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- VANLUBE RI-BSN - Neutral Barium Dinonylnaphthalene Sulfonate.
- VANLUBE RI-CSN - Neutral Calcium Dinonylnaphthalene Sulfonate.
- VANLUBE RI-ZSN - Neutral Zinc Dinonylnaphthalene Sulfonate.
- VANLUBE 0902 - Multi functional S/P package.
- VANLUBE 407 - Antioxidant.

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President’s Podium

Extended Bearing Life Greases
“Tried and True” or New Technology?
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Lubricating Greases: Lubricate Eyes or Bearings?
Dr. Anoop Kumar, Dr. Navendu and Bill Mallory

Synthesis and Characterization of Mixed Thickener Containing Greases for Bearing Applications
Sathwik Chatra K R and Dries Muller

ON THE COVER
As machinery is built to be more efficient and transmit more power, rolling bearing elements are increasingly required to operate at higher loads, speeds and temperatures. Read more beginning on page 6.

Published bi-monthly by NLGI. (ISSN 0027-6782)
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The NLGI Spokesman is a complimentary publication. Past issues may be found on the NLGI Website, www.nlgi.org
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The 2016 NLGI Grease Production Survey (GPS) is almost complete and will be available after the June 10th-13th Annual Meeting in Squaw Creek, California. This year’s survey results will be loaded with all of the important data upon which you’ve come to rely, and don’t forget that NLGI is now offering the data in an Excel format. NLGI members and survey participants will continue to receive the PDF survey results at no cost; additionally, those looking to customize the data will greatly appreciate the time-saving Excel file format available for purchase on the NLGI website.

How much grease was manufactured in 2016? Do lithium and lithium complex greases continue to represent the primary grease thickener type manufactured globally? Has the lack of availability and rising cost of lithium hydroxide affected production of these greases? The NLGI GPS has the answers.

The NLGI Production Survey counts the global production of grease, providing a snapshot of growth (by thickener type and base oil type) and demographic (by thickener type, base oil type, and global region). Both provide an opportunity to stand up and be counted, to make a difference, to have your voice (or your production) be heard. Has grease continued to lubricate the global economy? The GPS has the answers.

The NLGI Grease Production Survey continues to be the single most comprehensive global report on grease production. The 2015 survey identified a global decline of approximately 1% compared to 2014. Will 2016 show a reversal into growth again? Last year’s 2015 survey reflected an overall increase in calcium, aluminum, polyuria and clay thickened greases while combined lithium greases saw a slight decline. Not surprisingly, conventional mineral oils still represent the primary fluid used globally to produce greases. This is just the tip of the information iceberg contained in the NLGI Grease Production Survey. The Grease Production Survey continues to be one of the most important member benefits provided by the NLGI to its membership. The information in the Grease Production Survey Report is a valuable source of past results and can be used as an indicator of future trends. This makes the Grease Production Survey Report a very useful strategic management and production tool.

The Grease Production Survey Report data for 2016 will be categorized by primary thickener types, base fluid types, and geographical region. These classifications can provide answers to many questions you may have regarding the grease industry:

- What tonnage of lithium soap grease is produced in my region of the globe?
- What base fluid type represents the greatest percentage of grease production?
- Which region of the globe demonstrated the greatest year on year growth?
The NLGI Grease Production Survey has the answers. It is a wealth of useful information, structured and organized to assist in executing successful strategic decisions within the grease industry.

The value and accuracy of the NLGI Grease Production Survey requires your participation and accurate reporting; we need your support. Through your active participation in next year’s 2017 survey, we hope to attain a 100% participation rate, thereby reflecting the entirety of global grease production. Consequently, if you have not yet had the opportunity to participate in the NLGI Grease Production Survey, please contact Grease Technology Solutions at the address below:

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Round Hill, VA 20141-2392 USA
Phone: 1-540-338-8040
Fax: 1 540-338-8063
Email: chuckcoe@grease-tech.com

Please note that the information received from individual companies is held in strict confidence by Grease Technology Solutions LLC. The NLGI Grease Production Survey Report is a valuable member benefit and your participation is integral to its continued success.
Extended Bearing Life Greases
“Tried and True” or New Technology?

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Afton Chemical Corporation • Richmond, VA
Afton Chemical India Pvt. Ltd • Mumbai, India

Abstract
An experimental calcium sulfonate complex (CSC) grease and standard lithium complex grease (LiCx) were produced with the same base oil composition. The base greases were then formulated with two additive systems. The additive systems consisted of a primary antioxidant component and a multifunctional component containing antiwear and extreme pressure additives with secondary antioxidant function. The two base greases were treated with the single components and a combination of the two components. High temperature bearing performance of the six formulated greases was evaluated using ASTM D3527 wheel bearing life (WBL) test. Greases were also evaluated by a protocol designed to study the effect of thermal aging on the oxidative and shear stability of the greases. Specifically, the greases were statically aged at the same temperature (160 °C) and in the same inboard roller bearings used in the WBL test. Bearings were weighed before and after aging to determine weight loss. Pressure Differential Scanning Calorimetry (PDSC) was used to monitor oxidation stability before and after aging, and infrared (IR) spectroscopy was used to measure extent of oxidation. Changes in grease shear stability were measured using oscillatory shear rheology strain sweeps performed at 160 °C. The WBL data showed that CSC and LiCx greases had similarly short-lived performance when greases were treated with each of the single components while greases treated with the combined components produced extended lives. Although antioxidant synergism is expected between the two components of this study, the rheological analyses of the fresh and statically aged greases suggest that the additives can alter the thermal and mechanical stability of the greases in ways that positively and negatively impact grease life in high temperature wheel bearings.

Introduction
As machinery is built to be more efficient and transmit more power, rolling bearing elements are increasingly required to operate at higher loads, speeds and temperatures. The steady rise in bearing operating temperature has gradually expanded the demand for greases with improved oxidative and thermal stability. To enhance oxidation resistance, grease manufacturers have turned to higher quality base stocks such as Group II and III mineral oils and synthetic fluids like PAO's and esters. To improve thermal stability, the use of thickeners that impart higher dropping points is also trending upwards. Two grease types that have seen noticeable growth in their production volumes are lithium complex (LiCx) and calcium sulfonate (CS) greases. This increase is demonstrated in Figure 1, which compares production volumes in 2004 versus 2014:

LiCx grease is an established commodity with a proven service record while CS grease, although not new to the industry, is considered an up and coming technology. Greases based on lithium soap are good all-around performers that have excellent pumpability, shear stability and water resistance. The ability to complex simple lithium soaps and raise dropping points from about 200 °C to >260 °C have made LiCx greases the conventional choice for high temperature applications including automotive wheel bearings.

To achieve their high dropping points, the production of LiCx greases involves the use of complexing agents such as boric acid, salicylic acid and di-carboxylic acids. Of these
complexing agents, the di-carboxylic acids, azelaic acid and sebacic acid, are the most widely used. With di-carboxylic acids, the manufacturing process typically involves in-process saponification of the fatty acid, typically 12-hydroxystearic acid (12-HSA) and the complexing agent followed by heating and a cooling step that co-crystallizes both thickener components into micron sized fiber network2-3. Due to incorporation of the dibasic salt, it can be ascertained that the thickener network will have higher ionic character that increases melting/dissolution temperatures of the soap fibers and amplifies inter- and intra-fiber interactions that strengthen thickener network.

CS greases are thickened by overbased calcium sulfonates. The technology date back to 1960’s and recent NLGI papers by Denis et al. and Waynick offer excellent overviews on their compositions and preparation methods6-7. Overbased calcium sulfonates are lubricant additive that find their highest use in combustion engine oils where they neutralize oxidation-derived acids as well help suspend polar oxidation products. Their compositions are generally described as colloidal dispersions of amorphous calcium carbonate particles stabilized in carrier oil by sulfonate surfactant, typically calcium alkylbenzene sulfonates8. CS greases are formed when the amorphous calcium carbonate particles are converted to the crystalline allotrope calcite. This process is carried in base oil to form greases with gel-like structure that typically require 40 to 50 mass percent of the overbased calcium sulfonate raw material to produce NLGI 2 consistency grade greases. The high calcite content of CS greases provides excellent EP, AW and rust inhibiting properties. CS greases are also recognized for their shear stability, water resistance and high dropping points that commonly exceed 300 °C. On the other hand, the high demand and low thickening efficiency of overbased calcium sulfonates makes CS greases relatively expensive9. In addition, the gel-like structure of CS greases negatively affects their pumppability and limits oil release that is deemed an important property in bearing lubrication10.

