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On the cover – Group Photo of 16th Grease Education Course, Silvassa

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A heavy specialty oil for lubricating greases

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Abstract

A highly viscous base fluid containing residue from the vacuum distillation process with a kinematic viscosity of approximately 700 $m^2 \cdot s^{-1}$ at 40°C has been developed. This base fluid can be used for various applications where the color is not an issue, since it is black.
In order to explore the potential of this heavy specialty oil as a substitute to bright stock in grease formulations, several greases based on different viscosities have been prepared and characterized.
The residue oil based greases showed interesting characteristics such as having a positive impact on the thickener content, shear stability, water resistance and rheological behavior.

1. Introduction

The global ongoing rationalization of Group I production and its potential impact on the future availability of paraffinic bright stock has led several lubricant formulators to start evaluating alternative products.
The alternative available in the market right now is to reformulate by using a mix of heavy grade neutrals plus a synthetic oil component such as polyalphaolefin, heavy naphthenics and other sources. This paper suggests a cost effective alternative which may replace bright stocks in various grease lubricated applications where the color is not an issue.

2. Experimental work

It is well known that for industrial lubricated applications, higher base oil viscosities are required which usually consist of heavy neutral and bright stock blends. Hence, the starting point for the experimental work was to prepare some blends based on these two oils. In order to make the comparison as realistic as possible, two paraffinic oils (BS200 & SN500) and a highly viscous naphthenic oil (R1) has been chosen, the characteristics of the base oils can be seen in Table 1.

Table 1. shows the characteristics of the base oils.

Characteristics	Method (ASTM)	BS200	SN500	R1
Viscosity, 40°C (mm^2/s)	D445	838,28	95,39	690,81
Viscosity, 100°C (mm^2/s)	D445	43.19	9.61	29.22
Viscosity index	D2270	92	71	50
Flash Point, PM (°C)	D93	275	227	220
Pour point, (°C)	D92	-6	-24	-15
Aniline point, (°C)	D611	113	98.75	90
Density, 15°C (g/dm3)	D4052	918.9	892.4	940

Three different viscosities (235, 370 and 505 $mm^2 s^{-1}$) have been prepared by diluting BS200 and R1 with SN500 respectively. Table 2 shows the characteristics of these blends; A, B and C are blends of the two paraffinic oils (SN500+BS200), while A´, B´ and C´ are blends of paraffinic and naphthenic (SN00+R1).

Table 2. shows the characteristics of the blends which have been used for preparation of the greases.

Characteristics	Method (ASTM)	A	B	C	A´	B´	C´

Viscosity, 40°C (mm^2/s)	D445	234.6	372.2	503.6	235.2	369.0	506.6
Viscosity, 100°C (mm^2/s)	D445	17.9	24.7	30.4	16.2	20.8	24.8
Viscosity index	D2270	81	86	88	60	56	54
Flash Point, PM, (°C)	D93	229	239	247	223	223	223
Pour point, (°C)	D92	-12	-12	-12	-24	-24	-15
Aniline point , (°C)	D611	105.3	109.9	110.4	94.3	92.8	91.7
Density, 15°C, (g/dm3)	D4052	907.9	912.2	914.5	921.3	930.5	936.1
Cu- corrosion, rating	D130	1a	1b	1b	1a	1a	1a

3. Results ad Discussion

The grease samples have been prepared in an open kettle where the pre blends (see Table 2) were used. The target for these samples was NLGI grade 2. Notable is that the greases used in this study do not contain any additives.

Table 3. shows the characteristics of the greases based on the blends in Table2.

Characteristics	Grease A	Grease B	Grease C	Grease A´	Grease B´	Grease C´
Thickener content (%)	6.2	6.6	5.6	5.1	4.8	5.0
Penetration (60)	276	268	272	277	268	274
Dropping point (°C)	208	206	208	202	203	199
Cu-corrosion *	2c	1b	1a	1a	1a	1a
Penetration (100,000)	296	291	294	275	278	277
Water wash out (%) **	1.37	1.61	2.00	2.73	1.38	2.12
Water spray off (%) ***	55.6	53.8	31.2	43.8	N/A	18.8

*) 100 °C/24h; **) 79 °C/1h; ***) 38 °C/5min/40psi (276 kPa)

Based on the measured properties of the greases, shown in Table 2, some interesting behaviors for R1 based greases can be utilized such as: lower thickener content, excellent mechanical stability and superior water spray off.

Tribological measurements

The load carrying capacity of the grease samples have been measured by using a four ball machine. As it can be seen in Table 4, the wear scar of all 6 grease samples indicates that there is no significant difference between the samples; which are anyhow interesting since Grease A´, B´ and C´ contain lower thickener as well as lower viscosity index than grease A, B and C respectively.

Table 4. shows the measured wear scars of the greases.

Remarks	Grease A	Grease B	Grease C	Grease A’	Grease B’	Grease C’
Wear scar (mm)	2.73	2.71	2.77	2.73	2.70	2.67

The condition of the four ball test was: 140 kg, 60 sec, 1440 rpm

Rheological measurements

It is well known that lubricating grease is a viscoelastic material, in other words a material with a viscous part (the base oil) and an elastic part (the thickener). Parameters such as temperature and shear stress affect the oil and the thickener differently. Hence, flowability of the grease sample under controlled conditions can generate valuable information such as storage (elastic) modulus. This reflects the consistency or/and the elasticity of the grease at applied temperature. Hence, the higher the storage modulus (G´), the thicker the grease. In order to study the change of G´ as a function of temperature for the grease samples, the so called oscillatory program has been used. Notably the geometry of the plates was plate on plate and the temperature interval was from +50 to -30 °C. Figure 1 is an example of the generated result for greases C and C´.

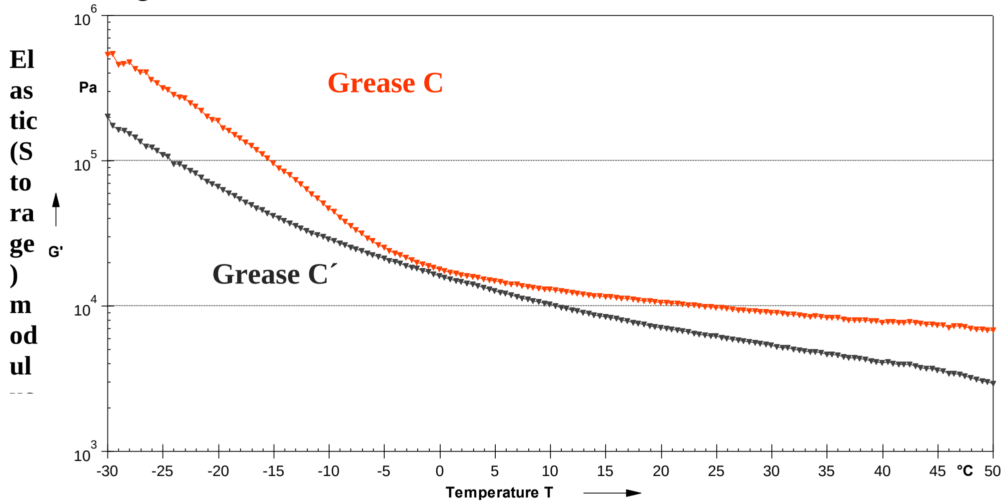

Figure 1 shows the behavior of Grease C and C' respectively as a function of temperature.

