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Load Carrying Behavior of Greases Using Highly Refined Oil with Viscosity Modifier

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1.0 Introduction:

The core question in this study is: can the use of viscosity modifiers in light, highly refined oils replace the inherent load carrying performance that heavy Group I oils are known for? As crude oil is refined to Group I, Group II, and Group III quality levels there is a conversion of dense, incompressible hydrocarbon rings (naphthenes and aromatics) into flexible paraffins.[1] **Figure 1**, below, compares the transformation of rings to paraffins with increasing refining severity.

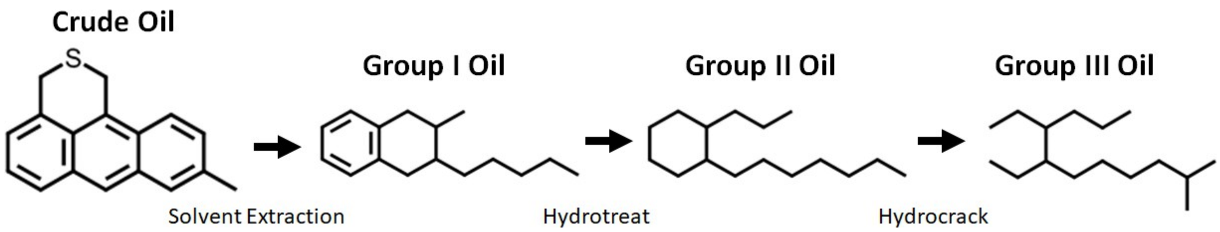

Figure 1: Idealized schematic of key differences in Group I-III lubricating oils

It is thought that these ring structures are critical in high load carrying capacity and is why Group I oils are the most favored oil for heavy duty applications. However, Group II-III or full synthetic PAO oils have become common in high performance EP lubricants due to their greater thermo-oxidative stability, low temperature fluidity, and improvements in EP additive package technology.[2] Greases remain slow to adopt viscosity modifiers.

The cost and available of Group II oil has outpaced Group I oil for much of the world in recent years.[3] Re-refined Group II-III oils are also gaining traction. Re-refined oils offer a cost effective and highly sustainable option in light of recent customer trends toward life cycle analysis, carbon footprint, and sustainability of petroleum.[4]

One property that higher refined base oils cannot compete with Group I on is base oil viscosity. **Figure 2** compares the viscosity of different oil types. Continued refined or re-refining strips out the cohesive attraction of aromatic carbons and waxes, and reduces molecular weight of hydrocarbons which results in a loss of viscosity. Group I oils are produced up to ISO 500 as bright stocks, Group II oils are produced up to ISO 110 as 600N, and Group III oils are produced to ISO 46. Re-refined oils are mainly available in ISO 10 to ISO 22 with limited options in the ISO 32 to 46 range. Lubricants use polymer viscosity modifiers to produce much of the final viscosity in applications including severe duty gear oils.[5]

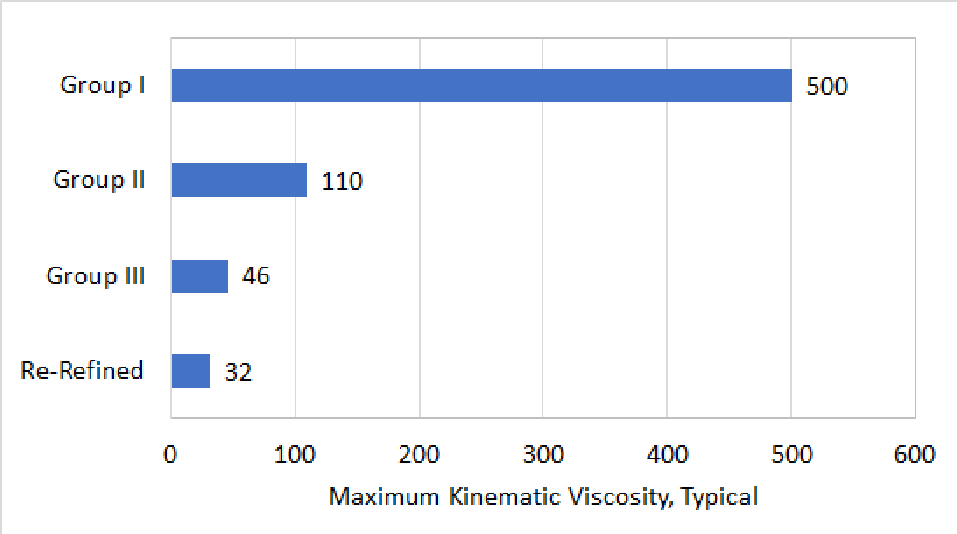

Figure 2: Typical viscosity ranges available for petroleum-based Group I-III and re-refined oils

The path forward in developing high viscosity products using Group II-III oil or re-refined oils is to find suitable ways to bring the base oil viscosity up to the proper ISO 220 – 460 viscosity range for an EP grease. An ISO 220 blend of bright stock and lighter Group I oil gets 100% of its viscosity from oil while an ISO 220 built from an ISO 25 re-refined oil gets only 11% of its viscosity from oil.