In 1985, the first CS grease classified as calcium sulfonate complex (CSC) grease was patented by Muir and Blokhuis. The development involved the use of relatively low levels of calcium soap thickener and boric acid complexing agent to reduced overbased calcium sulfonate content to levels approaching 30 mass percent. Further refinements have followed and calcium sulfonate contents have continued to drop mitigating issues with cost, pumppability and other drawbacks that hindered the use of CS greases in the past6-7; 9-11.

The focus of this paper is a preliminary assessment of an experimental CSC grease in the ASTM D3527 high temperature wheel bearing life (WBL) test. A fair amount of work has been done with this test. This includes Rhe’s use of PDSC and a modified Thermal Gravimetric Analysis (TGA) procedure to build a “decomposition kinetic model” to predict performance in the WBL test12; Ward and Fish’s use of PDSC and the WBL test to guide the development of grease with extended life in FAG FE-9 bearing rig test13; and the use by Kaperick et al. of high temperature oscillatory shear rheology to assess the ability of greases to perform in the WBL test14.

The first part of this paper involved WBL testing of the experimental CSC grease and LiCx benchmark both produced with the same base oil composition and formulated with same antioxidant systems. The second part of the paper involved measuring the effect of thermal aging on weight loss, oxidative stability, extent of oxidation and rheological properties of the formulated greases with the goal of finding the factors that most influence performance in the WBL test. Specifically, the greases were statically aged at the same temperature (160 °C) and in the same inboard roller bearings used in the WBL test. Bearings were weighed before and after aging to determine weight loss. PDSC was used to monitor oxidation stability before and after aging, and infrared (IR) spectroscopy was used to measure extent of oxidation. Changes in grease shear stability were measured using oscillatory shear rheology strain sweeps performed at 160 °C.

### Experimental

#### Materials

**Base Greases:** Two base greases, one LiCx and one CSC, were prepared for this study. Both greases were produced with the same base oil composition. The base oil used consisted of 3 parts of a 600 SUS Group I base oil and 1 part of a 2500 SUS Group I bright stock. The final viscosity grade of the base oil was ISO VG 150.

The two base greases were made by batch processes. The preparation of the LiCx grease was based on a procedure described in Example 1 of U.S. Patent Number 3,791,97315. The procedure used 12-hydroxystearic acid and azelaic acid at 2 to 1 mole ratio and the saponification of the acids was done in two steps. The experimental CSC base grease was prepared using a proprietary process. Basic properties for both base greases are provided in Table 1.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Method</th>
<th>CSC</th>
<th>LiCx</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dropping Point, °C</td>
<td>ASTM D2265</td>
<td>&gt;307</td>
<td>306</td>
</tr>
<tr>
<td>NLGI Grade, mm⁻¹</td>
<td>ASTM D1403</td>
<td>279</td>
<td>274</td>
</tr>
<tr>
<td>Oil Separation (40 °C; 188h), % wt. loss</td>
<td>IP 121</td>
<td>1.1</td>
<td>6.5</td>
</tr>
<tr>
<td>Roll Stability (4h; 80 °C), % change in Pen</td>
<td>ASTM D1831</td>
<td>0.7</td>
<td>14</td>
</tr>
<tr>
<td>Four-Ball EP (Weld Point), kgf</td>
<td>ASTM D2506</td>
<td>315(280)*</td>
<td>200 (200)*</td>
</tr>
<tr>
<td>Four-Ball Wear (1200rpm, 40kg, 75C, 1h), mm</td>
<td>ASTM D2266</td>
<td>0.500</td>
<td>0.88</td>
</tr>
</tbody>
</table>

*Actual weld point when measured 10 kgf intervals
Additives: The base greases were formulated with two additive systems. The additive systems were selected based on recognized performance in LiCx greases. The additive systems consisted of HT-1, a multifunctional component that is composed of zinc dialkyl dithiophosphate (ZDDP) and a sulfurized compound and HT-2, a primary antioxidant component. For the study, the two base greases were treated with the single components and with a combination of the two components to produce three LiCx and three CSC greases as summarized in Table 2:

<table>
<thead>
<tr>
<th>Components</th>
<th>CSC-1</th>
<th>CSC-2</th>
<th>CSC-3</th>
<th>LiCx-1</th>
<th>LiCx-2</th>
<th>LiCx-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSC base grease (wt. %)</td>
<td>97</td>
<td>99.2</td>
<td>96.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LiCx base grease (wt. %)</td>
<td></td>
<td>97</td>
<td>99.2</td>
<td>96.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HT-1 (wt. %)</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>HT-2 (wt. %)</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

Test Methods

Wheel Bearing Life: The high temperature life of the formulated greases prepared from the experimental CSC base grease and conventional LiCx base grease was compared using ASTM D3527 WBL test method. The method is a measure of service life for automotive service grease and is part of NLGI GB and GC wheel bearing grease specification. The test evaluates greases under high temperature and high shear conditions using tapered roller bearings operating in a modified front wheel hub-spindle-bearing assembly. A light axial load of 111 N is applied to the bearings while the hub is rotated at 1000 rpm and spindle temperature is maintained at 160 °C. The test duration consists of a 20 hour running period followed by a 4 hour resting period. This cycle is repeated until grease deterioration causes the drive motor torque to exceed a cut off that is calculated from an initial two hour running period. Failures normally occur after the resting periods and test results are typically reported in 20 hour intervals. GB and GC specification limits are 40 and 80 hours respectively.

Static Grease Aging: Formulated greases were aged at 160 °C in the inboard wheel bearings that are used the ASTM D3527 test rig. The bearings are the same as the bearings utilized in ASTM D1743 Bearing Rust Test. Thus, cleaning and packing of the bearings was done by ASTM D1743 procedures. Heating of the packed bearings was accomplished in an oven in a vertical position. Packed bearings were held in the oven by sliding them onto a horizontal bar held by a vertical stand. Three bearings per experiment were used to collect sufficient sample to conduct other testing. Grease from the three bearings was joined together to make a composite sample for ensuing evaluation.

Weight Loss: Bearings used for grease aging were weighed before and after packing and then again after aging. For each experiment, composite weight of the three bearings used per experiment was used to determine percent weight loss data. Percent weight loss was calculated by subtracting the total weight of the packed aged bearings from the total weight of packed un-aged bearings and dividing this value by total amount of grease packed into the bearings. The latter value was calculated by subtracting the total weight of unpacked bearings from the weight of packed un-aged bearings.

Oxidative Stability: The oxidative resistance of the formulated greases before and after aging was measured following ASTM D5483, Standard Test Method for Oxidation Induction Time (OIT) of Lubricating Greases by Pressure Differential Scanning Calorimetry (PDSC). The test temperature selected for this study was 180 °C.

Extent of Oxidation: Infrared (IR) analysis of the aged greases was conducted using Perkin Elmer FT-IR Frontier Spectrometer with Universal ATR Sampling Accessory. IR absorbance for carbonyl (C=O) bonds is excellent indicator of oxidation product build-up in the grease. As oxygen attacks hydrocarbon parts of the grease, products containing carbonyl bonds such as ketones, aldehydes, carboxylic acids and carboxylic acid esters are formed and produced a broad absorbance peak that ranges from 1700-1800 wavenumber. For this study, the relative intensity of this peak was used to determine the extent of oxidation of the formulated greases after aging.

Shear Stability: Oscillatory shear rheology was used to measure yield point, which is the shear stress where grease loses resistance to flow, and storage modulus at the flow point (flow point for short), which is the shear stress where grease loses structural stability and becomes fluid. An Anton-Paar oscillatory rheometer (MCR301) was used to measure these rheological properties of the base greases and formulated greases before and after aging. For each analysis, the greases were compressed between a temperature controlled Peltier bottom plate and a parallel top plate. A hood that contains a temperature controlled Peltier device was placed over the assembly and a strain sweep ranging from 0.01% to 1000% strain at 160 °C was performed. The yield point was measured at the point where the storage modulus (G’) deviated from the linear viscoelastic (LVE) portion of the sweep. Also, from the strain sweep, the place at which storage modulus (G’) and loss modulus (G”) are equal was determined (flow point). This is the stress at which the internal structure of the grease moves from grease-like material to a liquid-like material. An example of this strain sweep procedure is illustrated in Figure 2:

Results

The additives used to formulate the greases of this study were selected based on the established performance in other
LiCx greases. Specifically, the use of primary antioxidants to supplement the antioxidant capacity of ZDDP and sulfur compounds is a well-established approach to extending the high temperature life of LiCx greases and lubricants in general. Therefore, one goal of the study was to determine if the same approach was applicable to the experimental CSC grease.

