral oil based greases are widely used in many applications including automotive, industrial and defence. Despite sophisticated refining processes, mineral oils are still complex mixtures of compounds containing nitrogen, sulphur and oxygen with some inorganic impurities and so they are toxic to some extent and exhibit low biodegradability. Mineral oil based greases used in farming sector are likely to go into soil and may migrate to flora & fauna resulting in contamination of ground water posing health hazard. Advances in modern technology have geared up the development of synthetic lubricating greases that out perform at the temperature ranges beyond the capability of mineral oil based greases with additional advantages of low volatility, thermal stability, fill for life concept, biodegradability, etc.

This paper deals with the development of a semi-synthetic, eco friendly and biodegradable grease fortified with EP, Anti-wear and rust preventive additives. The grease has over 70% biodegradability and can be widely used in agri-machinery (Trolley, Rotary Tiller, Harvester etc.),, forestry, chain saw, lawn mowers, wood cutters, tractor, water pump, paper industries, etc.

Introduction

More than 10 Lakh ton lubricating grease is produced worldwide to meet the demand of Industry [1]. Use of lubricating greases in total-loss applications such as lubrication of open gears in cement plant, lubrication of railway wagon bearings, forestry equipments etc. is a major concern as the direct loss of lubricant to environment occurs, which adversely affects ecosystem for longer periods. To highlight the gravity of the issue, data collected on estimated release and re-collection of used oil in compiled in Table 1 & 2 respectively [2]. Therefore development and application of biodegradable lubricants has become important to protect eco-system. The biodegradability of finished lubricating grease is sum of the biodegradability of its constituents i.e. base oils, thickener and additives [3]. Typical biodegradability range of various types of base oils [4] is given in Figure 1.

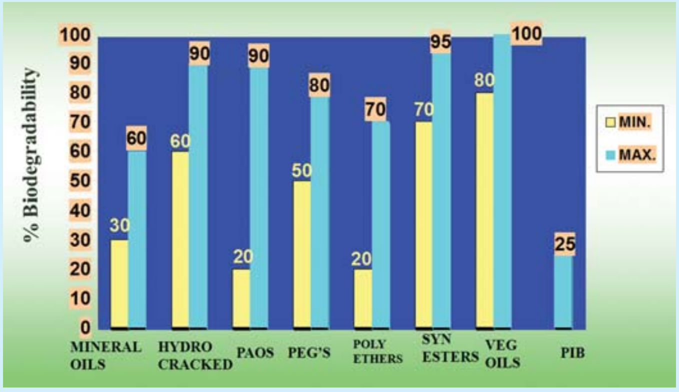

Fig. 1 : Biodegradability range of various types of base oils

Soap based thickeners (Lithium, Sodium, Calcium, Aluminum etc.) have reasonably good biodegradability. Biodegradability range for some common types of additives [4] is given in Table 3.

Table 1 : Reported Release of Lubricants to Environment

Country	Lubricants type	Estimated Release (kT/yr)
Germany	Engine Oil, Chain saw oil & Concrete mould release	44
Canada	Chain saw, hydraulic, motor & gear oils	19
Switzerland	Total loss	4
Australia	All type	80

Table 2 : Used Oil Collected from Various Countries (in MT)

Country	Oil Consumption / Annum	Used oil Collected	%
USA	8467000.0	4233000.0	50.0
EEC	4746000.0	1748000.0	37.0
Japan	2101000.0	882000.0	42.0
Germany	817000.0	473000.0	58.0
Italy	600000.0	180000.0	30.0
U.K.	800000.0	350000.0	44.0
France	890000.0	236000.0	26.0
Spain	570000.0	130000.0	23.0

No such compiled information is available for India.

Table 3 : Biodegradability range for common types of additives

S. No.	Additives	Biodegradability (%)
1	Corrosion inhibitors	60 - 90
2	Anti-wear	~ 90
3	Extreme Pressure	~ 90
4	Pour Point Depressant	~ 85

The development of reasonably biodegradable lubricating grease is based on the selection of correct raw materials as discussed above. Global research efforts on the development of biodegradable lubricants started long back and are well documented (Table 4). A variety of both EP and non-EP biodegradable lubricating greases are available from OMC s. Though volume of literature is available globally on biodegradable lubricants including lubricating greases [5]. Very little has been published on biodegradable lubricating greases from the Indian subcontinent [6]. Therefore, IOCL, R&D undertook a research programme to develop and commercialize a biodegradable lubricating grease suitable for variety of industrial applications.

Table 4 : Timeline of development of eco-friendly lubricants

S. No	LANDMARK	YEAR
1.	Biodegradable two stroke oil based on synthetic ester	1975
2.	Development of Biodegradability test, now CEC L-33A-94	1976-79
3.	Biodegradable Hydraulic Fluids and Chain Saw Lubricants	1985
4.	German “Blue Angel” specification for chain Saw Lubricants	1989
5.	Biodegradable Greases	1990
6.	Blue Angel for mold oils, Greases and other lubricants	1991
7.	Biodegradable Engine oils Tractor Transmission Fluids	1993
8.	DIN specification for Biodegradable Hydraulic oils	1994-95
9.	Standard Specification for Biodegradable Fire Resistant Hydraulic Fluids- ASTM D 7044	2004
10.	Standard Test Method for Determining Aerobic Aquatic Biodegradation of Lubricants or Their Components- ASTM D 5864	2005
11.	European Eco-label	2005

Experimental

Vegetable oils are preferred choice of base oils while formulating biodegradable lubricating greases. Products based on synthetic esters and glycols are also available in the market. In present research work, VG 160 base stock was based on the combination of mineral and synthetic oils. Lithium soap was used to thicken oil. Grease was prepared by batch process. Majour issues those were tackled during research were – compounding of base oils, manufacturing of grease, oxidation stability, low and high temperature issues etc. Standard ASTM or IP test methods were followed while testing of lubricating grease. A large number of experimental batches were prepared using various combinations of mineral and synthetic base oils. Thickener concentration was optimized to obtain an NLGI grade 2 grease. Carefully chosen additives were dosed to enhance performance of the base grease. A short-listed grease batch was subjected to rig evaluation. Following criteria was established for product to qualify for an "Eco-friendly lubricating grease" - No Lead, Barium, halogenated compound, nitrites and with a biodegradability of more than 70% [CEC-L-33-A-94 test method].

Results and Discussions

Typical test data of the developed eco-friendly lubricating grease is compiled in Table -5. With more than 70% biodegradability the grease suits well for total loss applications in industries such as agriculture, paper, steel, forestry etc. The grease was mechanically stable as evident from the values of P0

Table 5 : Typical properties of developed "Eco-friendly grease"

S. No.	Characteristics	Eco Friendly Grease	Test Method
1	Appearance	Homogenous	Visual
2	Colour	Light Brown	Visual
3	Texture	Smooth	Visual
4	Consistency of worked grease at 25+0.5°C		D-217
	a) 60 double strokes	282	
	b) 100000 double strokes, Change in units	+20	
5	Dropping point,°C	190	D-566
6	Oil extracted from grease a) KV at 40°C	160	D-445
7	Water Wash Out, 80°C, 1hr, %	8.0	D-1264
8	Biodegradability, %	>70	CEC-L-33-A-94
9	Evaporation loss, %wt	2.0	D-972

and P60 penetration value difference of +20 and roll stability penetration value change of ~10%. Dropping point ~190 °C, oxidation stability and evaporation loss data indicate that the developed grease is suitable for most of the industrial applications which use lithium base grease. The grease has good protection to metal corrosion. The grease has been fortified with suitable additives to impart EP characteristics to it. A unique feature of this grease is low water wash out. This makes it suitable for the application to machine parts prone to water ingress or flow.