This graph reveals some interesting information such as:

a) G´ of C is higher than C´ at 25 °C in spite of the fact that they have almost same penetration number at that temperature. This can be to some extend be explained if we look at the thickener content for these two greases. Grease C has 12% higher thickener than grease C´.
b) The change of the slope for grease C around -5 °C can be attributed to the presence of bright stock (BS200) and subsequent assumed wax formation and thereby the rapid increase of G´ at lower temperature.
c) The higher G´ for grease C at -30 °C, compared with grease C´ should consequently be the impact of the further wax crystallization of the bright stock. It is reasonable to believe that SN500, at least, is not the main source of this increase since both greases contain SN500.

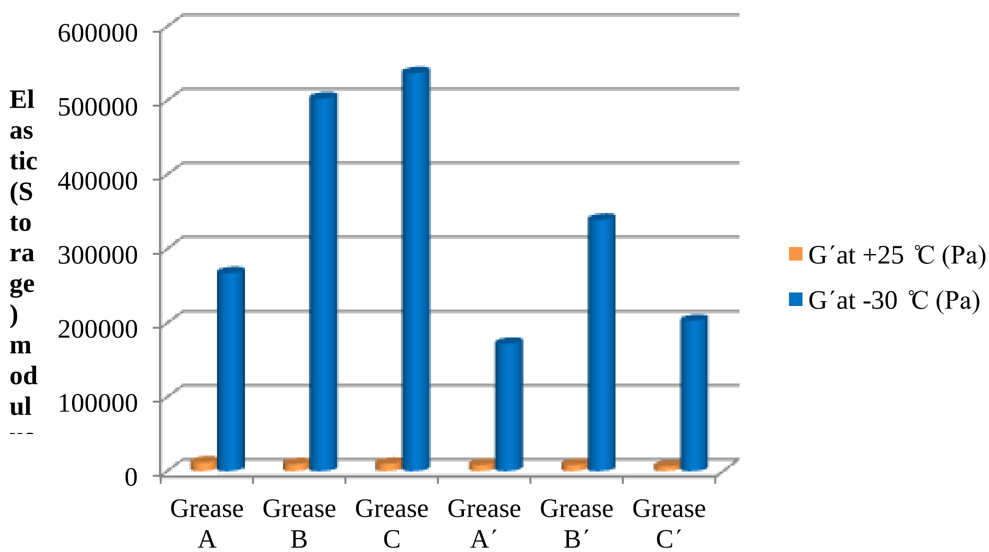

Figure 2. shows the increase of the G´ when the temperature reduced from +25 to -30 °C.

Based on the obtained elastic modulus, shown in Figure 2, the net increase of the elasticity for each and every paraffinic based grease (Grease A, B and C resp.) is significantly higher than for the paraffinic/naphthenic based greases with equivalent base oil viscosity at 40 °C.

4. Conclusions

A highly viscous naphthenic oil, as a substitute to bright stock, has been has been evaluated for grease formulation. Based on the obtained results in this study, it can be concluded that by substituting bright stock by R1:

I) 11to 27 % saving in thickener content.
II) Better mechanical stability, in spite of lower thickener content.
III) Excellent copper corrosion inhibiting, without any copper passivator additive.
IV) Very good water wash out, probably due to high adhesivity of R1
V) Similar wear scar as the bright stock based greases despite of lower thickener content and lower viscosity index.

However, the overall conclusion should be that it is possible to substitute bright stock in grease formulations with equivalent or better performance. Consequently, a more cost effective grease formulation could be achieved, provided the color is not an issue. It is well known that within industrial applications such as in mining color is often not an issue.

DEVELOPMENT OF ENVIRONMENTAL FRIENDLY HIGH PERFORMANCE SYNTHETIC GEAR OIL

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ABSTRACT:

Selecting the proper industrial gear oil is important for the long life and efficient operation of the gear drive. A gear box is one of the most important and complex engineering element. Gear oil must satisfy changing design and performance requirements. Oil volumes get smaller, oil circulation cycle increases and oil dwell time decreases. However the energy requirement of the lubricant increases. This leads to an increase in the thermal and oxidative load on the lubricant. In addition, the technical demand on industrial lubricant have changed dramatically over recent years. These have become more "stringent". With advanced base stock technology and gear oil design to provide optimum equipment protection and oil life even under extreme conditions. A high performance Synthetic Environmental Friendly Gear Oil was developed on the basis of the novel high viscosity base stock technology which has given better properties than synthetic hydrocarbon basestock based Synthetic Gear Oil. Besides its superior performance the new developed synthetic gear oil is biodegradable and has high renewable content to meet environmental compliance

INTRODUCTION

Gears are machine elements that transmit motion by engaging teeth. The main function of a gear lubricant is preventing wear and removing frictional heat generated in sliding and rolling contacts by providing a lubricating film between the gear mating surfaces [1]. Usually parts requiring gear lubrication are

reduction gear drive units, gear motors, chain cases, pinion saws, guides, slides, and several type of bearings [2].

Selecting the proper industrial gear lubricant is important for the long-term efficient operation of gear drive. There are many factors to consider when selecting an Industrial gear lubricant for a particular application. These factors are summarized in Table 1.

FACTORS	REQUIREMENT
Gearing Type	
• Spur and bevel	Low slide, high speed
• Helical and spiral bevel	Moderate slide, moderate to high loading
• Hypoid	High speed, high loading
• Worm	Excessive sliding, moderate to high loading
Loading	Highly loaded industrial gear drives require the use of extreme pressure gear lubricants
Surface finish	Rough surfaces require high-viscosity oils, smooth surfaces can use lower viscosity oils
Transmitting power	As load is increased, viscosity must be increased
Gear speed	The higher the speed of the gear drive, the lighter the viscosity of the oil
Material Compatibility	Some of extreme pressure additives can attack yellow metals such as brass and bronze
Temperature	The industrial gear lubricant's viscosity must be selected based on the lowest and highest operating and/or ambient temperature experienced

In addition to considering these products, the gear lubricant selected for a particular application should match the recommendations of the original equipment manufacturer [3].

Over the years, industrial gearing has been characterized by a continuously moving to greater power–to-size ratios in order to reduce cost to the end users. A significant consequence of the move to higher unit loading is that industrial

boxes tend to run hotter than in past. This has various consequences for lubrication conditions and provides opportunities for new gear oil technology – not just in terms of base stocks and additives but also in terms of scientific knowledge [4].

Synthetic lubricants perform better than petroleum base lubricants [5]. Synthetic industrial gear oils are used whenever mineral gear oils have reached their performance limit and can no longer meet the application requirements [6]; for example at very low or high temperatures, extremely high loads extraordinary ambient conditions, or if they fail to meet special requirements such as flammability. Even though additives can improve many properties of mineral oils. It is not possible to meet an unlimited influence on all their properties. Synthetic gear oils provide a number of advantages [7]. However, they do not necessarily out-perform mineral oil in all respects and may even results in some drawbacks despite their advantages.

ENVIRONMENTAL FRIENDLY SYNTHETIC GEAR OIL

For the past several years, the lubricants industry has been quietly looking into eco-friendly, readily biodegradable and non-toxic fluids. Our industry is responding because a vast quantity of industrial lubricants are finding their way into the environment. In fact, the National Oceanic and Atmospheric Administration estimates 700 million-plus gallons of petroleum products enter the environment each year, more than half of which are through irresponsible and illegal disposal. It damages living organisms including plants, animals and marine life for many years. When an environmentally preferable product is required outside common temperature ranges, a biodegradable synthetic is usually selected [8,9,10].