As summarized by Figure 3, the CSC greases had very similar WBL performance to that of the LiCx greases. Therefore, it can be deduced that the use of the primary and secondary antioxidant combination is also an effective way to prolong the WBL of the experimental CSC base grease. On the other hand, PDSC data, also shown in Figure 3, did not detect the antioxidant synergy in the CSC grease although the synergy was evident in the LiCx grease. Specifically, the long OIT but short WBL of the CSC-2 grease suggested that the extended WBL of the CSC-3 grease could not entirely be attributed to oxidation stability.

To further investigate the role of oxidation resistance, the formulated greases were statically aged at same temperature and in the same bearing assemblies that are used in the WBL test. An aging time of 40 hours was selected since 4 of the 6 greases failed to surpass 40 hour of grease life in the WBL test. The aged greases were then analyzed for weight loss, PDSC OIT, intensity of the IR oxidation peak and changes in oscillatory shear rheology behaviour.

As per Table 3, weight loss data correlated well with the OIT data collected on the un-aged greases with the greases with the longest OIT’s (CSC-3 and LiCx-3) having the least weight loss. As with the PDSC data, weight loss data for the CSC greases did not relate well with WBL and the data implied that other factors besides weight loss impacted grease life. This conclusion specifically applied to the CSC-2 grease that tested well for oxidation stability and weight loss but had a short life in the WBL test.

Also in Table 3 is the OIT data for the greases after 40 hours of aging in static bearings. The data revealed that the antioxidant capacities of all the greases were significantly depleted, this included greases with the longest before aging OIT’s (CSC-2, CSC-3 and LiCx-3). These PDSC results were unexpected as they denote that static aging was more severe than the dynamic WBL test. Aging to 80 hours on the greases with longest WBL’s (CSC-3 and LiCx-3), further supported the severity of the static aging as these greases became hard, unpliable and impossible to completely remove from the bearings. Although no further aging experiments were conducted, the accelerated degradation of the greases under static conditions could be linked to the procedures used to remove rust preventive from the bearings. In the ASTM D3527 WBL test, the bearings are only washed several times with n-heptane while bearings used for the static aging studies were cleaned by the ASTM D1743 Bearing Rust Test procedure. The latter procedure included two washings with hot mineral spirits and wiping of the assemblies after each washing to assure complete removal of the rust preventive. After the hot mineral spirit washes, the bearings were also rinsed two times with 1% ammonium hydroxide in isopropyl alcohol/water solution. Therefore, it is possible that the more complete removal of the rust preventive from the bearings used in the static aging experiments led to steel corrosion that accelerated oxidation due to Fe ion catalyst.

FT-IR spectra for greases after 40 hours of static aging are shown in Figures 4 and 5. Figure 4 shows full IR scans for the LiCx greases and Figure 5 compares the oxidation peaks of the LiCx greases to those of the proprietary CSC greases. Figure 4 also includes a spectrum of LiCx grease that failed the WBL test at 120 hours. The purpose of this spectrum is to provide an example of grease with a high degree oxidation. As per Figures 4 and 5, the oxidation peak of the grease that was degraded for 120 hours in the WBL test was much more intense than the peaks of statically aged greases. This meant that of none the aged greases reached
a point of severe oxidation and complete antioxidant depletion although their PDSC OIT's at the 180 °C test temperature were not significant as per the ASTM D5483 test method. The oxidation peak data also correlated well with un-aged OIT's for both grease types. Specifically, the greases that gave the longest OIT's (CSC-3 and LiCx-3) were also least oxidized. However, it should be stated none of the statically aged greases were severely oxidized although static aging was more severe than the WBL test. This again inferred that grease life in the WBL test is not solely dependent on controlling oxidation.

Rheology data is summarized in Figures 6 and 7. Figure 6 provides yield points of the un-aged base greases, un-aged formulated greases and aged formulated greases. A comparison of the un-aged greases revealed that the CSC grease produced higher yield points than the LiCx counterpart but was more affected by additives than the LiCx grease that gave stable yield points regardless of formulation. Although the yield points measured on the un-aged greases did not relate to WBL performance, the data provided evidence that additives affect the structural integrity of greases, especially at high temperature and high shear conditions. After aging, the CSC greases with poor WBL performance exhibited larger variability in yield points. Specifically, the CSC-1 grease lost resistance to flow and CSC-2 become much stiffer than all the other greases. Based on these results, it can be concluded that the poor WBL performance for the CSC-1 grease was due to a lack of thermal and shear stability. CSC-1 also showed the largest weight loss, another sign of structural instability. In the case of the CSC-2 grease, the poor WBL performance corresponded to a loss of fluidity that decreased the ability of grease to flow into bearing contact zones. The yield point data for the LiCx greases was not as conclusive with the exception of the LiCx-2 grease. As was the case with the CSC-1 grease, the LiCx-2 grease also lost its resistance to flow with aging, an indication of reduced thermal and mechanical stability that in turn leads to poor WBL performance. Modulus at flow point data is given in Figure 7. A comparison of data collected on the un-aged greases that included unformulated base greases reveals that the LiCx grease produced higher and more stable flow points than its CSC counterpart. Although higher and more stable flow points for the un-aged greases was not a predictor of WBL performance, the data implied that the LiCx greases were more shear stable over a wider range of shear stresses and were less likely to bleed out of the bearings. After aging, the key observation was an intermediate increase in the flow points of the two greases with longest WBL's (CSC-3 and LiCx-3). This behavior indicated that these greases had the ability to flow into the contact zone without an excessive rate of bleeding that might lead to premature bearing starvation and failure. It should also be noted that these greases produced the least amount of weight loss and were the least oxidized. Therefore, the increases in flow points that occurred with aging were not completely attributed to these degradation factors. A possible explanation for this unique behavior is that thickener network was strengthened by interactions with the additives. As for CSC-1, LiCx-1 and LiCx-2, their flow points did not change with aging despite significant amounts of weight loss that should have increased their thickener content and consistency. This response suggested a lack of structural stability that caused faster bleed rates and early bearing starvation. Finally, the flow point of the CSC-2 grease, like its yield point, showed that this grease became significantly stiffer than all the other greases. The loss of fluidity decreased the ability of the CSC-2 grease to flow into and adequately replenish bearing contact zones with lubricant, thus, explaining the short WBL of this particular grease.
Summary

The study demonstrated the importance of additives in determining the high temperature performance of greases regardless of thickener type. Specifically, it showed that additives influenced both oxidation and shear stabilities of greases, two critical properties in determining grease life in high temperature wheel bearings. In addition, the study showed that the effect of additives on the high temperature shear stability of grease was measurable by a combination of static aging and oscillatory shear rheology experiments.

References


4. NLGI Grease Production Survey Report, 2004

5. NLGI Grease Production Survey Report, 2007


Abstract:
Lubricating greases have invariably been used for the lubrication of machinery in almost all types of industrial and automotive applications since hoary past. It is believed that the majority of roller bearings used are grease lubricated. Prior to industrial revolution, processing and applications of lubricating greases were crude and undocumented. However, since then a great deal of advancement has taken place in equipment design, technical innovations and new product developments, thereby posing stringent application requirements. There is no universal grease for multiple applications, rather specific grease for specific application purposes have been developed. Now the important question lies, what are prevailing practices and yardsticks to measure the quality of the grease from the user stand point?

Technically, the criteria for selection of a grease for particular application should have been based on OEM recommended specification/industry specification, for example NLGI GC-LB where ASTM, DIN, IP test requirements are listed. Our experience on judging the quality of grease by end users has often been different. The primitive way of screening the quality of greases based on human sense organs like smell, touch (feel) and sight (color) still exists and perhaps are the initial indicators to judge the quality of greases. It is prevalent belief that these touch/feel, color, smell tests might have been used to screen the grease in those old days probably due to lack of analytical capabilities. However, surprisingly these practices are still prevailing in this modern world. Based on our last five years of customer complaints data, it is evident that the majority of these complaints are attributed due to either packaging, color, tack (appearance) and/or smell. The testing of these greases indicated that product was actually meeting the targeted specification in most cases and there were no issues from application stand point but were questioned/ rejected unfortunately on these primitive grounds.

It is generally perceived that if the grease color is red, it is good for high temperatures; green color is symbolic of green environment , white is depictive for food machinery, black for mining/heavy duty, smell of sulphur indicates good extreme pressure (EP) properties and tacky grease will stick better in bearings and will perform better in wet environment. Therefore, in this paper the effect of color and tackifiers on the physico-chemical and performance characteristics of lubricating greases has been presented.