Conclusions

An Eco-friendly NLGI grade 2, Lithium based lubricating grease was successfully developed. This is a cost-effective and eco-friendly having wide scope of applications in forestry, agriculture, paper, miming, earth moving industries etc. Biodegradation of developed grease is more than 70% making it suitable for the applications which are prone to land and water contamination. Developed lithium based grease has excellent water wash out characteristics. The developed grease is suitable candidate for the applications where the lubricant has a chance to come in contact with soil and aquatics.

Acknowledgement

Authors are thankful to the management of IOCL, R&D Centre, Faridabad, India for granting permission to submit and present paper in13th Lubricating Grease Conference, Ooty, February 3 – 5, 2011.

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Grease Composition for Steel Plant Application

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ABSTRACT

In this paper we are reporting the grease composition required for slip seal lubrication of the rotary kilns and coolers. The rotary kilns and coolers are used as important and essential equipment in sponge iron industries. The developed grease composition incorporates the calcium sulphonate complex base thickener, mineral oil and desired additives. This grease composition was prepared in-situ by reacting calcium sulphonate and other various reactants in the presence of mineral oil and desired additives. The prepared grease composition was evaluated for water resistant, evaporation loss, oxidation stability, life performance, extreme pressure and anti wear properties. The prepared grease composition possesses very high dropping point, outstanding oxidation stability, excellent water resistance properties, shear stability, anti wear and extreme pressure properties.

The developed grease composition showed promising results during the field trails and performed well as compared to the grease currently in use for lubrication of slip seals of rotary kilns and coolers of Sponge iron industry. The trial results showed a significant reduction in grease consumption for the application along with effective sealing between slip seals and reduction in power consumption as compared to grease earlier in use.

KEY WORDS: Slip Seal, Rotary Kilns and Coolers, Calcium Sulphonate Grease, High Temperature Grease.

INTRODUCTION

The calcium sulphonate greases are used for high temperature applications. The calcium sulphonate greases are frequently used in steel plant applications and also for slip seal applications. Theses greases have better performance as well as life than lithium complex greases. These are used for continuous casting applications in the steel plants. The greases are prepared in-situ by reacting calcium sulphonate and other various reactants in the presence of synthetic base fluid and mineral oil and desired additives. The complexing agent improves the thermal stability, mechanical stability, dropping point and water resistance characteristics of the grease. The end result is grease that can be used for a longer life over a wider temperature range than standard lithium complex greases (1-3).

The prepared grease composition was evaluated for water resistant, evaporation loss, oxidation stability, life performance, extreme pressure and anti wear properties.

The investigations reported in this paper are aimed at:

i) the preparation of calcium sulphonate grease for slip seal applications,

ii) the performance evaluation of the prepared calcium sulphonate greases by shear & oxidation stability, water resistance as well as rust tests,

iii) the high performance evaluation with respect to high load carrying capacity was evaluated by four-ball and Timken tests, and

iv) prepared grease was also subjected to field trials for lubricating the slip seals of rotary kilns and coolers of sponge iron industry

EXPERIMENTAL

1. Greases Used for Trials

a) **Calcium Sulphonate Complex Grease** - The calcium sulphonate complex grease was prepared in-situ by reacting calcium sulphonate and other various reactants in the presence of mineral oil and desired additives to achieve the properties required for lubrication of slip seals used in hostile environments of rotary kilns and coolers of the sponge iron industries. The prepared grease posses higher load bearing ability, good oxidation resistance, excellent water and corrosion resistance and excellent ability to withstand high temperature.

b) **Reference Grease** – The grease currently in use for lubrication of slip seal was taken as reference grease.

2. Apparatus

a) Extreme Pressure and Anti-wear Properties The test values are reported in **Table-1**.

i) **Extreme Pressure by Four-ball machine** - The test was conducted as per ASTM D 2596 (4). The duration of the test was 10 seconds. A series of tests were performed with the prepared greases until the welding point was reached.

ii) **Extreme Pressure by Timken machine** – The Timken OK load test was carried out by following ASTM D 2509 test (4). The test duration was ten minutes at each load unless the scoring occurs. The maximum load that does not produce scoring and the minimum load that produce scoring were reported as Timken OK loads and scores values. Timken machine produces condition similar to those of industrial gears and some slow speed automotive axles.

iii) **Anti-wear by Four-ball machine** - The anti-wear test was also conducted in a 'Four-ball' machine as per ASTM D 2266 (4).

b) Roll Stability test – This test was carried out using ASTM D 1831 test method (4).

c) Corrosion preventing test - The test values are reported in Table-1.

i) **Emcor rust test** - The Emcor rust test was used to assess the ability of greases to prevent rusting in following bearing operated in presence of distilled water. It was performed as per ASTM D 6138 method in SKF Emcor test rig (4). As per ASTM test method, the prepared greases were tested in ball bearing running at 80+5 rpm under no applied load in the presence of distilled water.

ii) **Rust preventing test** - In this test the lubricated tapered roller bearings were tested at 52+1oC for 48 hrs fewer than 100% relative humidity as per ASTM D 1743 test method (4).

d) Oxidation stability test - It was carried out using oxidation bomb method, ASTM D 942 (4). In this method the sample of the prepared greases were oxidized in a bomb heated up to 99+1oC and filled with oxygen at 110 psi. Pressure was observed and recorded at stated intervals. The degree of oxidation after 100 hrs was determined by corresponding decrease in oxygen pressure.

e) Water washout test - It was carried out at 79+1.7°C as per ASTM D 1264 test method (4).

3. Methodology for Trials

Slip seals used in rotary kilns and coolers of the sponge iron industries works in hostile environments. The following monitoring techniques were used to monitor the performance of the prepared grease for slip seals used in rotary kilns and coolers.