On the basis of novel base stock technology Synthetic Environmental Friendly Gear Oil developed. In new developed Gear oil a novel high viscosity base stock technology as alternative to high viscosity Synthetic Hydrocarbon base stock was used. The viscometric properties of this novel high viscosity base stock is similar to common high viscosity Synthetic Hydrocarbon but viscosity index is higher and additive solubility characteristic is superior.

New developed Synthetic Gear oil base on novel base fluid technology has given better properties than conventional high viscosity synthetic hydrocarbon fluid based synthetic gear oil. Beside its superior performance the new developed synthetic gear oil is biodegradable and has high renewable content to meet the environmental compliance.

TYPICAL FORMULATION OF SYNTHETIC INDUSTRIAL GEAR OIL

Two synthetic base fluid were selected for Synthetic Gear Oil formulation. One is high viscosity polyalphaolefin and second is novel technology base high viscosity ester type fluid. Table 2 summarizes the typical formulation of synthetic gear oil with both type of base stocks.

TABLE 2: TYPICAL FORMULATIONS

	COMMERCIAL SYN GEAR OIL WITH HIGH VISCOSITY PAO (CSGO)	NEW DEVELOPED SYN GEAR OIL WITH HIGH VISCOSITY NOVEL BASE STOCK (NDSGO)
BASE FLUID 1	LOW VISCOSITY PAO 30-50%	LOW VISCOSITY PAO 35-45%
BASE FLUID 2	ESTER 5-10%	-
HIGH VISCOSITY BASE FLUID	HIGH VIS PAO 40-70%	HIGH VIS NOVEL FLUID 50-70%
ADDITIVE	ADDITIVE PKG 1-4%	ADDITIVE PKG 1-4%

LABORATORY EVALUATION & DISCUSSION

In the wide variety of conditions to which modern Industrial Gear oils are exposed, Industrial gear oils need to provide many performance features to ensure optimum performance of gear boxes in a variety of applications. They are designed to reduce friction and wear, act as heat transfer agents, and protect against corrosion and rust. They also minimize oil oxidation, inhibit foaming and separate water readily.

Evaluation and comparison the overall performance of novel technology base fluid based synthetic gear oil was done. The test and results are as under.

VISCOSITY INDEX

Oil viscosity is affected by temperature changes during use. As a gear lubricant's temperature increase, its viscosity decreases, along with load-carrying ability. The degree of change that occurs is determined by ASTM D -2270 and referred to as the lubricant's viscosity index (VI) occurs between 40 C and 100 C. The higher the VI, the less is the viscosity change with temperature. A high VI is desirable and, indicates higher lubricant quality. It does not, however, represent a lubricant's high temperature viscosity or its load carrying ability.

Figure 1 shows the comparison of Viscosity Index NDSGO and CSGO. NDSGO shows much higher VI than other CSGO. Viscosity change with temperature is lower in NDSGO than in CSGO so it performs better. High viscosity index makes the product suitable for operating over a wide temperature range.

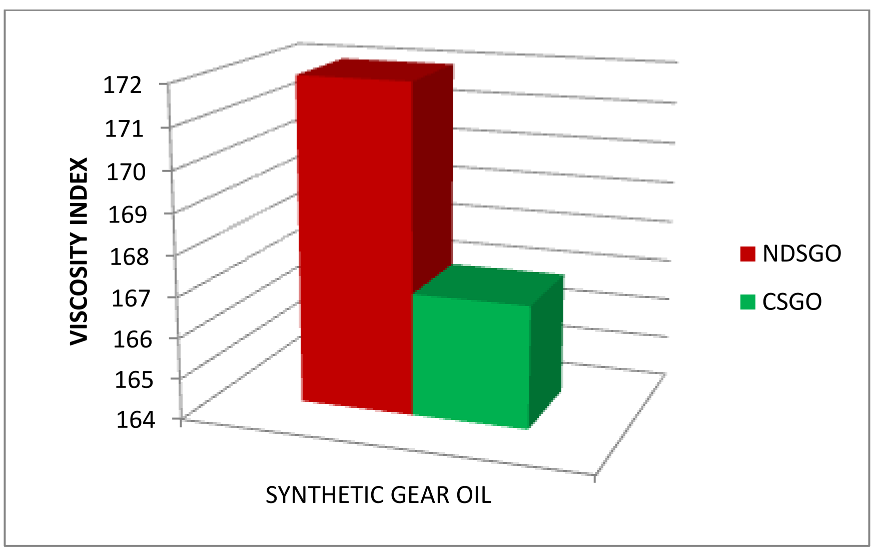

FIGURE 1

THERMAL STABILITY (MODIFIED ASTM D 2070)

A thermaly stable gear oil keeps critical parts clean, with respect to deposits and sludge, when subjected to sustained high temperature service, there by significantly extending equipment life. Non thermally stable gear oils oxidize and decompose when subjected to high temperatures. Oxidized oils can deposit sludge, varnish and carbonaceous deposits on gears, bearing seals. These deposits can cause premature wear, abrasive seals, premature seal hardening and brittleness. Increased operating temperatures and decreased gearbox efficiency.

In this test a sample of oil under test placed to ageing at 100 C for 72 hrs in presence of copper and steel rods. After test, rod colour evaluated and sludge calculated.

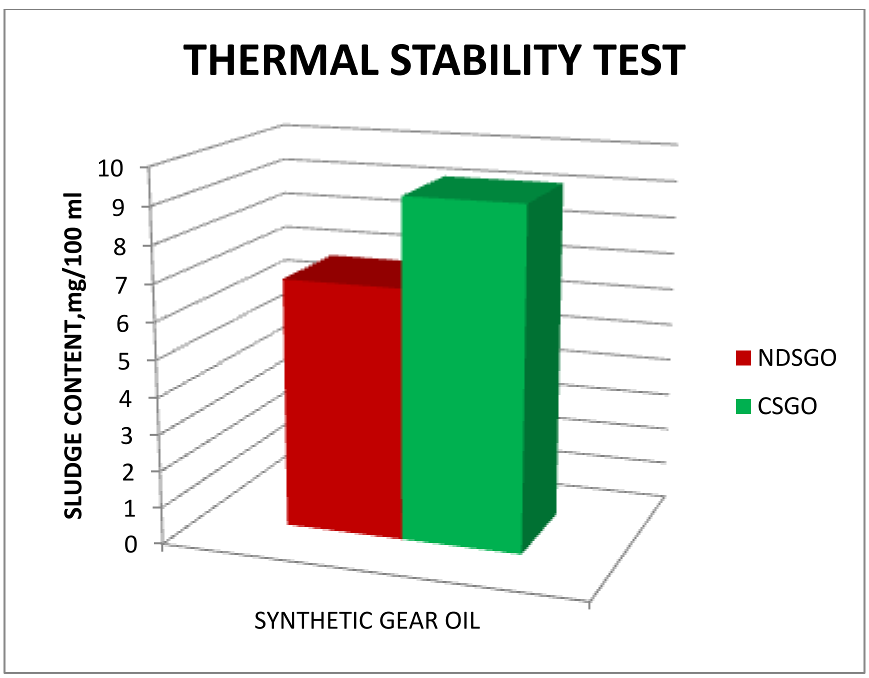

FIGURE 2

From the Figure 2 it is observed that new developed synthetic gear oil gives lower sludge content compared to conventional synthetic gear oil and has higher thermal stability.

DEMULSIBILITY CHARACTERISTIC

Gear lubricants are frequently exposed to water contamination. Water can cause rust and corrosion to machinery parts, rapidly accelerate oxidation of the gear lubricant and cause drop-out of the gear lubricant additive system. Gear lubricant's must possess the ability to rapidly separate from and resist emulsification with water and also quick and effective water removal from the system during static conditions. The ability of gear lubricant to separate from water is called demulsibility.