1.0 Introduction:
The history of lubricating greases dates back to Egyptian times where lubricant was found in hub of wagon from the tomb of Tut-ankh-amun around 1340 BC. Analysis of the hub of the 1450 BC Egyptian chariot indicated the presence of quartz, iron, fat and lime, a kind of lime/calcium base grease (1). From then till the discovery of crude oil in 19th century, progress in the field of lubricating greases has not been well documented. It is assumed that the methods of preparations and application of lubricating greases in those days would have been crude and primitive. In context of evaluating and testing, lubricating greases would have possibly been judged based on feel, touch or seldom taste, presumably due to lack to testing methodologies (2). Nevertheless, in the last couple of centuries, especially in the last century, there has been phenomenal progress in the field of lubricating grease development where many different types of greases viz., lithium / lithium complex, aluminum complex, calcium sulfonate, barium complex, polyurea etc. emerged in the market. From this different industry specific specifications like NLGI GC-LB for automotive applications, AAR M 942 for railways, country specific especially defense specifications like MIL, DEF Stan, GOST, DIN etc. came into existence. Various organizations like ASTM, IP, DIN, GOST etc. developed various standard test methods to test variety of grease properties such as consistency, water resistance, extreme-pressure, high and low temperature, anti-wear and oxidation stability etc.

However, lubricating greases are surprisingly still judged by appearance, feel and odor, though these have little influence on service or quality of lubricating greases. On the other hand the properties that do have influence on actual application such as consistency/mechanical stability indicating the thickness and stability (tested by ASTM D 217 method), extreme pressure and anti-wear properties (ASTM D 2596 & D 2266) indicating load carrying capacity of greases, water resistance (ASTM D
indicating the performance of greases under water ingress conditions, oxidation stability (ASTM D 942) and high temperature life (ASTM D 3527/3336, FE 8/FE 9) indicating thermal degradation, potential life of grease at elevated temperatures and get side lined sometimes due to these so called screening tests. On the other hand, a careful examination of some of the popular industry specifications like NLGI GC-LB and AAR M 942(3, 4) indicate that these appearance/perceptual based tests are not the part of these popular specifications.

It is difficult for the end user to test grease for all these above stated properties due to lack of test facilities where sometimes they have to rely on the data provided by a supplier. It has been experienced that the decision to use a particular grease is often influenced by feel, appearance, color and tack. There have been different criteria/practices adopted in different parts of the world depending upon their local conditions. For example, in India majority of industrial greases are without dye. As there are some spurious type of products in the market where low quality re-cycled oil is sometimes used to make grease that has a color. The logic is if the grease is colored, it may sometimes be perceived that color has been added to hide something and if grease is without dye, it is perceived that the grease is made with fresh/virgin oil. Similarly, if the color of the grease is on darker side, it is expected that grease possesses better extreme pressure (EP) properties to it. It is assumed that similar practices may be prevailing in other part of Asian countries and other markets. On the other hand, in North America, it has been observed that the greases are mostly dyed/colored and contain tackifier. Tackier grease is expected to be better by possessing better water resistance, better adherence on bearing surface.

From these conclusions different demo techniques have been developed by some marketers like 'Rat Trap' / 'Hammer Test' (Fig-1, 2) demonstrating the extent of tackiness/adherence of the grease.

We collected some data on our customer complaints for the last 5 years and categorized in color, tack, smell/odor, consistency/thickness, running out of bearing, inferior water washout, low extreme pressure properties, poor high and low temperature properties, etc. The data is compiled in Table-1. Surprisingly, this data indicate that the majority of the complaints we encountered are either on variation in color, odor, tackiness and softer/harder grease compared to actual performance related parameters (Fig-3-5). Once we tested the retain grease samples for its actual performance properties we found that majority of the grease samples met the requisite performance characteristics EP-AW, water washout/spray off, drop point, penetrations, rust etc. In general based on the customer base we handle, it has been very interesting to note that in mature market like North America, the quality of lubricating greases is still preferably judged by such primitive ways based on perceptive sense organs like color (eye), tackiness/texture (feel) and/or smell. These interesting observations prompted us to systematically study the effect of these various parameters on the performance characteristics of lubricating greases and find the answer whether or not, are we trying to lubricate bearings effectively or just lubricating our senses? In this paper, some interesting results have been discussed.
2.0 Experimental:

For making the samples, base grease has been primarily taken from plant production batches which have been manufactured as per existing proprietary composition, processes and finished in lab with addition of different colors, additives, tack with mixing performed in a Howard mixer.

This was followed by milling under warm conditions. All the lubricating greases discussed in Table-2 are lithium 12hydroxy stearate greases and lithium complex greases and were made in mineral oil having a combination of naphthenic and paraffinic oils whereas aluminum complex greases and calcium sulfonate greases were made in commercially available white mineral oil. All four greases used in this study do not contain additives. In Table-3, Calcium sulfonate, lithium 12-hydroxy stearate and lithium complex greases used in this study are plant produced batches based on mineral oil and these samples were mixed with asphalt (in percentage as indicated in Table-3) in Howard mixer followed by milling the sample. Similarly MoS2 and Graphite mixer (50:50) were mixed in plant produced lithium 12 hydroxy and lithium complex greases. After mixing for 30 minutes to 1 hrs at about 120-140 OF the samples were milled. All the grease samples have been tested as per ASTM test methods. The polymers studied under these investigations are Polyisobutylene (PIB) with average molecular weight 1300 Mn, Olefin copolymers (OCP) with dilution in oil of at least 1:10 ratio, polymeric latex with solid content of 62-63 % having brook field viscosity at spindle # 4 at 6 rpm of + 450 CPS.

3.0 Results and Discussion:

The studies conducted here are sub-categorized in two main parts; first with effect of color on performance of lubricating greases and second with effect of different polymers on the properties of greases.

3.1 Effect of Colors on the Performance of Lubricating Greases:

Life without colors is meaningless, as colors do play a very significant role in our lives, and therefore presumably have been an important part of lubricating greases. At a market place, one can find a variety of colored greases right from white to black, blue, red, green, purple, translucent, yellow/golden, with sparkles etc. There is varied opinion or perception about the colors of greases. As per Machinery Lubrication article (5), colors are being used by manufacturers to facilitate the identification of greases and make them more appealing. From user standpoint, this can help spot, if they are using the correct grease or not. Yulia-Sopina (6) blogs that while color could be significant what really matters to end users is the performance of grease in actual application. She adds that right color makes grease more attractive and can sometime be used by marketing to differentiate their product with competitors. As per Truckinginfo.com blog (7), color essentially has a cosmetic effect barring in some cases where it helps maintenance to identify that if a component is at all being lubricated or not.

To be more specific on colors, white greases are indicative of greases for food machinery and black greases are moly/graphite greases for severe operating conditions. Though there is no hard and fast rule, red color could be indicative of grease for high temperatures, blue color for cold temperatures and green for environment friendliness.

However in order to evaluate the effect of these colors on the performance characteristics of greases, we have systematically tested the different greases for different properties and subdivided greases in to following sub-categories

3.1.1 Effect of Grease Whiteners on the performance characteristics of greases:

White greases are regularly being used in food machinery applications and also in non-food machinery applications. Among non-food grade white greases are white lithium and lithium complex greases. The popular white food grade greases are based on aluminum complex, calcium, and calcium sulfonate or clay base greases. To make color of the grease white most manufacturers commonly used whiteners such as titanium dioxide (TiO2) and zinc oxide (ZnO) which are generally used as solid powders. Amount of these compounds in lubricating greases may vary from 0.5-5 % or even more depending upon the extent and other ingredients used to make base greases.