1) Monitoring techniques for checking grease suitability for the application:

i) Visual Inspection

(1) **Check for sealing:** The leakage of gas, flame and coal dust has increased or decreased. This kind of visual inspection helps to analyze the sealing characteristics of the grease

(2) **Check for grease leakage**: High temperature conditions - Check the grease leakage tendency of the grease

(3) Check for grease consumption

(4) Checking pumpability of the grease through the pump and the lines

ii) Monitoring through System:

(1) Monitoring the Ampere of the motor rotating the kiln

(2) Monitoring the Amperes of the motor rotating the cooler

(3) Monitoring temperature variations at slip seals of kilns and coolers

Table 1 : Typical test results for the prepared grease (NLGI 2 Grade)

Test	Ca Sulphonate Complex Grease	Reference Grease	Methods
NLGI Grade	2	2	NLGI
Penetration, 60X	286	288	ASTM D 217
Drop point, °C	>340	260	ASTM D 2265
Oil separation, 100°C, 30h, %	0.8	1.9	IS1448 P:89
Oil separation storage, %	Nil	Nil	ASTM D 1742
Deleterious particles, number of scratches	Nil	Nil	ASTM D 1404
Oxidation stability, 99+1°C, 100 h, pressure drop in psi	0.5	3	ASTM D 942
Water washout, 80°C, % loss	1.0	2.4	ASTM D 1264
Rust test	1	1	ASTM D 1743
Emcor rust test	0,0	0,0	IP 220
Copper corrosion, 100°C, 24h	Pass	Pass	ASTM D 4048
Wear test, 1200 rpm, 40kg, 75°C, mm	0.45	0.55	ASTM D 2266
Four ball weld load, kg	400	315	IP 239
Timken OK load, lbs	60	50	ASTM 2509

RESULTS AND DISCUSSION

The Table -1 records the various parameters obtained with laboratory performance test rigs as per standard test methods. This table includes the test results obtained on developed grease and reference grease. The tested grease shows higher drop point, good sheer stability as well as water-resistant properties. The prepared calcium sulphonate grease also showed better oxidation stability and higher values of four-ball weld loads of 400 kgf and Timken OK loads of 60 lbs**.**

Trial Results:

The prepare grease has shown better results for the Slip Seal applications in rotary kilns and coolers of Sponge iron industry also shown a significant reduction in grease consumption for the application as compared to reference grease.

The field trials results reveal that:

1) The developed grease showed effective sealing between the slip seals of rotary kilns and coolers, we interpret that the grease is capable of lubricating the slip seals under hostile conditions
2) The grease did not thin out during the operation and hence found suitable for high load and high temperature operations
3) The grease was found to be compatible with the automatic grease lubricating system and was easily pumpable through the system and pipelines
4) The prepared grease worked well in dusty environment
5) Grease consumption was found reduced as compared to the reference grease
6) Drop in ampere drawn by motor during trials by 3-4 A.

Thus on the basis of trial results, we can conclude, that the prepared grease is very effective in sealing the slip seals of rotary kilns and coolers and the consumption of the prepared grease was also reduced as compared to reference grease.

CONCLUSION

The prepared high temperature calcium sulphonate complex grease was found to be more effective in reducing friction and wear at sliding surfaces and increasing the load carrying capacities as compared to reference Grease. The prepared grease also showed lower values of wear-scar diameter and higher values of weld loads in the four-ball and Timken tests. The prepared grease also possesses excellent water resistant shear and oxidation stability and anti-rust properties.

The prepared grease composition showed promising results and performed well as compared to the grease currently in use for lubrication of slip seals of rotary kilns and coolers. The trial results also showed a significant reduction in grease consumption for the application and reduction in power consumption as compared to grease earlier in use.

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Development of Greases with Extended Grease and Bearing Life Using Pressure Differential Scanning Calorimetry and Wheel Bearing Life Testing

William Ward Jr. CLGS, and
Gareth Fish, Ph.D. CLGS

Numerous factors impact the design of greases formulated to provide extended grease and bearing life. Not only is the choice of base oil and thickener important, but antagonistic relationships between the components in grease may occur when formulating high performance greases. For long bearing life greases in particular, balancing oxidation, load carrying capacity and wear is a key requirement. To provide an acceptable package in global markets, environmental considerations must also be given to the grease components.

In North America, the ASTM D3527 wheel bearing life test represents a measure of performance for automotive service grease under GB or GC requirements of the ASTM D4950 standard classification. The test is considered a bearing oxidation test for grease. In Europe the FAG FE9 test is widely used to define bearing oxidation life. An alternative, non-bearing method of measuring the oxidative stability characteristics of grease is pressure differential scanning calorimetry (PDSC) as measured by the ASTM D5483 test. Some published work has shown a correlation between the two methods among different types of grease.

This paper describes the use of D5483 and the D3527 tests as a guide to formulate prototype extended bearing life greases and to understand the contribution of different formulation factors on oxidation, including base grease and additive. The performance of selected greases was then verified by using standard FAG FE 8 (DIN 51819-2) and FE9 (DIN 51821-2) testing.

Introduction

Lubricating grease represents a critical design component in the lubrication of rolling element bearings for both industrial and automotive applications. The grease functions by providing a film of lubricant to separate moving surfaces in a bearing that consists of rolling elements and a bearing raceway. Several types of bearings defined by the geometry of the rolling elements include radial, cylindrical, spherical, tapered and needle bearings as illustrated in Figure 1. Extending the life of the rolling element bearing application requires formulating the grease to extend its useful life and demonstrating that the application life is increased.

Grease Bearing Application and Lubrication

For industrial applications such as steel mills, mining, construction and transportation, the widest use of grease is in lubricated bearings. In many industrial applications, the bearing is fitted with a lubricator or zerk fitting and the maintenance schedule dictates when the bearing is to be re-lubricated and how much grease needs to be added to the bearing. The quantity of grease to be used is a function of the size and type

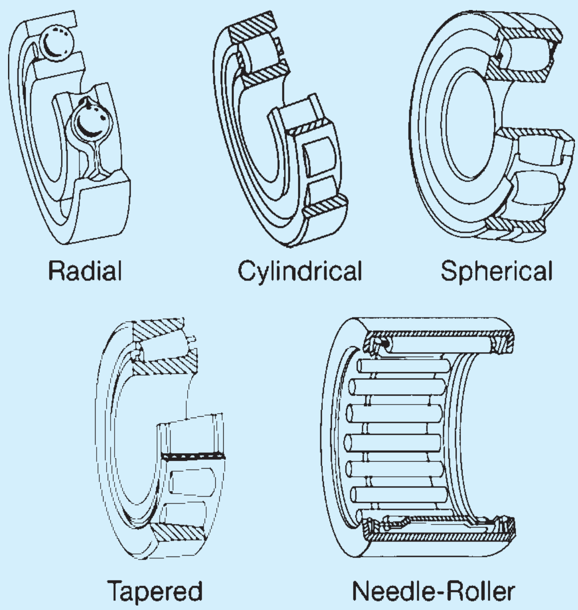

Fig. 1 : Types of Rolling Element Bearings

of the bearing and the loading, the speed and the operating environmental conditions define the re-lubrication interval. Greases operating at temperatures above the standard temperature of 70°C or in wet or dusty conditions need to be re-lubricated more frequently than those operating at normal temperature in a clean dry atmosphere.

For automotive applications, the emphasis for wheel bearings has changed from replacement at periodic intervals of 10,000 to 20,000 miles or so in the 1960's to sealed- for-life applications in which the grease and the bearing should perform for the life of the vehicle. This has occurred as the vehicle manufacturers have extended the warranty period from typically 3 years and 30,000 miles to 10 years and 100,000 miles. Enhanced grease performance is necessary to satisfy the increased demand placed upon the sealed- for-life bearing components.