FIGURE 3: Demulsibility Testing Equipment

ASTM D 1401 test method measures the ability of gear oil to separate from water. In this test 40 ml sample of oil and 40 ml distilled water are vigorously stirred together at a speed 1500 rpm in a special graduated cylinder for 5 minute a temperature of 82 ^{o}C.

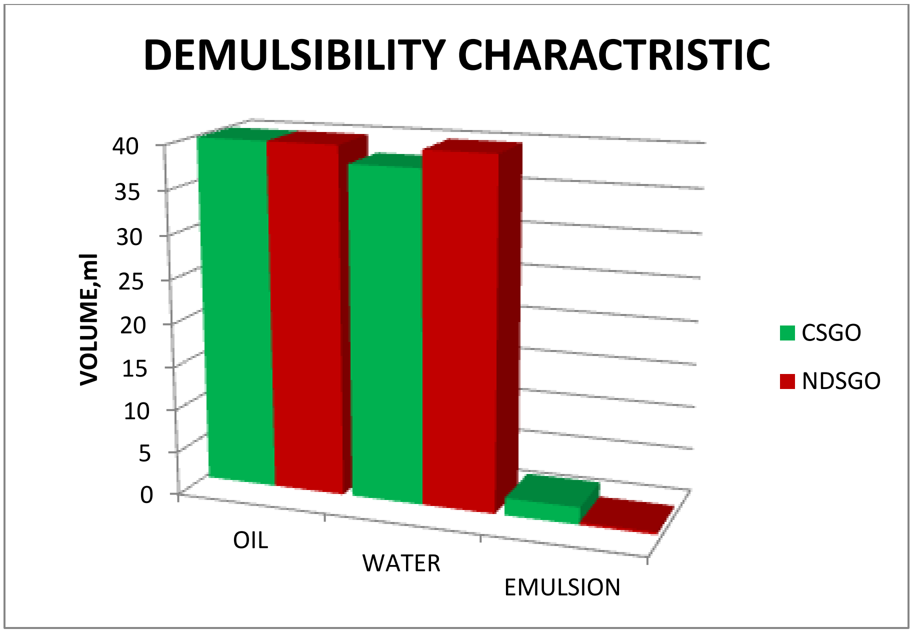

FIGURE 4

Figure 4 summarizes demulsibility data of newly developed synthetic gear oil and commercial synthetic gear oil. The new developed synthetic gear oil show much higher demulsibility characteristic compare to commercial synthetic gear oil. NDSGO reduce down time through water separation and demultification characteristics resulting in prolonged lubricant life and increased equipment reliability

GEAR WEAR PROTECTION

Industrial gear drives are operating under increased power density loads. These higher power density loads are result of increased horsepower and torque being applied to gear drives. These conditions result in increased stress on the gears and bearing that can lead to premature or catastrophic failure. The gear lubricant must protect against excessive gear and bearing wear, scoring, and pitting, especially when high torque, high shock load and high temperature conditions are encountered

FIGURE 5: Four Ball Machine

WEAR PREVENTIVE CHARACTERISTIC OF LUBRICANTS ASTM D 4172

This test evaluates the antiwear properties of fluid lubricants in sliding contact and under lighter loads than those used in the 4 ball EP test. It is conducted using the 4 ball antiwear test procedures and measurements which are different than the 4 ball EP procedure. The standard test parameters of the 4 ball wear test are 75 C, 40 kg load, 1200 rpm for 1 hr. The wear scar diameter of the three stationary balls is measured and average is reported as the wear scar in mm.

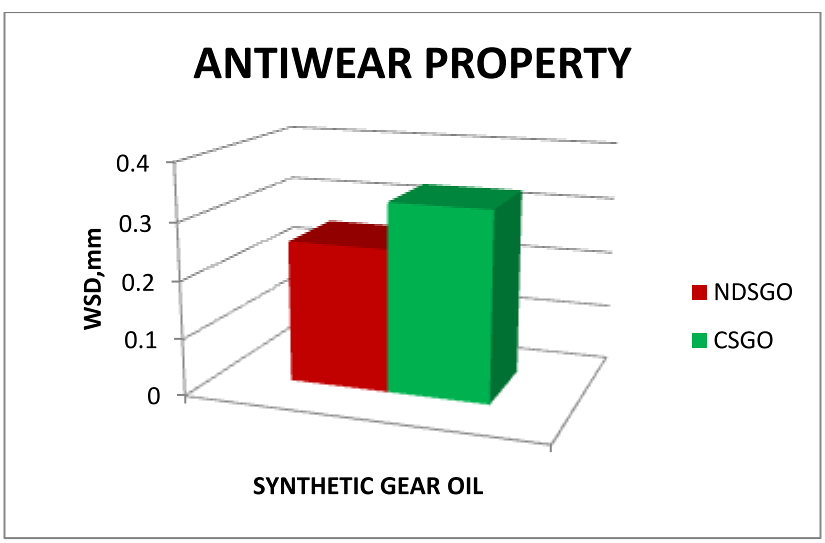

FIGURE 6

Figure 6 plots the comparative value of WSD as a function of antiwear properties. Under the identical test conditions lower WSD of NDSGO indicating lower wear compare to CSGO. In the actual applications under sever operating conditions NDSGO is expected to give good antiwear property which will lead to enhance life of the machine components.

FOAM RESISTANCE ASTM D-892

During differential operation, gears and bearings turn at high speeds churn the lubricant when air is introduced, foaming can occur, while gear lube is considered incompressible, air is compressible and when bubble pass between loaded areas, the bubbles collapse and metal-to-metal contact occur, causing wear. Foam can also increase heat and oxidation. Good foam control is important in gear lubricants.

FIGURE 7: Equipment For Foaming Tendency

Standard test method for Foaming characteristics of lubricating oils is ASTM D 892. This test method consists of three sequences performed at different temperatures (24^{o}C, 93.5 ^{o}C and 24^{o}C). The lubricant being evaluated is aerated by the use of a gas diffuser at the test temperature for that sequence with dry air at a flow rate of 94 ml/min. The sample is aerated for 5 minutes and amount of foam generated at 5 seconds after disconnecting the gas diffuser is reported. At the end of a 10 min settling time the amount of foam left is recorded.

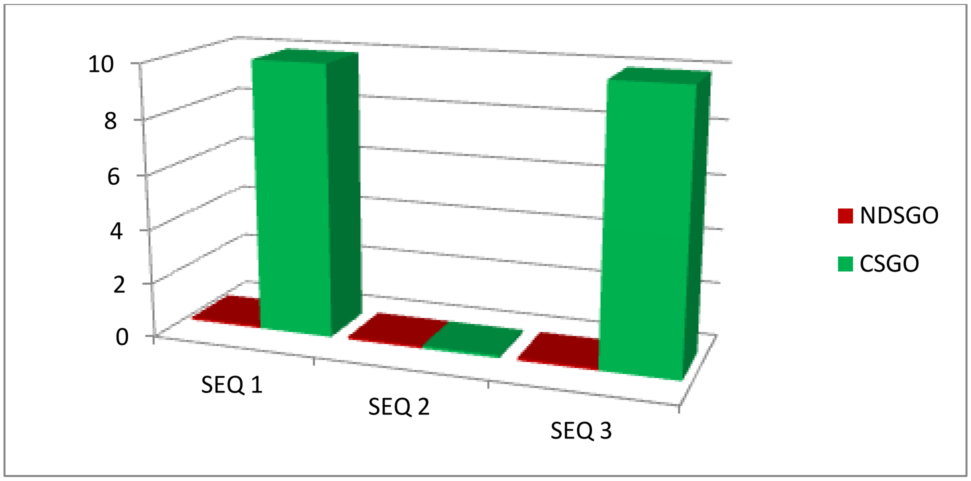

FIGURE 8

Figure 8 plots the all three sequences of foaming. Under the identical test conditions, NDSGO shows better foaming characteristics than CSGO. Good foam control is important in gear lubricants.