<u>2.0 Materials:</u>

2.1 Group I Reference Oil:

This study uses ISO 220 as the viscosity grade for each EP grease. ISO 220 Group I base fluid was prepared from a hydrotreated Group I bright stock (ISO 460) and a hydrotreated 150SN Group I oil in an 80:20 ratio by weight. Viscosities were 233 cSt at 40°C and 20.7 at 100°C; viscosity index was 104.

2.2 OCP Viscosity Modifiers:

Olefin copolymers ("OCP") are used to provide viscosity to a wide range of lubricants.[6] These materials are typically copolymers of ethylene and propylene, and may be supplied in bale, pellet, or liquid form.

The key selection factors for OCPs are ethylene content and shear stability index. Ethylene content determines the physical form and low temperature properties.[7] Shear stability index is a measure of molecular weight and degree of branching. SSI is measured by ASTM D6278 at 1wt% in oil.

The OCP viscosity modifiers used in this study were chosen for their amorphous quality and contain 40- 60% ethylene content which allows for very low crystallinity and high fluidity at ambient to low temperatures. **Table A** compares the OCP properties in solid form and at ISO 220 in 120N oil. Prior work shows this range will allow excellent low temperature grease mobility.[8] However, it should be cautioned that the excellent fluidity of highly amorphous polymer can invert at excessive treat rates due to the formation of a semi-dilute polymer network which induces structure and poor fluidity at low temperatures.[9] Moderation is key to all parts of formulating.

Table A: Properties of the raw olefin copolymer viscosity modifiers and their ISO 220 blends in 120N oil

	OCP-0	**OCP-1**	**OCP-2**	**OCP-22**	**OCP-35**	**OCP-45**
Form	Liquid	Liquid	Liquid	Bale	Bale	Bale
Shear Stability Index (SSI)	0	0.5	2	25	35	45
wt% in 120N for ISO 220	29.9	21.8	11.3	2.9	2.5	2.1
Kinematic Viscosity @ 40°C	223.5	233.4	231.5	222.4	224.3	228.2
Kinematic Viscosity @ 40°C	29.5	30.4	32.3	32.8	33.1	32.8
Viscosity Index	172	172	184	194	194	190

2.3 Group II Base Fluids with Viscosity Modifier

ISO 220 Group II base fluids were prepared using ISO 25 (120N) Group II+ re-refined oil. The oil was increased to ISO 220 using each of the OCP viscosity modifiers as shown above in Table Y.

2.4 Additives

The ashless EP gear oil package was based on sulfur/phosphorus chemistry and is suitable for industrial gear oils meeting US Steel 224 and AGMA 9005 at 2-3wt% in heavy paraffinic oils, and suitable for API GL-5 at 3.5-4.0wt% in multigrade automotive gear oils. The package passes ASTM D5182 FZG stage 12 and can produce EP weld loads up to 620 kg. The package contains 29.3% sulfur and 1.4% phosphorus.

Antioxidant was added during the high temperature processing of the pre-formed lithium 12-hydroxystearate thickener to preserve color and prevent unwanted oxidation of the base oils.

3.0 Methods:

3.1 Grease Production:

NLGI #2 simple lithium greases were prepared using pre-formed lithium 12-hydroxystearate thickener supplied in powder form. Lithium greases are the most popular grease type therefore the best system to demonstrate fundamental research.[10] **Table B** shows the typical final concentration of components in the #2 EP greases.

Table B: Typical formula for the #2 simple lithium EP grease

Component	**wt%**
Base Fluid	Balance
Pre-formed Lithium 12-Hydroxystearate	8 – 9%
Antioxidant (Phenolic/Aminic Mixture)	1%
Viscosity Modifier	1 – 30%
Ashless EP Gear Oil Package	3 – 8%

The lab-scale grease production followed an open kettle design and consisted of a Hobart C-100 mixer with 10L bowl, a 600W heating mantle, and two voltage regulators to control the mixing speed and heating rate of the system. Greases were milled twice using a Milwaukee 2646-20 M18 Grease Gun as a compact and efficient grease mill for small volumes of grease.

Greases were produced from starting batches of 1 kilogram using 87wt% base fluid (Group I, or Group II with no viscosity modifier), 12wt% hydroxystearate, and 1wt% antioxidant. The kettle was ramped to 180-190°C at 2°C/min until the lithium 12-hydroxystearate became molten. The heating mantle was removed and the batch was stirred without heat until cooled to 40°C. The grease was milled and left overnight before finishing.