The role of the grease in a rolling element bearing is to help maintain anti-friction bearing characteristics, seal the bearing, and provide performance. Rolling resistance due to deformation of the rolling elements and raceway under load is reduced by separation of the mating surfaces and sliding friction occurring between rolling elements, raceways and cage is minimized. In industrial applications, the performance of the sealing is typically much less than that seen in sealed-for-life automotive bearings but is important to prevent contaminant ingress. In many cases, the grease cannot totally prevent water from entering into the bearing and so the grease also needs to protect the mating surfaces from water induced corrosion through additives. Heavy loading and high temperatures necessitate other performance provided by additives in the grease including prevention of wear and scuffing through antiwear (AW) and extreme pressure (EP) additives and reduction of oxidation with antioxidants (AO).

Grease lubrication of bearings has been widely discussed. Cann (1) and Cann and Hurley (2) showed that the thickener played a full part in the lubrication of bearings by grease through work funded by the NLGI in the 1990's. It was demonstrated that the thickener helps the grease to form thicker lubricant films than the base oil alone when a plentiful supply of grease is present using a thin film optical elastohydrodynamic (EHD) lubrication test machine. Under parched (as defined by Cann(3)) or starved lubrication where grease supply or retention is reduced, the thickener plays an even more significant role in preventing metal to metal contact of the ball on the raceways and contributing greatly to boundary lubrication. Figure 2 shows a depiction of the film generated at the leading edge of rolling element motion.

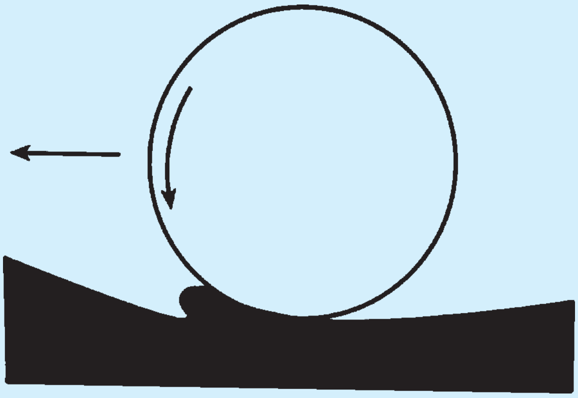

Fig. : 2. Lubrication of a Rolling Element

Grease Useful Life

One of the greatest factors related to the useful life of grease is oxidation resistance, although bearing tests that measure wear by grease lubrication are also important and available in Europe. Oxidation resistance of the grease can be determined through laboratory bench tests and bearing oxidation tests. Pressure differential scanning calorimetry (PDSC) as measured by the ASTM D5483 test (4) is an alternative, non- bearing method of measuring the oxidative stability characteristics of grease. The current D5483 test method was developed by Rhee (5) over fifteen years ago and has been used by Rhee (6) more recently to conduct studies to develop a kinetic model for grease degradation. The D5483 test was also compared with D3527 (7) high temperature bearing life tests giving limited correlation. The other bearing tests that measure the useful life of grease of interest in this paper are the FE9 (DIN 51821-2) (8) and FAG FE 8 (DIN 51819-2) (9) tests to be described later.

Roller Bearing Life

In 1939, Weibull (10) related bearing life to the probability of survival for a group of bearings assumed to have been homogenously manufactured. Later, in

1947, Lundberg and Palmgren (11) introduced equation 1 for L_{10}.which is known as the basic rating life. It represents that number of revolutions in millions of cycles that 90% of a population of bearings will survive when tested or run under identical conditions. The L_{10} has been used since the 1950s.

$$L_{10} = (C / P)^n \qquad \text{equation 1}$$

where C is the capacity of the bearing, P is the applied load and n is a factor depending on the type of bearing, but typically 3 for ball bearings and 10/3 for roller bearings.

Equation 2 (12) represents a revision of the basic life equation modified to include life factor coefficients a_1, a_2,and a_3 where Lna is the adjusted bearing life.

$$L_{na} = a_1 a_2 a_3\, L_{10} = a_1 a_2 a_3\, (C / P)_n \qquad \text{equation 2}$$

where a_1 represents higher reliabilities than 90%, a_2 adjusts for material and process improvements and a_3 adjusts for operating conditions such as lubrication, loading, and operating temperature.

In ISO 281 (13), life adjustment factors are included that modify the basic life rating, taking into account factors for increased or reduced loading, thicker or thinner lubrication films generated by the lubricant and differing degrees of cleanliness or contamination. However, the biggest assumption made in ISO 281 is that the grease does not undergo any form of degradation and is still in good condition when the bearing surfaces reach the end of their life and failure ensues. Other bearing life levels used include the L_{50} (50% survival) and L_{01} in which 99% of the bearings should survive without failure. The concept of testing for grease performance relies upon choosing operating conditions where the hardware should not fail, but where the grease will fail by loss of effectiveness due to degradation or attrition of additives that provide performance. The L_{50} is of particular interest as it defines the minimum level of performance in the FE9 (DIN 51821-2) test as required in DIN 51825 (14) and ISO 6743-9 (15) specifications that are used to define the maximum operating temperature for roller bearing grease. Recently, Coe (16) has emphasized the importance of choosing methods that have technical merit such as the FE9 to develop the high temperature claims used to define suitability for use of grease in roller bearing applications.

Current Work

This paper describes the use of D5483 and the D3527 tests as screening methods to guide the formulation of prototype extended bearing life greases and to understand the contribution of different additive factors on oxidation and degradation in a single base grease versus among various types of grease. The performance of selected greases were then verified by using standard FAG FE9 (DIN 51821-2) and FAG FE 8 (DIN 51819-2) testing as further proof of performance.

Experimental

Materials

Base Greases: Base greases used in this work were all prepared in a pilot manufacturing system. Both lithium and lithium complex greases were prepared from two base oil blends to compare bright stock versus polymer as a way to build the base oil viscosity of the oil phase of the greases. Table 1 shows a summary of the two base oils used to make the different contactor greases. Base oil 1 was composed of a combination ISO VG 68 paraffinic oil (325N API Group I) base stock and a BS 150 paraffinic bright stock; while, base oil 2 consisted of the same ISO VG 68 base stock as oil 1 with polymer added as a bright stock replacement. Both oils had similar kinematic viscosity at 40ºC and hydrocarbon distribution; base oil 2 had a slightly higher viscosity index.

Table 1 : Base oils used for the greases

Oil	**1**	**2**
Viscous Component	Bright stock	Polymer
Viscosity @ 40ºC cSt	167.0	168.0
Viscosity @ 100ºC cSt	16.0	17.3
Viscosity Index	96	111
Analyses		
% Saturates	76.6	74.1
% Mono-Aromatics	18.2	20.7
% Di-Aromatics	5.2	4.0
% Polars	<1	1.2

Table 2 : Graded greases used in the studies

Grease	A	A1	B	C	C1	D
Type	lithium	lithium	lithium	lithium complex	lithium complex	lithium complex
Oil	1	1	2	1	1	2
Graded[1]						
% soap	6.44	6.58	6.43	9.85	8.62	8.62
D217						
P0	284	284	267	258	264	288
P60	287	281	273	263	276	293
P10k	301	295	276	281	299	304
P100k	319	315	302	300	318	321
D128 acid/base number[2]	1.10B	0.62B	0.63B	0.90B	0.76B	0.43B
D2265 dropping point, ºC	201	188	207	>300	>300	>300

[1] graded values [2] B = basic

All greases were made in the Lubrizol pilot system, consisting of a Contactor™[1] and a finishing kettle as slightly stiffer soap concentrates. They were adjusted to grade with the same base oil as was used to make the soap and subsequently milled through a Charlotte™[2] homogenizer. The base greases used in the studies were stiffer than

NLGI#2 consistency and were typically NLGI#3 consistency. For some studies, smaller samples with the various additives were made by diluting the base greases at 80ºC with the original base oil, mixing in the pre-blended additive concentrates for 20 minutes, homogenizing on a triple roller mill, and de-aerating under vacuum. Penetration was checked on all greases to ensure that the greases were in the correct penetration range.