OXIDATION STABILITY ASTM D 2893

Oxidation causes degradation of oil over time. As oil oxidizes, it thickens (viscosity increases), the total acid number increases and deposit sludge, and the additives are being used up. This cause excessive wear and eventually, failure of various parts. The test method is used to measure the ability of extreme pressure gear lubricants to resist oxidation and the formation of deposits when subjected to high operating temperatures.

In the test a 300 ml sample of the gear lubricant being tested is placed in a 600 mm length test tube that contains a flow meter to regulate air flow. The entire assembly is placed into an oil bath that is held at a temperature of 100 ^{0}C for 312 test hrs, with dry air at a flow rate of 10 lt/hr

FIGURE 9: Oxidation Stability Bath

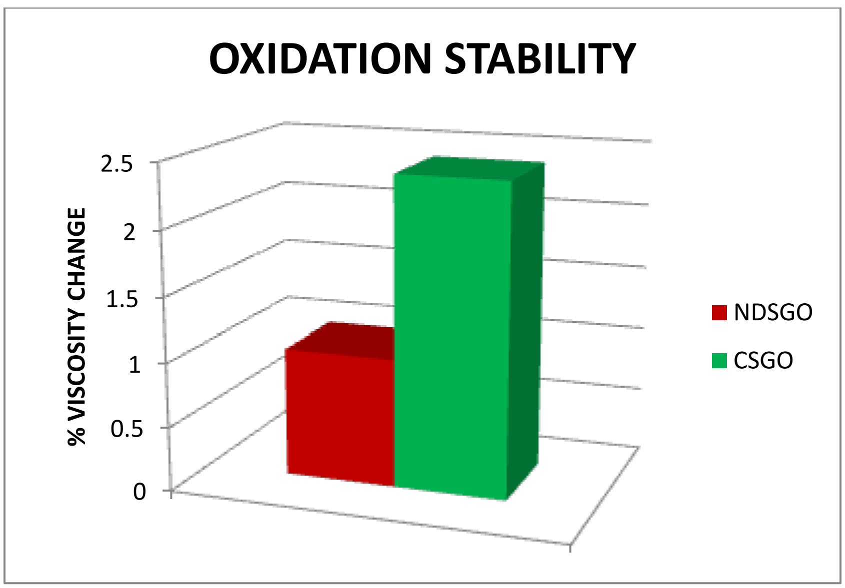

FIGURE 10

In figure 10, comparative results of thermal oxidative stability shows that the NDSGO has lower % viscosity change @ 100 C. So results indicates the superior thermal oxidative resistance of NDSGO compared to CSGO.

SEAL COMPATIBILITY

Seal commonly fail due to hardening or deposit formation. Specially when high operating temperatures are encountered. Therefore it is important that the gear

lubricant must incorporate a careful balance of additive chemistry base fluids in order to avoid seal failures.

Static elastomer test with NBR elastomer for 168 hrs @100 ^{0}C was carried out.

In figure 12 results of seal compatibility test indicates that the seal compatibility of NDSGO is comparable to CSGO.

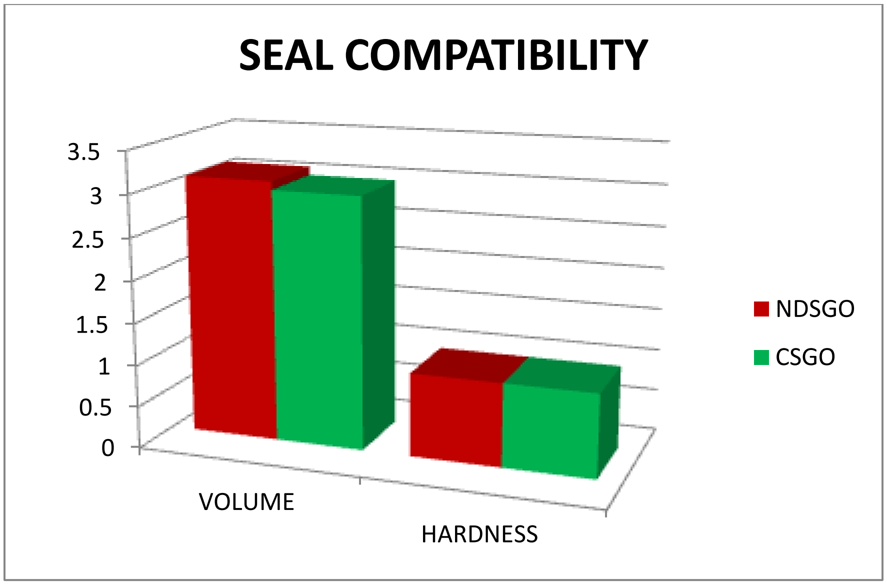

FIGURE 11

BIODEGRADIBILITY

Biodegradation is a process of chemical breakdown or transformation of a substance caused by micro-organisms (bacteria, fungi) or their enzymes.

Biodegradability is the ability of a substance to biodegradation measured in a standard test procedure. The most widely used test that measure this decrease is the CEC-L-A-94 of the Coordinating European Council (CEC).

As shown in figure 12, NDSGO has excellent biodegradability compare to CSGO. NDSGO is rapidly biodegradable and therefore intended for use wherever biodegradability is required. The CEC-L-33-A-94 standard states an oil is rapidly biodegradable when more than 70% of the lubricant is biodegradable within 21 days.

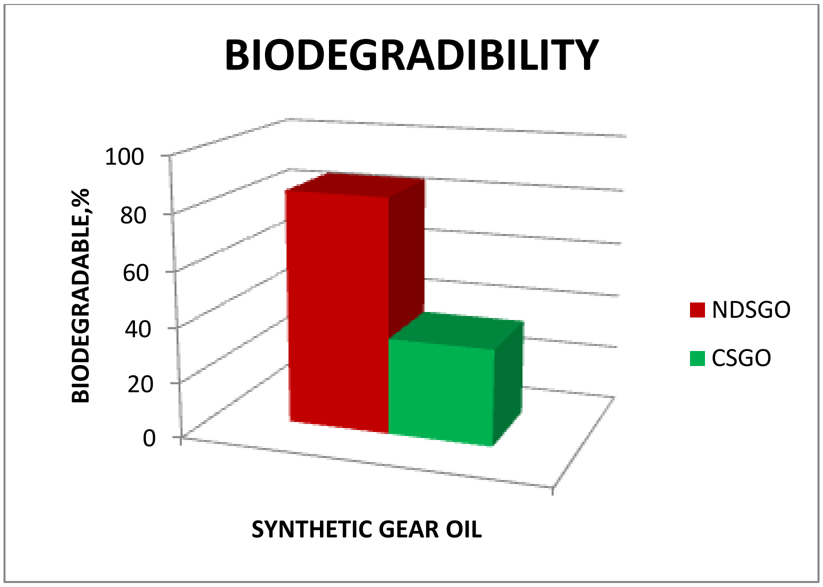

FIGURE 12

POUR POINT – ASTM D – 97

Pour point can vary greatly depending on the construction of the product. Pour point measure the cold-temperature properties of gear lube. Pour point is defined as the coldest temperature at which oil will flow before solidifying.