NLGI #2 base greases for the EP study were made by adjusting from an initial #2.5 to #3 grease with additional base fluid. When using OCP viscosity modifier, the OCP was pre-dissolved in the adjusting oil at the correction concentration to give a final wt% treat rate in the Group II base oil to produce an ISO 220 viscosity.

Finished EP greases were made from the #2 base greases by adding the ashless EP gear oil package to the base grease in increments of 0.5wt% until a 400 kg weld load was achieved.

3.2 4-Ball Extreme Pressure Testing

Extreme pressure ("EP") weld load of the greases was measured per ASTM D2596 using a Roxanna ASTM D2596 4-ball EP test rig with dead weights. Scars less than 1 mm were measured by a Ducom TR-30L image acquisition microscope accessory. Scars greater than 1mm were measured by handheld optical scope.

The EP performance of the different OCP viscosity modifiers in Group II versus the Group I oil was benchmarked by the wt% of ashless EP gear oil package required to meet a 400 kg weld load. 400 kg weld load is the requirement for the new NLGI High Performance Multiuse grease specification's 'High Load' subcategory (HPM-HL). Package was added in increments of 0.5wt%.

Treat rate of the EP package was found to vary from 3wt% package to >8% package (where no 400 kg weld load was achieved). Base fluid preparations with inherently better load carrying properties would require less active sulfur and phosphorus from the EP gear oil package to meet the performance.

3.3 High Shear Rheology

High shear rate (12,000 s^{-1}) dynamic viscosity was measured by a BYK/Gardner CAP-1000+ rheometer per ASTM D4287 method modified for temperatures of 20 – 60°C using spindle #03.

<u>4.0 Results & Discussion:</u>

<u>EP Package Treat for 400 kg Weld Load in Grease</u>

This study compares the effectiveness of different base fluids using Group I or Group II with OCP by the amount of EP package required to meet a 400 kg weld load per the NLGI HPM-HL specification. The more common approach in similar studies is to use a fixed wt% of package. However, the EP package is rated to 620 kg so a formulator can set the EP weld load to any target value by adjusting the treat rate. Any deficiencies of the base fluid can be compensated by using more package and any enhancement in load carrying can be offset by using less package.

Figure 3 compares the different treat rates of EP package depending on the base fluid: either an ISO 220 monograde Group I, or Group II blends using ISO 25 re-refined Group II oil with OCP-0 through OCP-45 to reach ISO 220 viscosity grade.