[1] Contactor™ is a trade mark of Stratco, Inc
[2] Charlotte™ is a trade mark of Chemicolloid, Inc

Table 2 summarizes graded greases that were used in this work in various studies. Greases A, B, C, and D were used in initial studies; while, greases A1, B, C1, and D were used for the later studies. Greases A and A1 are different batches of lithium grease, and greases C and C1 are different batches of lithium complex grease. The lithium (Li) greases were made from 12-hydroxystearic acid (12-HSTA) and the lithium complex (LiX) greases used 12-HSTA and azelaic acid at a 2 to 1 mole ratio.

Additives: The first additized greases tested contained a fully formulated additive package (ADDV1) containing zinc dialkyl dithiophosphate (ZDDP), sulfur extreme pressure additive (S-EP), phosphorus-sulfur extreme pressure additive (P-EP), corrosion inhibitor, and rust inhibitors. The ZDDP and P-EP were used to provide a basic level of oxidation inhibition to the grease. Matrix testing involved varying the ZDDP, S-EP, and P-EP levels to better understands additive contributions to grease degradation.

In follow-up work, matrix testing involved additive revision by removing P-EP and varying ZDDP, S-EP, and corrosion inhibitor (CI) and also introducing and varying antioxidant (AO) to the additive formulation. The follow-up work focused on evaluating the additives for their interactions addressing environmental labeling.

Tests

D5483: The D5483 PDSC test was used to evaluate the oxidation resistance of experimental greases. The grease (1-2 mg) is placed in a small aluminum pan inside a pressure vessel on a heat flow sensor. An empty pan is placed on a second sensor and the vessel is sealed. The chamber is then heated to the test temperature of 180°C and 500 psi (3500 MPa) of oxygen is introduced. The sample is held isothermally at the test temperature until the grease starts to oxidize. The resulting exotherm is detected by the sensor. The point at which the oxidation accelerates away is calculated and this is termed the oxidation induction time (OIT). The 180°C temperature was chosen to be most relevant to the operating temperature of the D3527 test where the test spindle as controlled at 160°C and the chamber is frequently 180°C or higher.

D3527. The ASTM D3527 wheel bearing life test represents a measure of performance for automotive service grease under GB or GC requirements of the ASTM D4950 (17) standard classification. The test is considered a bearing oxidation/degradation test for grease. Two tapered roller bearings are run under a low axial load of 111Newtons at 1000 rpm. Based on the bearing torque after a running in period, a cut-off torque is calculated and if the grease breaks down and the torque reaches the cut-off the test is stopped and the number running of hours to failure is recorded. The bearings are run for 20 hours, and then undergo a 4 hour rest period. At the end of the rest period the bearings are restarted. Normally on start up after a rest period there is an increase in torque. If the torque does not drop to below the cut-off torque within a short period of time (typically 30 to 90 seconds), the test rig shuts itself off and the grease is considered to have failed at the end of the 20 hour running period, and the number of hours to failure reported.

DIN51821-2: In Europe the DIN51821 (FAG FE9) (10) test is widely used to define bearing oxidation life. The test utilizes 62mm angular contact ball bearings (ISO designation 7206) bearings mounted in housings similar to the D3336, except that higher thrust and axial loads are applied. The test equipment is defined by DIN51821 part 1 and the test method is defined in part 2 of the same standard. There is a choice of 3 axial loads 1500, 3000, 4500 N, of 2 speeds, 3,000 and 6,000 rpm and of standard temperatures: 120, 140, 160, 180, 200 °C. In order to generate meaningful data, the test requires a minimum of four, preferably five bearings to be run to failure and the L_{10} (F_{10}) & L_{50} (F_{50}) lives in hours are calculated by using Weibull statistics. The conditions chosen are described under results. A L_{50} of 100 hours under a given set of conditions is defined as the acceptable criteria to meet DIN and ISO grease standards for rolling element bearings under DIN 51825 and ISO 6743-9.

DIN 51819-2: The FAG FE8 test which has been standardized under DIN51819 (13) utilizes one of three types of bearings that can be run depending upon the type of lubricant to be tested. For grease testing the bearings are designated type (A) 7312B angular contact bearing with a polyamide cage which can be run up to a maximum temperature of 120 °C, type (B) 7312B angular contact bearing with a copper/ zinc cage which can be run up to a maximum temperature of 200 °C and type (C) 31312 tapered roller bearing capable of running up to 200 °C. The bearings are pre-weighed and then filled with the appropriate amount of grease. They are run in pairs for 500 hours or until over-torque failure or until over-temperature failure and then the amount of wear on the rolling elements, cage and raceways is determined by weight loss. The DIN 51819 method lists a choice of temperatures from running at ambient with no applied heat to controlling at 120, 140, 160, 180, 200 °C, with 120, 160, 200 °C being preferred temperatures. After running four bearings, the amounts of wear occurring are plotted using Weibull statistics and the F_{50} wear / weight loss form the elements recorded. It is possible to use only two completed tests to determine the F_{50} weight loss but

Table 3. Grease study 1 results

Grease	E	F	G	H
Type	lithium	lithium	lithium complex	lithium complex
Oil	1	2	1	2
% soap	6.44	6.43	9.85	8.62
D217				
P0	289	274	288	308
P60	291	278	290	308
P10k	296	281	313	326
P100k	301	304	327	344
D2265 dropping point, ºC	204	216	>300	>300
D5483, OIT at 180ºC, minutes	27.6	22.3	22.5	20.3
D3527, hrs	20	60	60	120

the uncertainty of measurement is much higher than when running 4 bearings and the difference between greases has to be much higher to determine if the two results are statistically significantly different. A typical requirement for minimum performance is an F_{50} maximum of 35mg of weight loss from the rolling elements and 100mg from the cage.

Test Results and Discussion

Grease Study 1

Study 1 began by formulating bright stock-containing greases similar to greases A, B, C, and D in Table 4, except replacing some of the oil used to grade the grease consistency with an equal percentage by weight of ADDV1. The oxidative stability and D3527 were compared. Table 3 shows a summary of the findings.

It was found that there was no significant advantage on oxidative stability between oils 1 and 2, though, directionally the oxidation induction times favored the use of bright stock. The D3527 wheel bearing life did show an advantage in using lithium complex over lithium grease and a larger advantage in using oil 2 containing polymer over oil 1 containing bright stock.

Grease Study 2

Study 2 consisted of running a matrix of additives in greases to better understand their impact on oxidation and bearing life. The bright stock containing LiX grease was chosen for this work with the idea of reaching 80 hours in the wheel bearing life test required to meet the ASTM D4950 requirement for NLGI GC wheel bearing grease. The ZDDP, S-EP and P-EP components were varied as indicated in Table 4. In work that was previously reported it was shown that both S-EP and P-EP provided no inhibition to the grease alone or in combination with each other using oven storage testing (18).