In our study we have tried to use different percentages of TiO2 and/or ZnO in lithium, lithium complex, aluminum complex, calcium sulfonate greases and effect of these whiteners on the drop point, penetration, mechanical stability, anti-wear, storage hardening, and oil separation have been studied and results have been complied in Table-2. Table-2 indicates that addition of titanium dioxide and zinc oxide does not have any significant effect on drop point within the limits of repeatability of test method. Addition of TiO2 and ZnO in 2 % concentration did not show any noticeable difference on penetration however in 5 % concentration, it had indicated softening in penetration from worked penetration of 280 without whiteners to 296. There was no noticeable change in penetration numbers of lithium complex, aluminum complex, calcium sulfonate greases and effect of these whiteners on the drop point, penetration, mechanical stability, anti-wear, storage hardening, and oil separation have been studied and results have been complied in Table-2. Table-2 indicates that addition of titanium dioxide and zinc oxide does not have any significant effect on drop point within the limits of repeatability of test method. Addition of TiO2 and ZnO in 2 % concentration did not show any noticeable difference on penetration however in 5 % concentration, it had indicated softening in penetration from worked penetration of 280 without whiteners to 296. There was no noticeable change in penetration numbers of lithium complex, aluminum complex, calcium sulfonate greases and effect of these whiteners on the drop point, penetration, mechanical stability, anti-wear, storage hardening, and oil separation have been studied and results have been complied in Table-2. Table-2 indicates that addition of titanium dioxide and zinc oxide does not have any significant effect on drop point within the limits of repeatability of test method. Addition of TiO2 and ZnO in 2 % concentration did not show any noticeable difference on penetration however in 5 % concentration, it had indicated softening in penetration from worked penetration of 280 without whiteners to 296. There was no noticeable change in penetration numbers of lithium complex, aluminum complex, calcium sulfonate grease. In terms of mechanical stability tested as penetration after 10,000 double strokes, it indicated that the change in penetration from worked (60 double strokes) to penetration after 10,000 strokes is more when these whiteners are added either in 2 % or 5 % concentrations specially in lithium and lithium complex and aluminum complex greases whereas no significant impact was observed on calcium sulfonate greases. In terms of wear measured by wear scar diameter (WSD), incorporation of TiO2 and ZnO increased the WSD of all the 4 greases under test (Table-2). Oil separation or oil bleed in greases on account of adding TiO2 and ZnO was tested as per ASTM D 6184. Results in Table-2 indicate that there is an increase in oil separation in lithium and lithium complex greases. Oil separation appears more
pronounced in aluminum complex greases where as it does not have much impact on calcium sulfonate greases.

3.1.2 Effect of Black (Asphalt/MoS2/Graphite) on Properties of Lubricating Greases:

Black lubricating greases have been quite popular for a long time and are still being used profusely in the industry. The additives responsible to make grease black are MoS2, graphite, asphalt and carbon black. Asphaltic greases have traditionally been used in open greases and other bearing application and are considered to provide better adherence to metal surfaces and protect metal surfaces. On the other hand asphalt uses in greases are being discouraged due to environmental reasons and the asphalt loses its adherence when it comes in contact with water (8). Molybdenum disulfide (MoS2) and graphite are probably the most common solid lubricants used in industry to enhance the properties of lubricating greases especially in heavy and shock load condition. All three of these additives namely asphalt, MoS2 and graphite impart black color to the greases. In this paper the effect of asphalt on calcium sulfonate, MoS2 -graphite blend (50:50) on penetration, drop point, weld load, copper corrosion and oil separation has been studied with the results tabulated in Table-3. Asphalt in calcium sulfonate grease did not affect drop point and weld load adversely, but indicated some negative effect on penetration softening, slight deterioration on copper corrosion and increased oil separation in the treat rate of 5 % and 10 %. Similarly addition of MoS2 and graphite in lithium and lithium complex greases did not affect the drop point, penetration and copper corrosion considerably, but slightly deteriorated oil separation whereas significantly improved weld load, as expected. Slightly higher oil separation in presence of Moly or graphite may be attributed due to solid natures of these two compounds as grease in general have more bleeding tendency in presence of solids.

3.1.3: Effect of Sunlight, Heat and Water on the Color of Lubricating Greases:

It is believed that color does have a cosmetic value and adds to the aesthetics rating, however considering the environment that greases is normally subjected to such as sunlight and heat during operations and exposure to water due to application requirements or accidently due to rain when containers are not stored in ideal conditions. In these scenarios, it has been experienced that when grease is exposed to light intentionally or accidently, its color starts fading up and does not carry the same visual appeal as it did at the time of manufacturing / packaging (Fig-6). Similarly, when grease is exposed to heat / high temperatures either it gets dark or changes color to a dull/ light shade (Fig 7). In actual applications, greases are subjected to temperatures from ambient to + 450 0F , like in automotive wheel bearing applications, temperatures of the bearing could possibly be in range or + 250 0F and in steel mill it could go even beyond + 450 0F in extreme conditions . It has been our experience during manufacturing of greases and also during application that, color actually starts deteriorating after 200 0F and tend to disappear beyond 300 0F. Additionally in actual application, color of grease whether red, blue or green tends to darken. After some time it is hard to find the original color of the grease. In presence of water, it does not do well either. In Fig-8 red grease was floated on water for 24 hrs which not only emulsified but also changed its color from red to orange. Therefore from actual application point of view, color of the greases hardly adds any value to the overall performance of the greases.

3.1.4: Effect of Liquid Dyes on The properties of Lubricating Greases:

Though the intricacies of dyes used in greases are not well known as these are proprietary to the suppliers, however, dyes used in lubricants are based, in general, on azo and/or anthraquinone. These dyes are used in greases in extremely small proportions and should not be a concern in totality, however these classes of dyes potentially pose environmental and health concerns (9, 10). In general, red, blue, green, purple, black, yellow etc., dyes are used to color the greases. The concentration of dye may vary depending on the intensity of color needed by the customer and may vary, in general, from 0.001 % to 0.5 % or even more. In order to study the effect of dyes on properties of lubricating greases, varied amount of different color of dyes have been added to the greases and tested for various properties (Table-4). As per Table-4, different colored greases were tested for penetration drop point and copper corrosion. As per these
studies, color of dye does not affect penetration, and drop point, however it does affect copper corrosion adversely at higher concentration of 0.1 % or beyond. This gives indication that if we use dye in higher concentrations it might adversely impact the corrosion resistance properties of the greases.

3.0 Effect of Polymers/Tackifiers on Different Properties of Lubricating Greases:

3.1 Effect of polymers on Lithium, Lithium Complex and Calcium Sulfonate Greases:

The effect of polymers/tackifiers/structure modifiers on the properties of lubricating greases has extensively been reported (11-13), however how much of these polymers affect the properties of lubricating greases favorably and how much they effect adversely has not been reported to a significant extent. The literature survey indicate that both cohesion and adhesion phenomena work simultaneously when polymers are added to improve the properties of greases. The adhesiveness in greases, by large, refers to the extent of grease adhering to metal surface whereas cohesiveness provides grease the string forming tendency of lubricating greases. The polymers are incorporated in grease to improve the consistency, shear stability, water resistance, and adhesion and tack properties. On the other hand polymers may adversely affect the cold temperature and flowability properties of lubricating greases. The extent of improvement greatly depends on the chemistry of polymers and the quantity added. Some polymers enhance adhesion more than cohesion or vice versa (12). It is not always the case that polymers are supposed to be added in lubricating greases. There are some specification like AAM 942 where use of any VI improvers is prohibited (4). In general the type of polymers used in lubricating greases are ethylene-propylene, Olefins copolymers (OCP), styrene-ethylene-butylene (SEB), poly isobutylene (PIB), polymethylacrylates (PMA), natural rubber/latex etc. There are various tests method and practices available in the market to measure the adhesiveness, however most of them are used in adhesive industry and are not suitable to that extent in grease industry. Touching the grease between thumbs and pulling by finger is probably the oldest and still prevailing practice to test the tackiness of greases (Fig-9, 10). Different demo techniques like hammer, rat-trap tests (Fig-1, 2) etc. are being commonly used by grease marketers to demonstrate the tackiness capabilities of lubricating greases.

In this paper, effort has been made to study the effect of different type of polymers in various type of thickeners on their mechanical stability, storage stability, water resistance and low temperature flowability. In Table-5, different polymers viz., PIB, OCP, ethylene-propylene, polymeric latex in lithium 12-hydroxy stearate (lith 12), lithium complex, and calcium sulfonate complex have been studied. All these three greases contain conventional EP-AW, anti-oxidant and rust inhibitors but no other additives except as stated in the Table-5. In lithium 12-hydroxy stearate grease (lith-12) all the polymers slightly improve the dropping point of greases and also penetration to a harder side but there is no significant impact of mechanical stability as depicted by 10,000 strokes penetration (10 K Pen). Interestingly, the penetration after 6 month of storage with PIB was not affected significantly whereas the greases with other two polymers i.e., ethylene-propylene and latex were significantly softened. The reason for this softening though not completely investigated appears that the polymers at the time of manufacturing through the process of milling hardens the grease initially when grease is warm. Once the grease is cooled down enough, it starts losing it’s affinity to stay thick. There could also be other possibilities like homogenizer pressure / milling gap, temperature and time of mixing or stage of addition of polymers. Roll stability in presence of 10 % water and water spray off properties were found to be improved by PIB and ethylene-propylene co-polymer but deteriorated by polymeric latex. The possible reason for this unexpected behavior with latex is that with higher concentration of polymer, grease becomes too tacky and it’s hard to stick the grease on test plate. Due to poor adherence of grease on plate, similar complaints have also been reported from field where grease due to very high tackiness did not stick to metal surface (Fig-11).