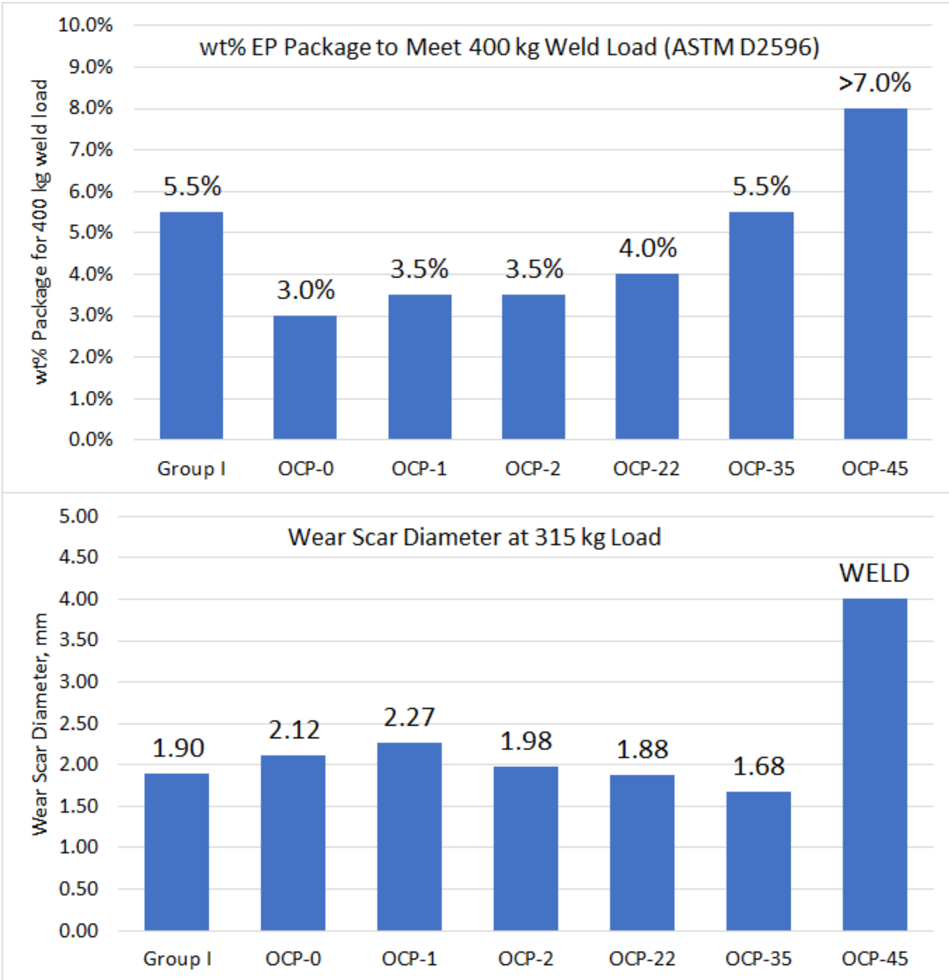

Figure 3: Comparison of wt% package to meet 400 kg weld load (top); WSD at one stage before weld, 315 kg (bottom)

The relative performance between the ISO 220 Group I monograde oil and the ISO 220 Group II olefin copolymer blends is surprising. ISO 220 OCP blends using the 35 SSI copolymer (OCP-35) required the same amount of EP gear oil package as the ISO 220 Group I oil to meet the 400 kg weld load for the NLGI HPM-HL specification. Olefin copolymers of 22 SSI or less (OCP-0 to OCP-22) required less EP package than the Group I oil – this implied that the OCP copolymers had inherently better capacity for EP performance and required less package to treat this deficiency.

Do Group I oils really have special load carrying capacity?

The popular understanding for Group I vs. Group II load carrying performance is that Group I oils contain higher molecular weights and more ring hydrocarbon structures. However, work on hydrocarbon compressibility by Cutler in 1958 shows that molecular weight and rings have little effect on compressibility to up ~1 GPa which is on the order of Hertzian contact pressures. Straight chain C18-C30 paraffins exhibited no changes in compressibility.[11]

Figure 4, below, compares the volumetric compression of different linear and branched alkanes, naphthenes, and aromatics at 135°C up to 1 GPa (10,000 atmospheres). Volumetric compression of low molecular weight isoparaffins, naphthenes, and aromatics show relatively similar compression curves.[12] Saturates are compressed by 25-27% at 1 GPa, branched naphthenes and aromatics by 23- 24%, linear bicyclic species by 21-22%, and a 4-cycle ring by 17%. Accurate crude oil compressibility curves show that behavior over 1 GPa pressure continues as expected.[13][14]

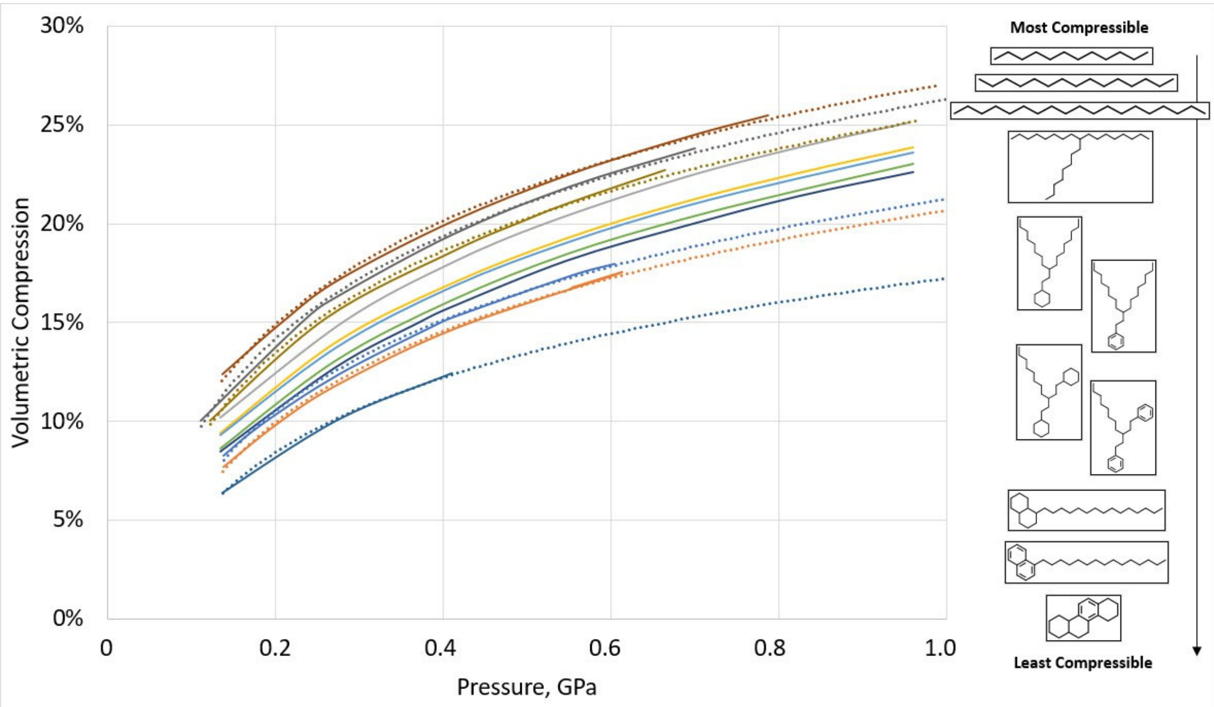

Figure 4: Compressibility data digitized from Cutler with chemical structures list on the right from most to least compressible [12]