The Pour Point Test consists of a glass jar filled with gear lube which is cooled to a temperature close to its pour point. The gear lube is checked at intervals of 3°C (5°F) for fluidity. When the gear lube no longer flows, the pour point is recorded at the last temperature of fluidity.
Figure 13 shows the low pour point of NDSGO compared with CSGO. So results indicate the better performance of the NDSGO at low temperature.

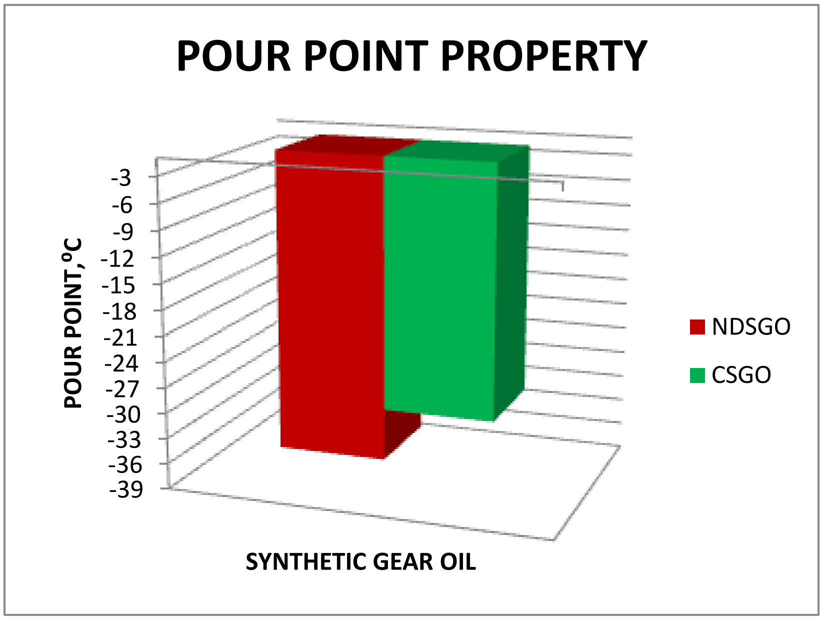

FIGURE 13

Copper Corrosion Resistance - ASTM D-130

Extreme-pressure additives in gear lubricants become more chemically active when subjected to heat. Copper and brass are soft metals and are subject to attack from acids, sulfur compounds and other chemicals in gear lubricants. When corrosion attacks these components it can be seen as a discoloration and occasionally forms buildup on the surface of the component. Acidic corrosion results in wear, which can lead to component failure.

The standard Copper Corrosion Test is designed to assess the corrosive characteristics of lubricants. In this test a polished copper strip is immersed in a test tube with a given quantity of sample fluid. The entire test tube is then immersed into a bath which is heated to 100°C (212°F) for three hours. The copper strip is then removed, washed and evaluated according to ASTM Copper Strip Corrosion Standards.

The results of copper corrosion resistance are given in table 3 which indicate that the NDSGO is comparable to CSGO.

RUST RESISTANCE ASTM D-665 A&B

Water can became mix with gear lubricant and rusting of ferrous parts can occur. The standard test method for test rusting characteristic of gear oil is ASTM D -665 A&B.

A mixture of 300 ml of the oil under test is stirred with 30 ml of distilled water or synthetic sea water, as required, at a temperature of 60 °C (140 °F) with a cylindrical steel test rod completely immersed in. It is recommended to run the test @ 1100 rpm for 4 hrs; however, the test period may be varied, at the discretion of the

FIGURE 14

contracting parties, be for a shorter or longer period. The test rod is observed for signs of rusting and, if desired, degree of rusting.

The results of rust resistance are given in table 3 which indicate that the NDSGO is comparable to CSGO.

TABLE 3: TECHNICAL DATA OF NEW DEVELOPED SYNTHETIC GEAR OIL AND CONVENTIONAL SYNTHETIC GEAR OIL

S.NO.	PEROPERTIES	TEST METHOD	NDSGO	CSGO
1.	Appearnce	Visual	C&B	C&B
2.	KV @ 40 ^{o}C	ASTM D 445	217.89	218.12
3.	KV @ 100 ^{o}C	ASTM D 445	29.0	28.2
4.	VI	ASTM D 2072	172	167
5.	TAN	ASTM D 972	0.82	0.35
6.	Density @ 29.5 ^{o}C	ASTM D 1289	0.8915	0.889

7.	Pour Point	ASTM D 97	-36	-30
8.	Flash point, ^oC	ASTM D 93	254	248
9.	Foaming Tendency Seq 1@ 24 ^oC Seq 2@ 93.5 ^oC Seq 3@ 24 ^oC	ASTM D 892	 0/0 0/0 0/0	 0/0 10/0 0/0
10.	Demulsibility	ASTM D 1401	40/40 (10min)	40/38/2 (30 min)
11.	Oxidation Stability % Viscosity Change	ASTM D 29983	 1.1	 2.4
12.	Cu-Corrosion	ASTM D 130	1b	1b
13	Rust Resistance i)A(With Distilled water) ii)B(With syn sea water)	ASTM D 665	 Pass Pass	 Pass Pass
14.	Wear Scar Dia,mm	ASTM D	0.25	0.33
15.	FZG A 8,3/90		>14	>14
16.	Biodegradability, %	CEC-L-A-94	85	35
17.	Static Elastomer Test, NBR 28 elastomer, @ 100 for 168 hrs -% Volume Change -Hardness Change		 +3.2 +1	 +3.0 +1.0

CONCLUSIONS

Changes in industrial gearing and applications continue to lay increased stress on the capability of industrial gear lubricants. Novel fluid technology based newly developed Synthetic gear oil has excellent biodegradability, high viscosity index, low temperature property, high thermal sand oxidation stability. New developed synthetic gear oil shows good rust protection, improved foam control and bearing protection. From these studies, it can be said that NDSGO has good potential for use as a Environmental friendly Synthetic Industrial Gear Oil.

On the basis of laboratory evaluation novel fluid technology base new developed Synthetic gear oil showed superior performance compared to conventional synthetic gear oil.

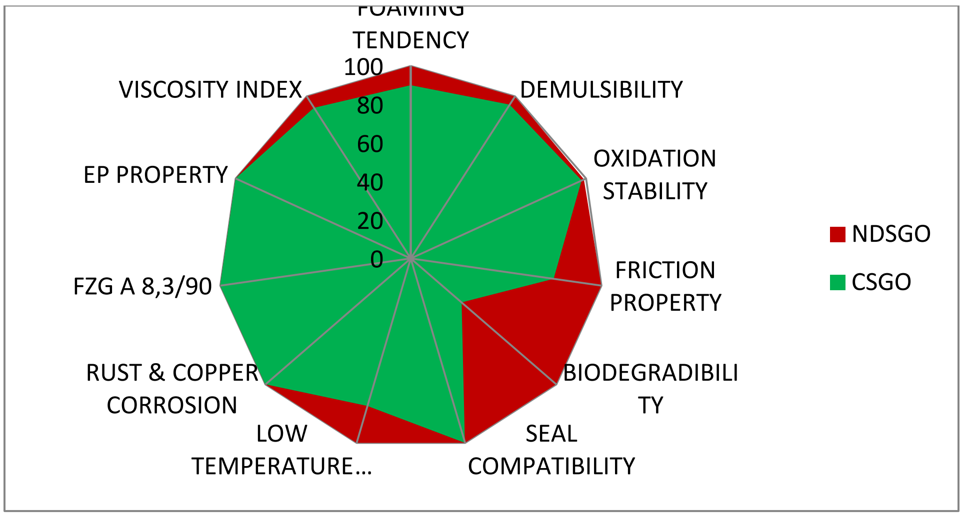

FIGURE 15

The figure 15 compares the overall performance of NDSGO with CSGO.
The key performance feature of NDSGO is its balance nature compared with CSGO.