3.2: Effect of Polymers on Aluminum Complex and Clay Base
Greases:
Effect of OCP and polymeric latex on the properties of aluminum complex both in mineral oil and made in vegetable oil has been studied. Similarly effect of ethylene-propylene co-polymer and polymeric latex has also been studied in clay base greases. Some interesting results have been tabulated in Table-6. Table-6 indicate that aluminum complex grease both in mineral and vegetable oil do not have appreciable effect on drop point. This however shows harder penetration by incorporation either of OCP or latex polymer, where a similar effect is seen in clay base greases as well. However both mineral oil and vegetable oil base greases exhibited more hardening on storage as tested by worked penetration after 6 months than the grease without polymer. Polymer in general improved the water resistance properties as indicated by roll stability in presence of water, but grease with 2.0 % latex showed poor resistance compare to 1.0 % latex; probably because latex makes grease too stringy. In clay base greases, drop points were not measured as they are considered non-melt greases. Incorporation of OCP and latex in clay base grease resulted in harder grease with polymer. After storage, like Aluminum complex grease, this greases also showed some hardening tendency but not to the extent of aluminum complex grease. There was no noticeable change on the water resistance properties of Clay base grease with or without polymer.

3.3: Effect of Polymers on Pumpability of lithium 12 Hydroxy, Lithium Complex and Synthetic Calcium Sulfonate Greases:
In previous section we have studied the effect of different polymers on the properties of lubricating greases. By and large polymers improve the thickening capabilities of lubricating greases and also water resistance properties. In this section, attempts have been made to study the effect of polymers on the pumpability at various temperatures. Data has been tabulated in Table-7. Lithium 12 hydroxy grease ( lith 12) used for these studies was a mineral oil based grease in VG 220 base oil, in NLGI # 2 consistency and contain EP-AW, anti-oxidant and rust inhibitors. The Pumpability of various samples was tested as per US mobility test. Pumpability test results in lith 12 grease indicate that there is no significant effect on pumpability at 77 0F with or without polymers, but at lower temperatures, 0 0F and -20 0F, the pumpability capability with polymers was significantly reduced and found to be dependent on amount of polymer added. The similar results were found with lithium complex greases. Synthetic calcium sulfonate grease was prepared in PAO and was in NLGI # 2 consistency grade. Pumpability was tested at 0 0F, -20 0F and at -40 0F. The results in Table-7 indicate that pumpability is drastically reduced at lower temperatures with the greases containing polymers.

4.0 Conclusions:
There have been different kinds of standard and sophisticated test techniques available in the industry to test the quality of lubricating greases, however the practices like smell, touch to test tack and color primarily for cosmetic reason are prevalent in the industry. There have been different reasons and justification for all those non-technical screening methods. However our studies has indicated that color does not add value to any property in lubricating greases, rather dyes deteriorate some properties like copper corrosion. Addition of TiO2 and ZnO that impart white color to greases, do not add any value to any of its properties but may increase wear and oil bleeding when used in higher dosages. Tack does improve certain properties of greases like water resistance, yield and oil bleeding, however excess amount may be counterproductive as its adherence power weakens and cohesion start predominating. It has been observed that certain amount of polymer does increase the hardening tendency of aluminum complex grease especially made with vegetable oil. Similarly, lubricating greases with polymers have indicated comparatively inferior pumptability/mobility especially at lower temperatures.

5.0 References:
2. CJ Boner , ”Modern lubricating greases” 2.1
1. Abstract

Lubricating greases are widely applied in rolling bearings. Their main purpose is the separation of the contacting surfaces during bearing operation, thereby improving bearing life. Grease lubrication also provides inherent sealing, corrosion protection, and friction reduction. Limitations of grease lubrication include deterioration of lubrication conditions over time due to, e.g., grease hardening, aging, and depletion of oil from grease fractions stored close to the rolling contact. In many instances, bearing performance also suffers from a relatively unfavorable distribution of grease inside the bearing. The current study presents novel greases based on a mixed thickener system, showing a potential for improving grease characteristics and performance properties relevant for bearing applications. Grease candidates containing the mixed thickener were manufactured on laboratory scale, using a process of reduced complexity and lower energy requirements. The greases were highly stable and showed good oil separation properties. Excellent corrosion resistance was obtained at low additive concentration. Bearing studies showed improved lubricant film formation, friction reduction, and lower self-induced temperatures compared to reference greases tested. The mixed thickener containing grease, showing reduced friction and better film formation, provides a potential for longer relubrication intervals and improved bearing service life.

2. Introduction:

Lubricating greases are semi-solid product containing a thickening agent and base oil. Grease also contains various performance enhancing additives. The type and concentration of additives depends on its field of application. Greases are the most common lubricants in bearings. Bearings are important mechanical components involved in the rotating parts of all the equipment utilized in different fields the automotive and railway industries, machine tools, aviation, marine industries, off highway equipment, paper mills, cement industries and many more. Oil lubrication is better understood in rolling bearings compared to grease lubrication in similar types of tribological systems, although greases are the most preferred lubricants for rolling bearings. Greases are important to rolling bearings for the purpose of reliable operation, low friction and longer life. The primary reason for using lubricating grease is to prevent the lubricant from flowing out of the bearing systems. Along with providing a basic lubricant functionality, lubricating greases can provide a good sealing effect, thereby avoiding external contamination of the rolling bearing system. Under conditions of insufficient lubrication there is no effective separation of the contacting rolling elements, increasing surface distress and the risk of bearing failure. The primary function of any lubricating grease is therefore to avoid stresses through the formation of a low-friction lubricating film in the rolling contact. Avoiding wear and corrosion of the rolling element surfaces is a second important functionality. The most widely used mechanism to describe grease lubrication in rolling bearings is from Booser and Wilcock. They postulated that the grease acts as an oil reservoir where the oil is slowly released into the contacting surface of the rolling bearings. Over the time period of application the lubrication conditions in rolling bearings deteriorate due to grease hardening, aging and oil depletion from grease fractions stored close to the rolling contact. Due to the presence of churning inside the rolling bearings, the grease is pushed out of the contacting surfaces into the edges of the bearings. In many instances bearings suffers from a relatively unfavorable grease distribution over a time period. Greases are sensitive to physical and chemical aging, which is generally an irreversible process. Due to the aging or hardening, replenishing greases by relubrication can be difficult. Along with inherent limitations of greases in the rolling bearings, the grease manufacturing process itself is a complex and energy intensive process. Performance reliability and product quality requires extensive experience and in
depth knowledge of grease processing technology. The most commonly used greases in rolling bearings are lithium-based greases. Hence, the largest fraction of grease produced by manufacturers consists of lithium-based greases. The cost of lithium hydroxide, the principle raw material source for production of lithium-based greases, has increased threefold in the recent months and is expected to increase further in near future (11). There is a growing shortage of lithium hydroxide in the global market due to the rapidly increasing demand for lithium in manufacturing of Lithium batteries, e.g., for hybrid cars (12). Many of the grease manufacturers and their end-users are looking for alternative grease products to lower costs, while retaining equal or superior performance of the lithium greases. In general greases may be considered as gel- or sol-based colloidal solutions. Gels are made up of a dispersing medium dispersed in the dispersing phase. The principles of grease as a product is similar to sols where a thickening agent or thickeners acts as the dispersing phase and the base oil acts as the dispersing medium. There are many products available in the market, which are made up of thickening agents as dispersing phase and base oil or thinner as dispersing medium, for example candles, paints, paste, resin based adhesives etc.(13).

In the present study, greases are synthesized by utilizing the combination of two different polymers as the dispersing agents and traditional base oil as the dispersing medium. Hence, the polymer fraction thereby forms the grease thickener. The synthesized, finished products were tested for its grease properties, such as oil separation, corrosion resistance, film forming capability in the bearings, dropping point, load carrying ability and anti-wear properties. Performance-enhancing additives were used to improve grease properties, e.g., anti-corrosion properties.

3. Materials and Methods:

Polymers for thickening the greases were obtained commercially with purity level of more than 95 percentile. Synthesizing materials required for synthesis process such as glass reaction vessels, temperature controllers and quenching plate were obtained commercially.