Overall, the compressibility of branched hydrocarbons fall within 20-25%. The relative incompressibility of naphthenes and aromatics are not double that of alkanes. These fundamental studies in hydrocarbon compressibility support the findings that the Group II OCP blends could outperform the Group I oil blend on EP performance. There are notable differences in the compressibility of individual hydrocarbon species, but as mixtures Group I, II, and III oils can contain any combination of these components which will average out extreme differences.

There may be unconsidered tribochemistry that is not captured in the isothermal compressibility measurements. Thermo-oxidation of Group I and Group II oils in an EP test may yield different types of residues as barriers on the wearing metal surfaces.[2]

<u>Working viscosity under shear</u>

Why the re-refined Group II and OCP blends outperformed the Group I oil may involve the Stribeck curve and viscometrics. The parameters of the Hersey number – the x-axis of the Stribeck curve which separates the regimes of boundary, EHL, and mixed lubrication – are Speed, Viscosity, and Load.[15] The equation does not factor in tribochemistry from AW/EP additives or functionalized base stocks, but it is assumed to be useful in comparing similar formulas like the Group I and Group II greases in this study which vary only by the arrangement of hydrocarbons in the base fluid.

With this in mind, the dynamic viscosity of the ISO 220 Group I and ISO 220 Group II / OCP blends were investigated with a high shear rheometer (10^4 s^{-1}) over a range of temperatures to investigate what the effective viscosity term of the Hersey number would be.

Figure 5 compares the relative dynamic viscosity of the different base fluid blends at 40 and 60°C. The key observation is that the lower VI of Group I oil (VI 104) will increase the viscosity of the base oil relative to the higher VI (VI 170+) OCP blends. This will make the ISO 220 lower VI oil better at carrying load versus ISO 220 high VI formulas for temperatures < 40°C. At 60°C, the behavior inverts and the Group I is lower viscosity than the OCP-0, OCP-1, and OCP-2 blends due to their higher VI and will remain higher viscosity as the temperature continues to increase.

OCP-22 and above show a decrease in viscosity with increasing shear stability index (i.e. molecular weight) due to the temperature shear thinning effects of high speed shear which occurs in the 10^4 s^{-1} rheometer and high shear tribology testing like ASTM D2596 or ASTM D2266 4-ball test methods. This is most commonly exhibited in data from High Temperature High Shear (HTHS) testing in engine oils.[16][17]

For 22, 35, and 45 SSI OCP viscosity modifiers, the enhanced high temperature viscosity cannot compensate for the temporary loss of viscosity. Viscosity and therefore the load carrying performance as a matter of Hersey number under tribological conditions of high speed and shear are most favorable for the OCP-0, OCP-1, and OCP-2 (SSI value of $\leq$2). These OCPs also gave the best EP performance in terms of lowest required usage of EP package to meet the 400 kg weld load. The correlation of these points is likely not a coincidence.