The D3527 results showed very little variation with only one exception. The exception was that the P-EP appeared to detract from the ability to achieve a longer time. Item 8 in Table 6 contained all three additive components, but it gave 40 hours which was about typical of most of the greases tested. Item 5 containing ZDDP and S-EP lasted 140 hours providing a GC level of performance. The D5483 and D3527 results did not correlate (R^2<0.2).

To better understand what was happening to the grease, the states of the greases from the bearings

Table 4 : D3527 LiX grease degradation comparisons

Item	Additives			PDSC	D3527				
	ZDDP	S-EP	P-EP	OIT, min	hours	Grease state - visual	Grease state - FTIR	Ox rate FTIR/hr	Conclusion
1	0	0	0	2.3	20	dried residue	Ox 5.0*	0.250	result looks valid
2	1	0	0	44.6	40	still greasy	Ox 4.6	0.115	May be early failure
2	1	0	0	44.6	60				
3	0	1	0	2.6	60	dried residue	Ox 5.5	0.092	result looks valid
4	0	0	1	1.9	20	dried residue	Ox7.5	0.375	result looks valid
5	1	1	0	43.0	140	dried residue	Ox 7.25	0.052	result looks valid
6	1	0	1	38.1	20	still greasy	Ox 2.0	0.100	Certain early failure
6	1	0	1	38.1	60	SB dried, LB dry & lumpy	Ox 3.3 / 5.5	0.055/ 0.0917	result looks valid
7	0	1	1	2.3	20	SB still greasy	Ox 4.2	0.210	LB dry+SB ok
8	1	1	1	42.0	40	LB still greasy	Ox 3.0	0.075	SB dry+LB ok
9	0.5	0.5	0.5	27.3	40	grease was lumpy	Ox 3.3	0.083	May be early failure
9	0.5	0.5	0.5	26.6	40	LB still greasy	Ox 5.25	0.106	SB dry+LB ok

* Ox : oxidation measured at 1712 cm-1 normalized to new base grease
LB= large bearing; SB= small bearing

were examined visually to assess test validity and by FTIR using a Thermo Electron Nicolet 380 instrument with a diamond horizontal attenuated total reflectance (HATR) crystal. This technique was used because it gave semi-quantitative data, did not block out bands between 700 and 400 wave numbers (cm^{-1}), and only required a very small sample size to assess the degree of oxidation. A range of visual appearances were observed for the large and small bearings from the tests. The visual appearance did not clearly relate to the number of hours run as some greases appeared visually greasy but only lasted 20-40 hours long. The FTIR showed oxidation/degradation as a major peak of 1712 cm^{-1}, with other significant peaks at 1730 and 1770 cm^{-1} forming a degradation triplet. Allowing for differences in instruments, the peaks at 1730 and 1770 cm^{-1} tied-in well with the findings of Cann (19) of base oil oxidation being at 1733 and 1768 cm^{-1}. The extent of degradation was normalized to base grease at 1712 cm^{-1} for all greases tested. The rates of oxidation were calculated by dividing the carbonyl level by the number of hours tested. This showed that item 5 in Table 4 that ran 140 hours had the lowest rate of oxidation forming a dry residue upon failure. All greases after the end of D3527 tests showed some level of oxidation.

Grease Study 3

In study 3, the formulation strategy included three distinct parts using D5483 and D3527 test methods as performance measures, as well as considering environmental criteria. First, the additive formulation was optimized through a designed experiment that consisted of a matrix of 15 formulation runs. Second, the LiX base grease D, using base oil 2 from Table 4, was emphasized for additive evaluation. Third, a chosen additive was then evaluated in LiX grease C1 from Table 2.

Matrix testing in grease D: Additive optimization included a reformulation and removal of P-EP from the previous grease formulated. The ZDDP and S-EP additives identified in Study 2 were the two main components evaluated. In addition, corrosion inhibitor (CI) and antioxidant (AO) were used as a supplement to enhance the corrosion and oxidation resistance of the grease. ADDV1, as represented by grease H (Table 5), provided adequate wheel bearing life but required that the grease be labeled with the “dead tree/dead fish” symbol in the EU. The additive was reformulated such that the eco-toxicological label would not be required. This reformulation is represented by item 15 in Table 8. All other additive components were held constant.

Table 5. Comparison of D5483 and D3527 data

Item	Matrix*					D5483	D3527
	Code	A	B	C	D		
	Additive	ZDDP	S-EP	AO	CI	OIT min	HOURS hr
1		1				15.0	40
2			1			2.3	40
3				1		27.8	20
4					1	2.0	32
5		1	1			16.2	40
6		1		1		73.8	60
7		1			1	11.3	40
8			1	1		79.3	60
9			1		1	2.7	20
10				1	1	29.6	40
11		1	1	1		95.5	80
12		1	1		1	12.3	80
13		1		1	1	70.2	120
14			1	1	1	67.9	40
15		1	1	1	1	104.9	200,80,120

* Codes are assigned for statistical analyses

Table 5 shows a summary of D5483 and D3527 wheel bearing life results for greases made using a revised additive matrix over study 2. Items 1-4 represent greases containing a single additive component. Items 5-10 are greases formulated with pairs of the individual components. Items 11-14 represent greases formulated with a combination of three components, and item 15 contains all four additives. All of the greases were additized at a final soap level of 8.62% in base grease D and milled for homogeneity.

The D5483 gave a range of OIT values (minutes) which were analyzed using Minitab®[3] statistical software to determine which additives had a significant impact on the results. The OIT minutes measurement was first transformed using the logarithmic transformation in order to give the response a more normal distribution. The half normal probability plot constructed in this analysis is shown in Figure 3. The half normal plot included single component, binary component, and ternary component effects. Effects not falling on or close to the line in Figure 3 were not significant.

Points for AO, ZDDP, the ZDDP-AO interaction, and S-EP fell furthest from the line and were estimated as significant and the most important factors impacting oxidation. Figure 4 shows the final reduced model fit. The value in the "Coef" column estimates the effect of adding that component on log (OIT minutes). While the magnitude of the S-EP effect is much smaller, it was significant. Its effect also depends on the ZDDP and AO presence. CI had no impact. The model fits the data quite well, as can be seen by the significance of all the terms ($P<0.05$) as well as by the high R^2.

The overall impact of the significant components and their interactions is depicted by the plot in Figure 5 that shows predicted combinations of the presence and absence of the most significant additives in a nested fashion.

[3] Minitab® is a trade mark of Minitab, Inc.