3.1. Synthesis of Mixed Thickener containing grease: The total concentration of thickener was fixed to 19.2 weight percentile, in that 12.48 weight percentile was made up of polymer-I, which is an ester-terminated polymer. Polymer-II has two components, one is a homo polymer with concentration of 6.384 weight percentile and the second component is a copolymer with concentration of 0.336 weight percentile. Three different types of base oils were evaluated, having a viscosity of 65cSt of viscosity at 40°C, i.e., mineral oil, ester oil and Alkylated naphthalene oil were utilized. To enhance grease performance, 4 weight percentiles of additives was added. Synthesis of the grease was performed in a 1L glass reaction vessel under a flow of dry nitrogen. Polymer-I was charged into the vessel, which was connected with temperature controller, thermocouple, dry nitrogen gas and a motorized mechanical stirrer. The reaction vessel was heated to a temperature above the melting point of Polymer-I. Base oil was slowly added to the reaction vessel with constant stirring and keeping the temperature above than melting point of Polymer-I. The temperature of the reaction vessel was raised to 180±2°C, where after Polymer-II (containing of homo and copolymer) was added. After completely melting Polymer-II, the temperature was raised to 200±2°C and kept under constant stirring for about 30 minutes. Afterwards the entire reaction mixture was poured out onto a solid metal quenching plate at room temperature. Upon cooling the mixture solidified into a semi-solid state, was scooped from the plate, and stored at room temperature. The above process was repeated to obtain a batch grease quantity of three kilogram. Following are the details about different characterization tests utilized in the present work.

3.2. Dropping point of the grease: DIN ISO 2176 was used to determine the droppint point.

3.3. Mechanical Stability: 3.3.

(i) Work Penetration after 60 Strokes ASTM D217 method was followed to perform work penetration test after 60 strokes. Three determinations are made and average was taken as final penetration.

(ii). Roll stability test: Mechanical stability was determined using ASTM D1831. A grease sample of 50 g was subjected to mechanical shearing enclosed in a cylindrical metal container with internal roller. Testing was performed for 50 hours at 80°C. Afterwards the worked penetration was measured as per ASTM D217 method.

3.4. Four ball weld load: The maximum load carrying capacity of the greases was determined using a modified ASTM D2596 method. The test was performed at a rotating speed of 1450±50 rpm. Lubricating greases were brought to room temperature and then subjected to a series of tests of 60 seconds duration at increasing loads of 200N, until welding occurred.

3.5 Four ball wear scar: The anti-wear properties of different greases were evaluated using a modified ASTM D2266 method. Testing was performed at a load of 1400N for 60 seconds.

3.6 EMCOR- Anti corrosion Test: The DIN 51802 EMCOR method was used to evaluate the capability of grease to protect against corrosion when steel bearings in contact with distilled water and 3% salt water. The bearing type used was 1306K/236725 (special bearing with stamped steel cage). After
dismounting, outer ring was visually inspected for the presence of rust spots and rated according to the standards.

3.7 Oil Separation/ Bleeding rate: The DIN 51817 standard method was utilized to find the tendency of grease to separate the oil under stationary conditions.

3.8 Characterization of bearing performance: Bearing performance was evaluated using a specialized test rig referred to as the SKF vertical rig. The test rig consists of an axially loaded thrust TRB mounted between a stationary upper column and a rotating lower column. The rotational torque is measured on the upper column using a strain gauge. This provides an accurate measurement of the friction torque. The relative film thickness is monitored by measuring the capacitance over the rolling contact, using the SKF Lubcheck system. Self-induced temperature is measured at the TRB outer ring. Base greases were tested with standard SKF tapered roller bearings (TRB-30204J2/Q). Experiments were performed at a spindle speed of 1500 rpm, under an applied load corresponding to 1 GPa of maximum contact pressure, for a duration of 3 hours. The load is applied step-wise during the initial part of the test to enable proper running-in. The total amount of grease applied before starting of experiment to the bearing was 1.25g.

4. Results:
Compositions of grease with three different base oils, i.e., mineral oil, ester oil and Alkylated naphthalene oil were synthesized. The thickener consists of two different polymers. The process is simple and less energy consuming compared to the traditional grease synthesis process. In the first place, grease compositions are synthesized using a single heating step. Second, the synthesized grease does not require any processing or shearing following the cooling step. Generally, extensive processing is required during grease synthesis, to achieve the required level of homogeneity, grease consistency, and mechanical stability. The current grease compositions do not require such processing, as the proper grease consistency is obtained even without processing. In turn this allows the grease to distribute properly inside the bearing and bearing housing during application. The synthesized greases are sensitive to mechanical and thermal stress; grease fractions in the vicinity of the rolling contact will be very easy to work out once exposed to high-shear conditions. During application in bearings, under shear, the grease will soften close to the rolling contact, comparable to NLGI grade or 1 grade. The synthesis process of traditional greases such as, lithium-greases are laborious and require intensive energy input into the process. After the grease synthesis, post processing also requires energy to soften the greases to the required NLGI grade. Due to higher shear resistance of traditional greases, it requires an intense milling process to get proper mixing of additives and to obtain the smoother texture of the greases. The newly synthesized greases do not require high energy consuming milling process. The synthesized greases were characterized for its grease properties and for its bearing application. Commercially available Lithium soap based grease with mineral oil as base oil was considered as a reference sample for all the properties. Following are the test results for the characterization of newly synthesized greases.

4.1 Dropping point:
Dropping was tested as per DIN ISO 2176 standard. Dropping point of different greases are shown in the table-1. The mixed thickener grease with ester oil showed a higher dropping point compared to the mineral oil and alkylated naphthalene oil. The reference lithium grease showed the highest overall dropping point. Experimental results showed that dropping point is not only related to any one of the raw material of the composition, instead it is the synergistic combination of the raw materials. Although thickener of all three synthesized greases is similar, the difference in the base oil results in a difference in the dropping point. It appears that the synergistic interaction between the ester oil and thickener provides the higher dropping point. It was observed that, after finishing the dropping point test for reference grease, outer layer of the grease in the sample cup turned to brown and in many instances into the dark brown, which indicates the oxidation of the grease.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Dropping Point in °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed Thickener Grease with mineral oil</td>
<td>146</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Ester oil</td>
<td>179</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Alkylated naphthalene oil</td>
<td>157</td>
</tr>
<tr>
<td>Commercial Lithium grease with Mineral oil</td>
<td>185</td>
</tr>
</tbody>
</table>

Table-1: Dropping point of different greases

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Work 60 Strokes in mm/10</th>
<th>NLGI Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed Thickener Grease with mineral oil</td>
<td>215</td>
<td>3-4</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Ester oil</td>
<td>207</td>
<td>3-4</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Alkylated Naphthalene oil</td>
<td>178</td>
<td>4</td>
</tr>
<tr>
<td>Commercial Lithium grease with Mineral oil</td>
<td>240</td>
<td>3</td>
</tr>
</tbody>
</table>

Table-2: NLGI grade of different greases
4.2. Mechanical stability

Mechanical stability of the grease was measured by using grease penetrometer. There were two different methods were employed to test the mechanical stability of the synthesized grease and reference commercial grease.

(i) Work penetration after 60 strokes. Test grease samples were worked for 60 strokes with the standard manual grease worker (Table-2). After the 60 strokes test grease samples were measured for work penetration test as per standard ASTM D217 method.

(ii) Roll stability test: The mechanical stability was determined after 50 hours at 80°C using the ASTM D1831 method. The penetration results are show in in Table 3. Newly synthesized greases had low penetration values, corresponding to very high NLGI grades. In normal instances the higher NLGI grade greases are not recommended to use in the bearings. However, the new grease compositions show the ability of reversible softening under applied shear stress or thermal stress, and are therefore applicable even at a higher initial stiffness. Following completion of the roll stability test, the greases transformed into an (estimated) NLGI 0 or 1 grade, derived from visual observation. That is, immediately after testing the grease appearance was almost liquid. Surprisingly, after equilibrium at ambient conditions the grease properties instantly reversed, such that the greases hardened, enough to reach the consistency equivalent of NLGI 2 or 3 grades. This is clear indication of a thixotropic behavior. It is expected that this has a great impact under application conditions, where conditions of shear in the rolling bearing will result in grease fractions becoming softer, allowing entry into the narrow contact region to replenish the lubricant film. Thereby, the impact of the mixed thickener greases could result in enhanced lubricant functionality. It is expected that only during the actual shear process, softening occurs. As can be seen comparing table 2 and table 3, the initial consistency is not fully recovered after roll stability testing and equilibration. As the grease studied here have not been post-processed after synthesis, the increase in penetration could be a post-processing effect occurring during the roll stability test. Therefore, the greases are expected to be fully reversible in consistency should post-processing be applied.