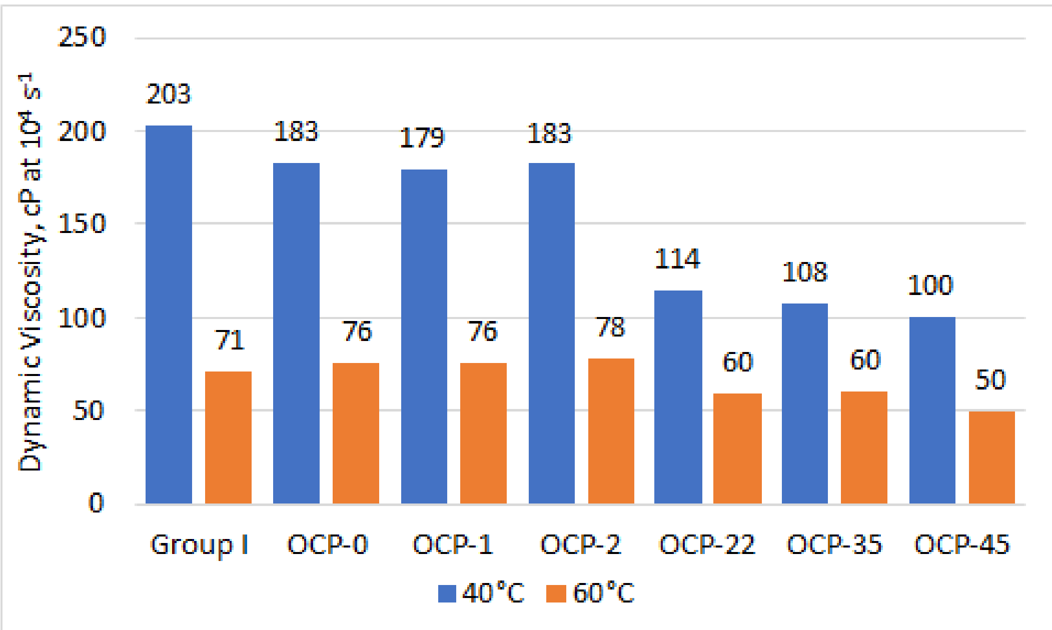

Figure 5: Dynamic viscosity of the Group I and OCP blends at high shear rate at 40°C and 60°C

5.0 Conclusions:

Heavy Group I base stocks like bright stock coming under scrutiny for sustainability and economy. Newer base oil technologies are able to produce highly refined oils at lighter viscosity grades. The required ISO 220 – 460 base oil can be reproduced using synthetic viscosity modifiers or base stocks to add viscosity. For shear stable viscosity modifiers, the viscosity added to a light ISO 25 re-refined oil is able to provide a better starting point for formulating EP greases meeting the NLGI HPM-HL specification. This may be due to the overvaluing of 'special' inherent load carrying properties in Group I and the undervaluing of the effect of viscosity index when oil is heated and under load in tribological conditions.

Future work entails investigating the effects of the OCP blend base fluid on the ASTM D226 4-ball wear prevention properties of the grease, the resistance to SRV wear, and prevention of fretting wear.

6.0 Acknowledgements:

Thank you to Bruce Neading for his technical expertise and generous time spent building the necessary improvements to the 1kg-scale grease production unit.

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Tailor-made Sulphonate Complex Grease with Improved Performance in Presence of Water

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ABSTRACT

Water resistance is a desirable characteristic in greases for many industrial applications especially which are exposed to water. Calcium sulphonate complex greases are commonly used in such humid environments and these greases get emulsified due to presence of water. In order to maintain structure and performance, sulphonate complex greases are required to have fine balance between water repellency and water tolerance properties. Over emphasis on water repellency can cause performance issues due presence of free water. Free water can result in a loss of the hydrodynamic oil film leading to excessive wear. Certain extreme pressure and anti-wear additives are readily hydrolyzed by water, resulting in both additive mortality as well as formation of acidic by-products. These acidic by-products can cause corrosive wear, particularly in components containing soft metals.

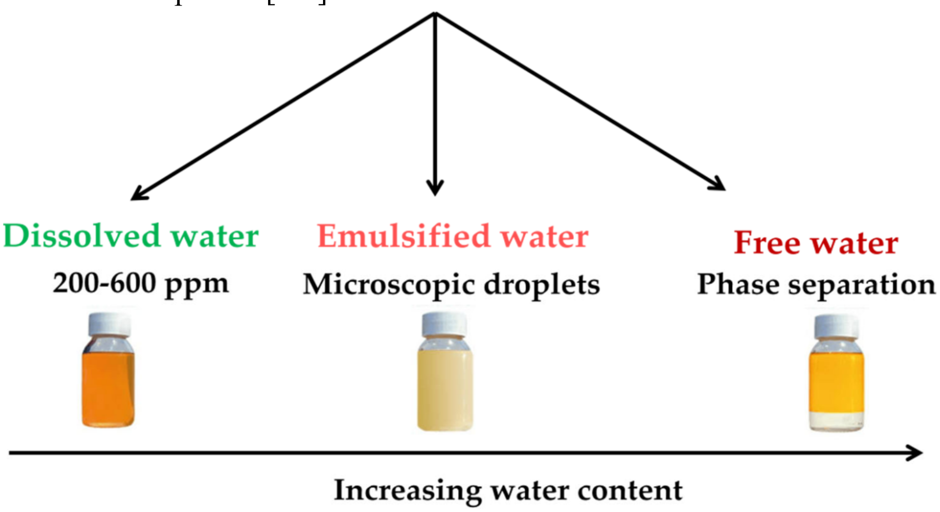
In view of this, water resistance is desired greases in place of water repellency and we have attempted to achieve this by incorporating poly-functional compounds in different types of overbased calcium complex grease chemistries. Significant and sustainable improvement in anti-wear performance can be achieved modifying the compositions of calcium sulphonate complex greases. Lubricating performance of sulphonate complex greases is thoroughly discussed with respect to their structural properties.