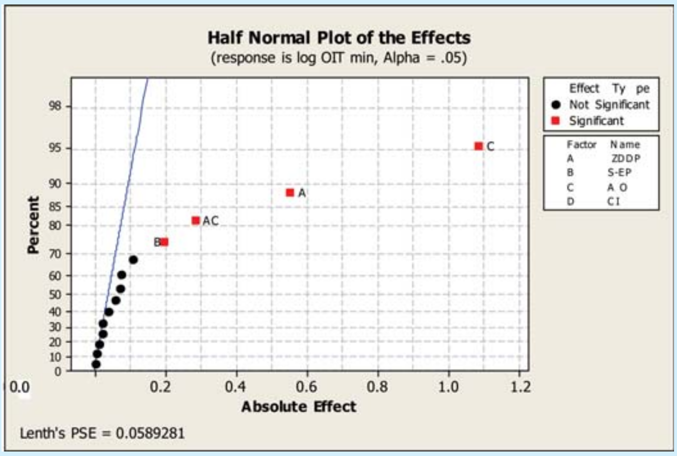

Fig. 3. : D5483 Half Normal Probability Effects Plot

Estimated Effects and Coefficients for log OIT min (coded units)

Term	Effect	Coef	SE Coef	T	P
Constant		0.2556	0.05412	4.72	0.001
ZDDP	1.7633	0.8817	0.06137	14.37	0.000
S-EP	0.3272	0.1636	0.06137	2.67	0.029
AO	2.4495	1.2247	0.06137	19.96	0.000
ZDDP*S-EP	-0.3478	-0.1739	0.06469	-2.69	0.028
ZDDP*AO	-1.0551	-0.5275	0.06469	-8.15	0.000
S-EP*AO	0.3978	0.1989	0.06469	3.07	0.015

S = 0.0613706 PRESS = 0.132691

R-Sq = 99.40% R-Sq(pred) = 97.37% R-Sq(adj) = 98.95%

Fig 4 : D5483 Model Fit

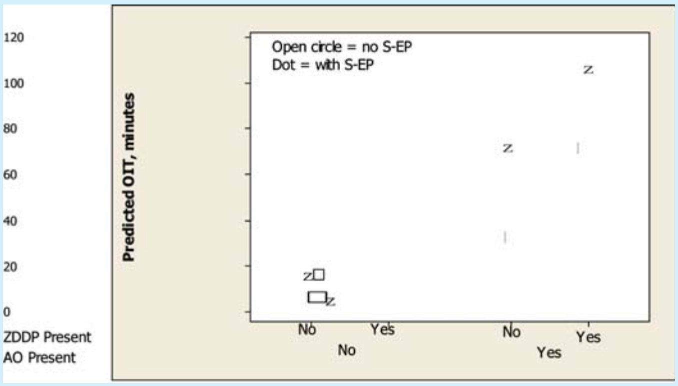

Fig. 5 : Predicted Effects of Key Components

A similar analysis was completed for the D3527 data where the response is hours until overtorque (generally in the start-up phase). Both the actual and log (hours) were analyzed, but neither yielded a model that explained a high percent of the variation. Only the use of ZDDP and AO appeared to be significant. The effect of these components is shown in Figure 6. Repeat results are joined by a line. Poor repeatability in this test makes it difficult to cleanly detect chemistry effects. Estimates for a model fit using ZDDP and AO are in given in Figure 7. While the R^2 is low, the error in the predictions from this model is what we would expect based on test repeatability, thus this is the “best” we could expect from a model.

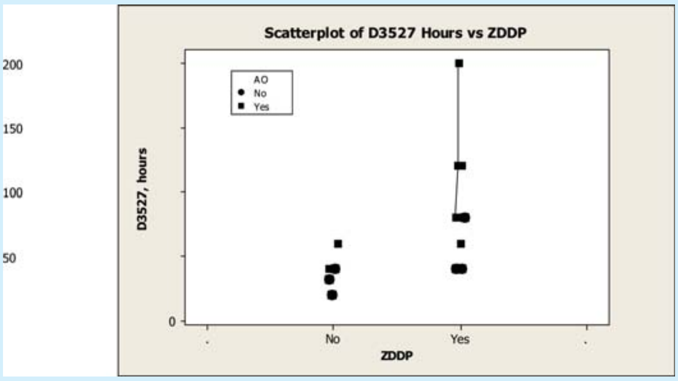

Fig. 6 : D3527 results vs. ZDDP and AO

Estimated Effects and Coefficients for Log D3527 hours (coded units)

Term	Effect	Coef	SE Coef	T	P
Constant		1.3932	0.08507	16.38	0.000
ZDDP	0.6825	0.3412	0.08897	3.84	0.002
AO	0.4695	0.2347	0.08897	2.64	0.019

S = 0.180470 PRESS = 0.669804

R-Sq = 61.40% R-Sq(pred) = 43.29% R-Sq(adj) = 55.88%

Fig. 7 : D3527 Model Fit

Grease C1 testing: The combination of additives of item 15 from table 5 was assigned the designation of ADDV2. The ADDV2 was also evaluated in bright stock- containing grease C1 and assigned designation grease I. ADDV2 formulated in grease D was assigned the designation grease J. The two greases use the same graded characteristics as greases C1 and D in Table 4 except with additive replacing a like weight of grading oil. The characteristics of the formulated greases are given in Table 6. Both greases were formulated to 8.62 %wt soap. Grease I was 22 units stiffer than grease J. The D5483 OIT was 34% shorter for grease I and the D3527 hours to failure of grease I were 75% shorter than grease J based upon triplicate results.

Table 6 : Lithium Complex Grease Comparisons

Grease	I	J
Type	lithium complex	lithium complex
Base grease	C1	D
Oil	1	2
Heavy component	Bright stock	Polymer
% soap	8.62	8.62
D217		
P0	293	320
P60	302	324
D2265 dropping point, ºC	299	261
D5483, OIT at 180ºC, minutes	68.9	104.9
D3527, hrs (average of 3 separate tests)	33	133

Environmental considerations: Formulating finished greases for global acceptance requires due health, safety and environmental considerations, particularly related to eco- toxicity. Europe is a good example of a market where improved environmental acceptance may prove to be a significant differentiating attribute. The guidance for classification, packaging and labeling of chemical substances, Directive 67/548/EEC (20), has been in existence for more than 30 years. Directive 67/548/EEC and its subsequent amendments impact individual chemicals while Directive 1999/45/EU (21) relates to preparation or formulation of two or more chemicals together, including the environmental risk phrases and symbols used to identify such materials in the market. It also covers calculation methods to define risk phrases of complex components mixtures typically found with lubricants. There has been a push toward risk assessment in EC regulation 1907/2006 for Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) and its subsequent amendments (22). This is consistent with Directive 2000/53/EU on the End of Life Vehicles (23), proscribes hazardous chemicals from being included in vehicle componentry which could prevent the vehicle components from being re-cycled or re-used. A good summary of directives, legislation and labeling may be found in Lubricants and Lubrication edited by Mang and Dresel (24). Directive 1999/45/EC was amended in 2006 requiring preparations to be labeled for ecotoxicity, as well as danger to the environment. This was taken into consideration in this work in order to improve the labeling of greases to avoid the “dead tree/dead fish” hazard symbol (24).

Figure 8 summarizes the outcome of the testing of additives between the different base greases. ADDV2 was able to exceed the performance of ADDV1 and reduce the need for environmental labeling. The D5483 results are not included as they were run on different builds of the same machine in two different time periods making the data incomparable with each other.