RECOMMNDED APPLICATION

New developed synthetic gear oil is readily biodegradable, environmental friendly ecologically responsive, non-toxic, thermally stable and thermally durable extreme pressure lubricant that can operate in both high and low temperature environments. Typical applications include wind turbine, high;ly loaded gear boxes used in paper Industry, steel industry, Oil and cement industries, etc., where gear protection and long lube life are required.

FUTURE PLAN

To assure the quality and performance ability of new developed product rig testing and field trials are planned.

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Temperature-Dependent Rheology and Tribology of Lubrication Greases

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Abstract

Compared to oils greases have a number of advantages with respect to the construction and service of lubricated components. However, due to the visco-elastic behavior of greases there are certain constraints to consider regarding the flow and tribological properties. Therefore having an instrument and methods to investigate the visco-elastic and frictional behavior of greases over an extended temperature range is highly desirable. A tribometer as well as a rheometer requires speed and normal force control, and torque measurement to acquire tribological data. An air bearing supported rotational rheometer allows the measurement in the whole range necessary for advanced rheological measurements. Oscillatory amplitude sweeps are very well suited to investigate the visco-elastic behavior and consistency of lubrication greases. Valuable information on the visco-elastic behavior, i.e. the storage and the loss moduli, as well as on the stress values at the flow point, i.e. the yield stress, is obtained. Further, rheometers allow for tribological measurements of lubricating greases employing a ball-on-three-plates measuring accessory. Measurements, e.g. Stribeck-curves, at a broad range of speeds especially down to very low speeds with high accuracy are possible. In addition a special ball bearing fixture is available, thus allowing the measurements of friction factors as well as the static friction. Both rheological and tribological measurements on model grease samples in a temperature range from –40°C up to 60°C are presented. The impact of temperature was monitored and a correlation between rheology and tribology was found.

Introduction

Viscometers are used to measure the viscosity of fluids with a simple relation between force and deformation rate. Viscosity of Newtonian fluids is constant independent of the applied shear or deformation rate. Complex fluids in general are non-Newtonian and visco-elastic. For the characterization of such kinds of fluids a sophisticated rotational rheometer is required. In order to cope with the diversity of rheological testing, different applications like polymers melts, food, cosmetics, building materials, coatings, adhesives, to name a few, modern rotational rheometer offers a large flexibility with respect to environmental conditions and the ranges of the applied or measured properties like torque, deflection angle, rotational speed, or normal force.

In shear rheology the sample is confined in well-defined geometries like cone-and-plate, parallel-plate, or concentric cylinders with fixed gaps. This allows for the calculation of respective rheological properties from stress – strain relations relevant for the measuring geometry used [1].

In tribology, on the other hand, are the measuring geometries parts of the investigated tribo-system. Two measuring bodies are pressed together by a normal load and the coefficient of friction (COF) in relation to relative sliding speed is determined.

In shear rheology the surface of the fixtures do not have any influence on the rheological data as long as the conditions of laminar flow are met. In some cases the surfaces are treated or roughened in order to prevent slip and to assure a laminar flow field. Therefore rheology uses the test fixtures to apply deformation onto the sample, whereas they are part of the test specimen in tribological tests.

However, in tribological tests forces, movements and normal loads need to be applied or measured as in the case of rheology. Modern rotational rheometers are equipped with excellent speed and torque control as well as an accurate normal force detector and a precise control of the temperature in a wide range. All these features can also be used for tribology as well, which led to the idea to design an accessory enabling tribological measurements on a conventional rotational rheometer. A rheometer employing a ring on disc setup has been used to measure tribological behavior [2]. Although this device works for some applications it turned out that the lack of backflow of lubricant and the occurrence of isolated friction contacts is limiting its range of use.

The aim of this paper is the twofold. The rheometer setup as it is used to measure the rheological properties of greases and design of the tribological cell and the corresponding accessories is presented. Secondly, the results of the rheological measurements and correlations with tribological measurements are discussed. The discussed applications cover the more traditional measurement of Stribeck curves for various oils, the determination of the rheological and tribological behavior of different greases at 25°C and -40°C and the characterization of grease in ball bearings.

2 Background on greases

Greases are used in a lot of applications. Compared to oils they have a number of advantages with respect to the construction and service of lubricated components. However, due to the visco-elastic behavior of greases there are certain constraints to consider. For example, when greases are used in automotive application the greases should show good performance characteristics over a large temperature range. Car manufacturer for example demand that greases used in cars should perform at temperatures as low as -40°C. Therefore having an instrument and methods to investigate the visco-elastic and the tribological behavior of greases over an extended temperature range is highly desirable. Of special importance from the rheological side are the yield and the flow behavior, i.e. the apparent yield and flow points of the greases. They determine the stresses at which the greases are yielding or flowing, respectively [3]. One method to measure theses stresses, which will be integrated in the DIN standard [4] is a so-called oscillatory strain sweep. In such a measurement oscillatory movements with increasing amplitude of the strain are applied to the sample and the resulting oscillatory stress response is measured. Oscillatory measurements have the advantage that it is possible not only to measure viscous behavior at a constant rotational movement, but giving information on viscous and elastic, and thus, visco-elastic behavior of the sample. More information on rheological measurements and specifically on the strain controlled amplitude sweep can be found for example in [1].

From the tribological side friction at a certain sliding speed and static friction are relevant for a good performance of the greases. Greases are used to eliminate noise and to guarantee a smooth movement. In order to measure static friction, the friction force at which the system is starting to move has to be determined. This is similar to the yield behavior in rheology where the stress value is measured when the sample is starting to move. The friction behavior itself is normally presented in Stribeck curves which correlate the friction coefficient with the sliding speed, the viscosity and the normal load.

3 Instruments and Methods

3.1 Rheometer

An MCR 301 rotational rheometer from Anton Paar was used for all tests. The rheometer employs an air bearing supported electronical commuted synchronous motor (EC-Motor) [5]. The electronical commutation represents a non contact way to excite the rotor and can be used in combination with an air bearing thus enabling measurements at low torques. The EC-motor is sometimes called a brushless DC motor, since the motor current is commutated electronically and there are no brushes or contacts to excite the motor, but rather the motor is excited by special permanent magnets with a high flux density. It is a synchronous motor because the rotor rotates at the same speed, i.e. synchronous, with the stator field. The rotor field is produced by high energetic permanent magnets, which are mounted at fixed positions on the rotor. Since the positions of these permanent magnets are known, the rotor field is known. The EC control makes use of this knowledge. It is possible to adjust the electro-magnetical torque in such a way that it is linear to the total amount of the stator current, i.e. $M \sim I_s$. The applied electrical current is a direct measure of the torque. In addition an optical encoder measures the angle and the speed of the rotational movement.

The instrument controls the rotational speed and measures the resulting torque very accurately. In addition force controlled measurements are possible by applying a torque and measuring the resulting speed. The normal force can be set and recorded during all tests. The MCR 301 rheometer used features the following measurement ranges: rotational speed: 10^{-6} to 3000 rpm; torque: 10^{-7} to 0.2 Nm; normal force: 0.01 to 50 N.