INTRODUCTION

In industries like steel where application temperatures are obviously high, operating loads are moderate to high and also the service conditions are very demanding and hostile, all expectation are centered on a proper selection of lubricant. An integrated steel plant consists of four major units namely iron making, steel making, rolling and finishing. The equipments associated with rolling and finishing units are subjected to high impact load, speed, water ingress and hostile environment. Bearing in these units are normally lubricated periodically with grease through centralized lubrication systems [1]. High performance greases are required for these applications and these greases should have following characteristics

- ✓ Base oil viscosities typically VG460 or higher
- ✓ High dropping points
- ✓ Working/rolling shear stability
- ✓ Resistance to water washout and spray off
- ✓ Corrosion protection
- ✓ Low wear and high load carrying

During operation of these systems, cooling is done with spray of high pressure water spray. Though systems are properly sealed, yet the ingress of water cannot be avoided. No contaminant is more complex, intense and confounding than water. During lubrication, Water can exist in following three states or phases [2-3].



The water-cooling may emulsify the grease; as a consequence, normal high temperature grease gets washed away, thus affecting its performance. This is a perennial problem in steel plant leading to loss of grease and finally bearing failures, loss of production etc. Overbasd calcium sulphonate complex (OCSC) greases are commonly used in such humid environments and these greases get emulsified due to presence of water [4].

Resistance to water washout (ASTM D-1264) for OCSC greases is claimed in some patents for example US patent 4560489, 5387351, 7407920. In US patent 8563488, sulfonate grease compositions having resistance to water spray off are reported through use of hydrocarbyl substituted dicarbonyl derivatives and co-polymers derived from olefins and unsaturated dicarboxylic acids [5].

OCSC grease packages are commercially available and claimed to have superior resistance to water spray off in comparison to conventional OCSC greases. There are few reports in literature in which fresh calcium sulfonate complex greases mixed with water have been evaluated for extreme pressure and anti-wear performances. In a study done by Johan Leckner [6], OCSC mixed with 10% water was evaluated for extreme pressure and anti-wear properties and reported similar wear scars for fresh grease and grease mixed with water. It is desirable to develop product/process in which products have improved anti-wear characteristic as water in presence high temperature can cause degradation of such properties. In order to maintain structure and performance, OCSC greases are required to have fine balance between water repellency and water tolerance properties. Recently, over emphasis has been given to water repellency with use of polymers etc. However, more than required water repellency leads to free water which can result in a loss of the hydrodynamic oil film leading to excessive wear. Hence optimum water resistance is required greases and we have attempted to achieve this by incorporating poly-functional compounds in different types of OCSC chemistries.

EXPERIMNETAL WORK

OCSC Grease Details

Considering severity of application in steel industries in terms of load, rpm, temperature and water ingress, OCSC greases were selected for present study. In present work, we have selected four types of OCSC chemistries for evaluation. All greases were developed in ISO VG-460 viscosity grade to provide better film thickness; water spray off and water wash out characteristics. No performance additives were used and all studies were done with base greases. Details of different greases along with physico- chemical tests data are given in Table-1 below;

Table-1: Different OCSC greases for present study

OCSC Chemistry	**Grease Packages**		**Conventional Composition**	**IOC Composition**
Sample ID	OCSC- GP1	OCSC- GP2	OCSC- **HSLS**	OCSC- LSHS
400 TBN OBS/Package	65.0%wt	50.00%wt	45.0%wt	45.0%wt
Complexing acids	No	No	Yes	Yes
Additives	None	None	None	None
Added base oil	Group I Bright Stock,VG460	Group I Bright Stock,VG460	Group I Bright Stock,VG460	Group I Bright Stock,VG460
Manufacturing	1 Step process		2 step process	1 step process
Grease finishing	Colloid milling			
Characteristics	**OCSC- GP1**	**OCSC- GP2**	**OCSC- HSLS1**	**OCSC- LSHS**
Worked Consistency (D217)	270	279	269	283
Water Spray Off (D4049), %wt	< 30	> 70	> 70	< 30
Water Resistant	Yes	No	No	Yes

OBS – Over based sulphonate

RESULTS AND DISCUSSION

OCSC Greases Performance in Presence of Water

All OCSC greases were mixed with 25 wt% water through mechanical stirring at constant RPM and time. Fresh and water mixed OCSC grease samples were worked at 80 ^{o}C in roll stability tester (ASTM D1831) for 16 hours. Fresh, water mixed and worked OCSC grease samples were tested for 4 ball weld load, 4 ball wear scar dia and dropping point and test results are given in Table-2.