4.3. Four Ball weld load:

Modified ASTM method D2596 was used to determine the weld load, measured at 1450 rpm for 60 seconds. The results are shown in the Table 4. Although all the greases had same type and concentration of thickener and additives but difference in base oil type and its interaction with the thickener and additives provided the ester oil containing Mixed Thickener grease better load carrying capability.

4.4. Four ball wear scar:

Modified ASTM method D2266 was utilized to test the four ball wear scar for the different test greases, at a normal load of 1400N for 60 seconds. The wear scar diameter was measured under the optical microscope, and is shown in Table 5.

4.5. EMCOR corrosion:

Shown in Table 6 is the EMCOR anti-corrosion performance of the greases with distilled water and 3w% salt water. All the greases showed excellent anti-corrosion properties with distilled water. Greases with 4wt% of additives (anticorrosion-2.5%, antioxidant-0.5% and anti wear-1%) were also tested for its anticorrosion properties with 3 Salt water. EMCOR test showed all the greases were able to provide good corrosion resistance in the presence of distilled water. Interestingly, the mixed thickener greases show a very typical phenomenon. As can be seen in Figure 1, the appearance of the grease is bright white after the corrosion test. The only reasonable explanation that can be given at this point is that the grease absorbs water during the course of testing. However, this has no impact on the good anticorrosion performance. It appears that the mixed thickener system is very effective in preventing corrosion in the presence of water, rather than by expelling water, as would...
be considered a favorable property for standard greases. It was observed that, little squeezing of grease expelled absorbed water. Polymer-I, as part of the mixed thickener system, has a relatively high polarity, e.g., compared to typical polymers based on polymerization of alpha-olefins. In addition, polymer-I is esterminated, allowing compatibility with lubricating base oils. The EMCOR observations shows that this leads to favorable grease properties. One could argue that the mixed thickener therefore represents a new mechanism for “waterresistance”.

4.6. Oil separation test:

DIN method 51817 was used to evaluate the tendency of the greases for oil separation can be seen that the overall oil separation is low, see Table 7 Whereas the oil separation of the reference lithium-based grease tested here is low, the oil separation of the mixed thickener greases is comparable, or even lower. Thus, oil separation is found to vary from 0.3wt% for the grease based on alkylated naphthalene, to 1.6wt% for the grease based on the ester oil. The low oil separation may be correlated to the relatively high stiffness of the greases, i.e. the high NLGI grade. It is fair however, to observe that the bleeding rate is very low when considering the thixotropic nature of the greases as discussed above.

4.7. Bearing testing

4.7.1. Grease Characterization with TRB (Tapered Roller Bearings)

Friction Torque of grease samples in TRB: No leakage of grease was observed during or after testing. Under the vertical arrangement of the test rig this indicates a stable grease distribution. Hence, grease leakage resulting from softening does not occur. During testing, the commercial grease showed consistently varying friction torque. For all greases the friction torque was highest initially, showing a peak value after applying the maximum load. This was followed by a relatively strong decrease in friction torque, reaching a plateau value after some 2 hours. In comparison, the mixed thickener grease with ester oil and mineral oil showed uniform friction torque throughout the test.

Self-induced temperature of grease samples in TRB: Following the behavior of the friction torque, the mixed thickener greases showed a lower self-induced temperature compared to the lithium-based grease. The mixed thickener grease containing the ester oil showed the lowest self-induced temperature.

Based on the self-induced temperature, the lubrication conditions may be estimated using kappa viscosity ratio. This represents the ratio of the actual viscosity in the bearing at a given temperature, over the required viscosity to achieve sufficient separation between the rolling surface elements. Thus, for the mixed thickener grease containing the ester oil, kappa = 1. For the Lithium grease kappa = 0.7, indicating a deterioration in lubrication conditions.

Lubricant film thickness of grease samples in TRB:

The Lubcheck signal represents a capacitance measurement over the rolling contacts of the rolling bearing. The capacitance will increase when a nonconducting lubricant film is present simultaneously in the rolling and sliding contact of the tapered rolling bearing. The Lubcheck signal is therefore a qualitative, or relative, indication of lubricant film-formation. Using the Lubcheck system the signal varies from 0 Volt (metal-metal contact) to 3 Volt (full separation).

In figure 4, it can be seen that in comparison, the mixed thickener grease containing the ester oil showed good film forming capability, whereas the commercial grease was least capable of film-formation. This is an indication that the mixed thickener plays an active role in enhancing the lubricant film, in combination with the type of base oil.
Overall, bearing characterization based on tapered rolling bearing testing suggests that the mixed thickener grease containing the ester oil shows a better performance compared to the other greases tested here, including the commercially available grease.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Oil bleeding rate in %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed Thickener Grease with mineral oil</td>
<td>1.1</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Estor oil</td>
<td>1.6</td>
</tr>
<tr>
<td>Mixed Thickener Grease with Alkylated naphthalene oil</td>
<td>0.3</td>
</tr>
<tr>
<td>Commercial Lithium grease with Mineral oil</td>
<td>0.8</td>
</tr>
</tbody>
</table>

Table 7: Oil bleeding rate of different greases (base greases)

After equilibration the consistency quickly recovered to that of NLGI 2-3 grade grease. Obviously, this type of beneficial behavior could not be observed for the commercial lithium-based grease.

Bearing testing showed that there was no grease leakage from the open, vertically arranged bearings, confirming a stable grease distribution. Under conditions of axial loading TRBs typically show a tendency for temperature and friction increase under non-optimized lubrication conditions. The mixed thickener greases showed stabilization of the self-induced temperature indicating the ability to lubricate both flange and rolling contact. In turn, this suggests that the grease softening is controlled and limited to churning and grease shearing only in the rolling contact (14). Friction torque during bearing testing showed that the grease containing the ester oil notably reduced friction, when compared to reference grease and mineral oil based mix thickener containing grease. This correlated well with the lowest selfinduced temperature found for the mixed thickener grease containing ester oil. A study conducted by Cousseau et. al. (15) under similar conditions showed that the type of the base oil defines the friction torque. They tested seven greases on an axially loaded thrust ball bearing and found that bearing friction was primarily function of base oil type and not much on the type of the thickener or additive package. In their study PAO showed lowest friction.

5. Discussion and Conclusion:
In this paper the synthesizing method and lubricant characterization for bearing application of mixed thickener containing greases were studied. The objective of the study was to develop grease technology with reduced complexity and energy consumption during manufacturing and processing. The study also compares the grease properties, and the lubrication performance in selective bearing testing, against that of commercial lithium grease. At present lithium greases are facing an increased cost scenario and reduced availability of raw material for its synthesis. Alternative, mixed-thickener containing greases were synthesized with three different base oils, i.e., mineral oil, ester oil and alkylated naphthalene oil. The mixed thickener greases synthesized had a consistency corresponding to an NLGI grade of 3 and 4. Important, during application under shear the greases showed a reversible softening. The softening effect reduced consistency only during a shearing process, resulting in a temporary decrease in consistency comparable to NLGI grade 0 or 1. This is a mechanism that is very beneficial for facilitating the lubrication process in the rolling contact of bearings.
torque and was followed by ester oil and then mineral oil. Similar results were also found by De-Laurentis et. al. (16) in a single contact based experiments. For the mixed thickener greases, the relative lubricant film thickness during testing showed that the ester oil containing grease developed a better film, which correlates its lower friction and self-induced temperature.

In terms of functional grease properties, EMCOR corrosion evaluation showed excellent anti-corrosion properties of the mixed thickener greases. It could be observed that a relatively large quantity of water was absorbed by the mixed thickener greases, nevertheless not resulting in any sign of corrosion on the raceway surface of the specimen. Interestingly, corrosion studies showed similar results between base grease and grease containing performance additives. This highlights the potential ability of mixed thickener to prevent the corrosion. The main problem with water in bearings is often associated with the formation of free water, causing corrosion and in addition, it may also induce starvation. Therefore, once water has entered the bearings, grease which can absorb large quantities of water may be preferred over water-repelling greases (17).

Considering the increased cost scenario of Lithium hydroxide at present, and the requirement to improve grease processing technology, there is a need for new grease technologies to having benefits over traditional grease products in both cost and technology of grease making. The present study presents grease based on mixed thickeners using novel polymers that may be formulated using different base oils. All three formulations presented here showed potential improvements in grease properties, whereby the ester oil based formulation showed better lubrication performance in bearing testing, compared to the commercial reference tested. An improvement in dropping point was achieved over the actual melting point of the polymers, whereby the ester oil containing grease even showed a dropping point comparable to the reference lithium-based grease.

6. Reference:

11. Asian Metal News, "China Lithium Hydroxide prices increase on tight supply", 2015 December
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