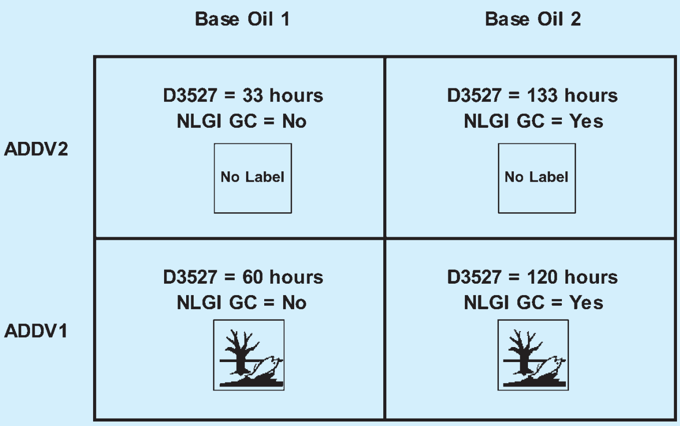

Fig. 8 : Additive comparison

Grease Study 4

The final testing focused on further evaluation of greases I and J containing ADDV2 in relevant bearing life tests for oxidation and wear. The standard FAG FE9 (DIN 51821-2) and FE 8 (DIN 51819-2) were chosen as the test methods. Weibull statistics calculations provided in this paper were performed by the authors with Minitab® statistical software using the maximum likelihood estimate. The FE9 test is used to evaluate upper operating temperature for both

Table 7 : DIN 51821-2 (Method A/1500/6000) FE9 Results

Grease	T ºC	Bearing fail hours					Life estimates			
		1	2	3	4	5	L01	L05	L10	L50
I	140	291	267	195	344	317	158	199	221	288
J	140	307	333	381	292	409	213	258	280	349
J	160	102	178	214	117	144	54	81	96	152

DIN 51825 and ISO 6743-9 specifications where bearing grease is desired. To accomplish this, the grease is run on four or five bearings at the desired upper operating temperature and must give a L_{50} (F_{50} used interchangeably) of at least 100 hours. Table 7 summarizes the data for an operating temperature of 140 ºC for greases I and J. The data clearly shows both greases to be acceptable. Grease J was further tested at 160ºC consistent with the temperature specified in the D3527 test. Once again, grease J provided 150% of the required F_{50} (L_{50}) for bearing life.

Figure 9 shows a cumulative plot of percent failure based upon best fit Weibull model for the FE9 test. The 50% cumulative failure line references the minimum number of hours that F_{50} (L_{50}) must meet to conform to the DIN 51825 (30) and ISO 6743-9 (31) standards. The curve crossing the 50% line indicates the pertinent value of F_{50} for a given grease and temperature. The comparison of greases I and J at 140 ºC shows that the choice of base oil has an impact on bearing life, although not considered statistically significant between greases I and J. The comparison of grease J at 140 ºC and 160 ºC shows the much greater impact that temperature has on bearing life. The results are statistically significant between temperatures.

The DIN 51819-2 was the second test method used to assess ADDV2 performance, but only grease J was tested (Table 8). Both low-load, high-speed and high-load, low- speed testing was performed using the 31312 tapered roller bearing (TRB). The method assesses rolling element and cage wear. The individual wear amounts are identified as weight loss from components (i.e. cage, raceways, rolling elements) and Weibull statistical methods applied to determine the F_{50} weight loss. The results are reported as the F_{50} (mw50, mk50) weight loss after 500 hours. If the bearings fall short of the full running time, then those run hours are the defined life. The DIN 51819-2 states that wear above 100mg for rolling elements and raceways, and above 200mg for cages, are significant in practice. Only one pair of bearings was tested in this study. Both conditions tested provided low rolling element and cage wear for grease J containing ADDV2.

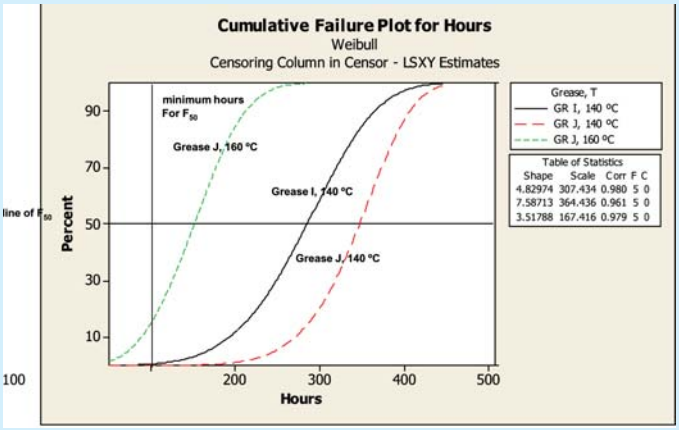

Figure 9. Comparison of FE9 Weibull cummulative failure plots

Table 8. DIN 51819-2 FE8 Results

Grease	Test parameters				Rolling element wear in mg			Cage wear in mg		
	load kN	speed rpm	duration h	T ºC	1	2	mw50	1	2	mk50
Target									<100	<200
J	10	3000	500	120	5	13	8.8	17	6	11.1
J	80	75	500	monitor	1	13	4.6	26	35	31

Summary

Greases with extended grease and bearing life were developed by static D5483 and dynamic D3527 tests as a guide to vary the oil type, grease type and specific additive components consistent with today's market. The greases developed are pertinent to the North American and European markets based upon industry standard tests and specifications.

Greases with extended grease and bearing life were developed by using a variety of static and dynamic tests as a guide to vary the grease type, oil type and specific additive components consistent with today's market. The greases developed are pertinent to the North American and European markets based upon industry standard tests and specifications.

A combination of D5483 PDSC and ASTM D3527 wheel bearing life test allowed changes in oil and grease type and additive components to be evaluated for their merit as components to formulate improved grease.

Grease Study 1 showed that lithium complex grease with base oil thickened with polymer and containing ADDV1 was preferred over the same viscosity base oil thickened with bright stock and containing ADDV1.

Grease Study 2 showed that dynamic D3527 testing was significantly more severe than static PDSC testing, though antagonistic additive components like P-EP and beneficial components like ZDDP could be identified and acceptably applied in a formulation with necessary components for extreme pressure characteristics such as S-EP.

Grease Study 3 further showed that combining the beneficial base oil and additive knowledge with the further supplemental additives like AO provided overall improved grease. Specific additives and additive interactions that favored improved life and bearing were statistically identified. The improvements not only could show significance in oxidation stability and dynamic bearing life, but could also address and impact the environmental labeling of the improved grease as represented by ADDV2.

Finally, Grease study 4 showed that the improved additive provided significant dynamic bearing performance on both oxidation and wear over a range of operating temperatures and conditions. The DIN test methods used in this assessment shows that the grease containing ADDV2 may provide fairly universal application in the global market.

Conclusions

The use of screening tests to look at the significant factors affecting bearing life have been utilized and allowed the development of greases and grease additive packages for extended bearing life as measured by industry standard tests. Not only was it shown that the choice of base oil was important in improvement of bearing life, but that the specific balance of types of additives were important also in achieving improved bearing life through the use of the D5483 and D3527 tests as a guide to formulate grease to North American and European bearing requirements while improving eco-toxicology of the finished grease.

Further work

It is planned to make further studies looking in more detail at urea thickened greases and at lithium complex greases with sebacic and adipic acid as the complexing agent.

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