Table-2: Test data of OCSC greases with and without water

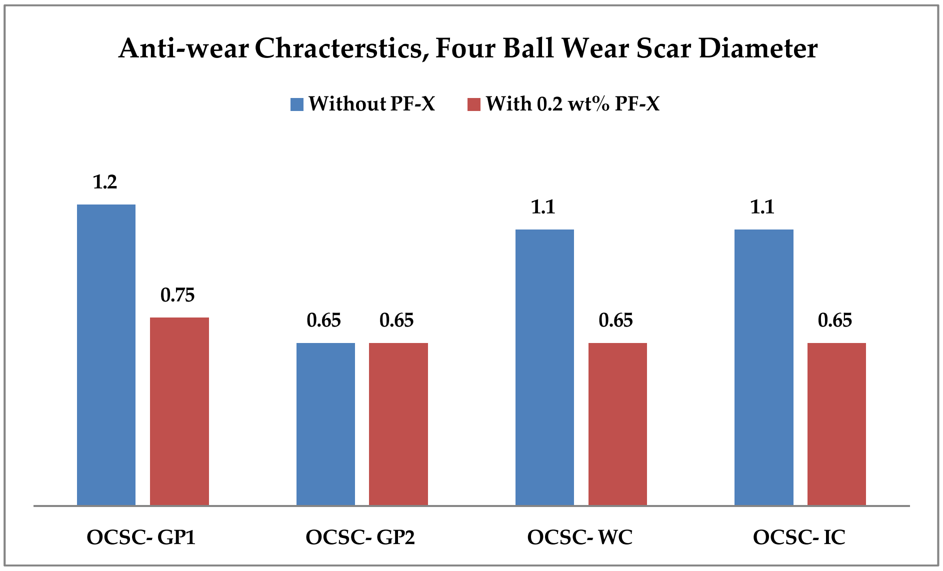
Fresh Grease				
Test Characteristics	**OCSC- GP1**	**OCSC- GP2**	**OCSC- WC**	**OCSC- IC**
IP239, Weld Load(Kg)	355	315	315	280
D2266, Wear Scar Diameter (mm)	0.55	0.55	0.60	0.60
D2265, Dropping Point, ^{O}C	> 316	> 316	> 316	> 316
Fresh Grease mixed with 25%wt water				
Test Characteristics	**OCSC- GP1**	**OCSC- GP2**	**OCSC- WC**	**OCSC- IC**
IP239, Weld Load(Kg)	315	250	250	250
D2266, Wear Scar Diameter (mm)	**1.20**	0.65	0.75	**1.20**
D2265, Dropping Point, ^{O}C	> 316	> 316	> 316	> 316
Fresh grease mixed with 25%wt water and worked for 16 hrs in roll stability tester at 80 ^{O}C				
Test Characteristics	**OCSC- GP1**	**OCSC- GP2**	**OCSC- WC**	**OCSC- IC**
IP239, Weld Load(Kg)	225	225	225	225
D2266, Wear Scar Diameter (mm)	1.20	0.60	1.10	1.10
D2265, Dropping Point, ^{O}C	> 316	220	> 315	> 315

Test data indicates that water resistant greases OCSC- GP1 and OCSC- IC gave very high four ball wear dia in comparison to OCSC- GP2 and OCSC- WC when grease tested with 25% water. This may be due to presence of free water in OCSC- GP1 and OCSC- IC greases due to high water repellency. On the other hand, water is trapped inside the matrix in OCSC- GP2 and OCSC- WC greases due to emulsification. All greases mixed with 25% water were found to be non-drop (>316 ^{O}C) and slight decrease in extreme pressure characteristics observed as shown in Table-2. When greases roll worked in presence of water, all greases except OCSC- GP2 gave very high wear scar dia. Wear scar diameter also increased in OCSC- WC indicating loss of water entrapment efficiency or matrix breakdown. Decreased dropping point was observed in OCSC- GP2 grease.

Improvement of OCSC Grease Performance in Presence of Water

To further improve the grease performance in presence of water, different poly-functional low molecular weight polydydroxy compounds (total five) were evaluated in treat rate of 0.1-0.5 %wt. These poly-functional compounds were added prior to gelling in all OCSC greases. No significant changes were observed in base greases when tested with and without these compounds. However, significant changes were observed in test data of these greases which were roll worked in presence of water with one poly- functional compound (PF-X). In all greases spiked with PF-X, wear scar dia was found to be in range of 0.65-0.75 mm indicating significant improvement in anti-wear performance in presence of water as shown in Figure-1. OCSC- GP2 grease spiked with PF-X was also found to be non-drop. No significant change in extreme pressure performance was observed.

Figure-1: Evaluation of PF-X in 16 hrs roll stability Test in presence of 25% water



CONCLUSIONS

Resistance to water washout/water spray off is a critical requirement for industrial applications which are exposed to water. Water resistance performance of high performance greases can be tailor made by changing the base oil viscosity, NLGI grade and use of polymers. However, very little emphasis is given to actual lubrication in mating surfaces in presence of water. Presence of free water can lead to serious bearing failures due to high wear and corrosion. Hence optimum balance of water repellency and water absorption is required especially in overbased calcium sulphonate complex greases. As shown above, anti-war performance of overbased calcium sulphonate complex greases can be improved by incorporating selected chemicals in suitable amounts.

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