3.2 Temperature control

For rheological testing a Peltier temperature control system was employed. It consists of a Peltier controlled bottom plate and an additional Peltier controlled hood. The upper measurement geometry used was a parallel plate with 25 mm diameter and the sample was located in a 1mm gap between the bottom and the upper plate. In Figure 1 a rheometer and the temperature control system are displayed.

Figure 1: MCR Rheometer and Peltier system with a temperature controlled hood.

The additional use of a Peltier controlled hood ensures a uniform temperature distribution within the sample over the whole measurement range. This is crucial since a temperature gradient within the sample will induce misleading results, as it occurs when for example only the lower plate will be temperature controlled [6]. The same Peltier controlled hood was used in combination with the tribology cell as will be described later.

3.3 Rheological measurement procedure

The zero gap was set at the test temperature and the sample filling was done at +25 °C. The cooling rate was 10 K/min to reach -40 °C. The cooling rate, having a significant influence on the structure at low temperatures, can be altered in order to check different test conditions. After reaching -40 °C the sample was equilibrated for 10 minutes. As inert gas N_2 was used to prevent ice formation. Oscillatory strain sweeps with an angular frequency of ω = 10 rad/s and strain amplitudes from γ = 0.001 % up to 100 % have been performed. With strain sweeps it is possible to investigate the visco-elastic behavior at rest and to determine the apparent yield stresses.

3.4 Tribological measuring setup

For testing tribological properties an accessory was used, which is mounted onto the MCR rheometer. It makes use of the large measurement ranges as well as the motor control mechanism of the rheometer. The setup has been described in detail elsewhere [7]. Here only the main parts are mentioned. The device is based on the ball-on-three-plates-principle (or ball-on-pyramid) consisting of a geometry in which a steel ball is held, an inset where three small plates can be placed, and a bottom stage movable in all directions on which the inset can be fixed. The ball-on-three-plates setup has been used before in a dedicated device to measure static friction coefficients [8]. Figure 2 and Figure 3 depict the tribometer setup schematically and in photos, respectively.

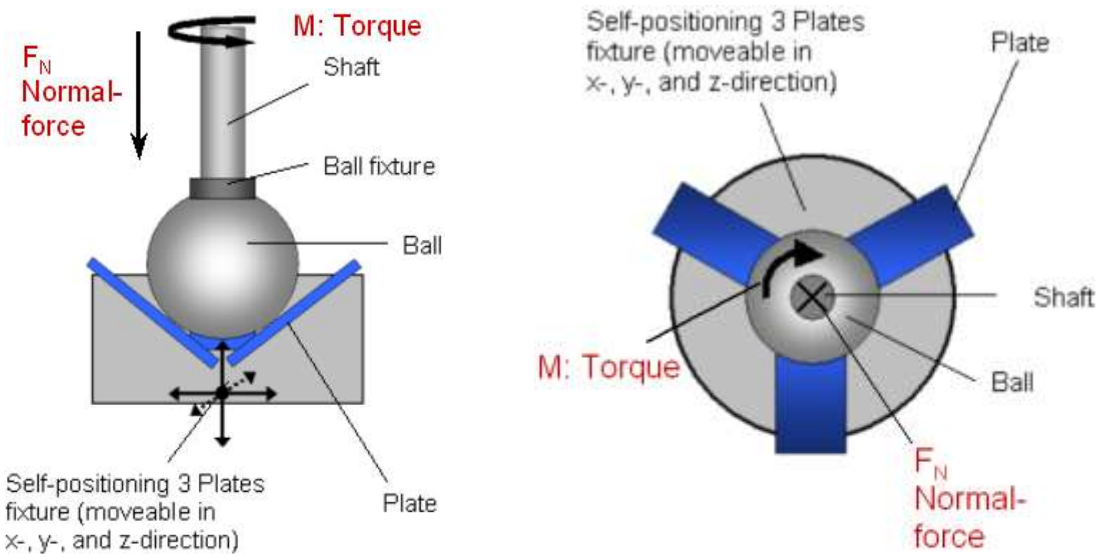

Figure 2: Schematic setup Tribology accessory in side and top view. The torque and the normal force applied by the rheometer are indicated by arrows.

Figure 3: The Tribology accessory without (left) and with (right) additional Peltier hood.

The flexibility of the bottom plate is required to get the same normal load acting evenly on all the three contact points of the upper ball. The rotating sphere is adjusted automatically and the forces are evenly distributed on the three friction contacts. The ball as well as the plates for the inset can be exchanged so that the system can be adapted to desired material combinations.

The rotational speed applied to the shaft is producing a sliding speed of the ball with respect to the plates at the contact points. The resulting torque can be correlated with the friction force by employing simple geometric calculations. The normal force of the rheometer is transferred into a normal load acting perpendicular to the bottom plates at the contact points.

The relations between the normal force (F_N) and the torque (M) of the rheometer to the normal load (F_L) and the friction force (F_F) experienced by each sample are:

$F_L = 2 \cdot \cos \alpha \cdot F_N/3$, $M = \sin \alpha \cdot 3 \cdot F_F \cdot r_{ball}$

with r_{Ball} being the radius of sphere and α the angle of the plates, respectively. The following dimensions have been used: $\alpha = 45$, r_{ball} = ½inch = 6.35mm. Based on the geometrical dimensions the tribological properties can be calculated. The rheometer used as a tribometer has the following measurement ranges (per plate): Normal Load: 0.3 to 23 N; Friction Force: up to 19 N; Sliding Speeds: 10^{-8} to 1.41 m/s.

The tribology setup is temperature controlled by Peltier elements. As for the rheological measurements the same additional Peltier controlled hood can be used for measurements performed other than room temperature. The Peltier hood ensures the same temperature at the bottom plates and at the upper ball. The possible temperature range covers temperatures from -40 up to +200 °C.

3.5 Ball Bearing fixture

Two standards are established for low temperature torque measurements of ball bearings in IP 186 and ASTM D1478 [9,10], respectively, for starting and running torque determination. These standards describe defined measurement procedures on specialized and elaborate instruments using bearings with a diameter of 47 mm, applying an axial force of 45 N and having a fluid circulator temperature control. The measurements should be performed at a temperature below -20 °C where temperature control based on a fluid circulator is not the best choice as the temperature setting is time consuming and there is a certain temperature loss in the tubing. A Peltier temperature control as it is established in rheometers for many years is a much faster and more accurate temperature control solution. It allows for temperatures as low as -40 °C while purging the sample with dry gas preventing ice formation.

Based on these facts a ball bearing fixture for the tribological accessory has been designed. The intension was to make use of the technical capabilities offered by the rheometer and to archive the best possible testing capabilities instead of following strictly the existing old standards. The maximum torque of the rheometer is limited to 0.2 Nm therefore the ball bearing fixture will not be able to cover all applications of the specialized instrument described in the standards. The ball bearing fixture, which is depicted in Figure 4, can be mounted on the tribological accessory instead of the plate holder so that bearings with diameters up to 40 mm can be investigated. The outer ring of the bearing is held by the fixture whereas the inner ring can be moved by the rotor. The conical shape makes the geometry adaptable to various inner radii so that it can be pressed with a specific normal load of up to 50 N onto the inner ring. The motor of the instrument is driving the geometry and measuring the starting and running torques of the ball bearing. The entire system can be temperature controlled by the Peltier system in the range from -40 up to +200 °C.

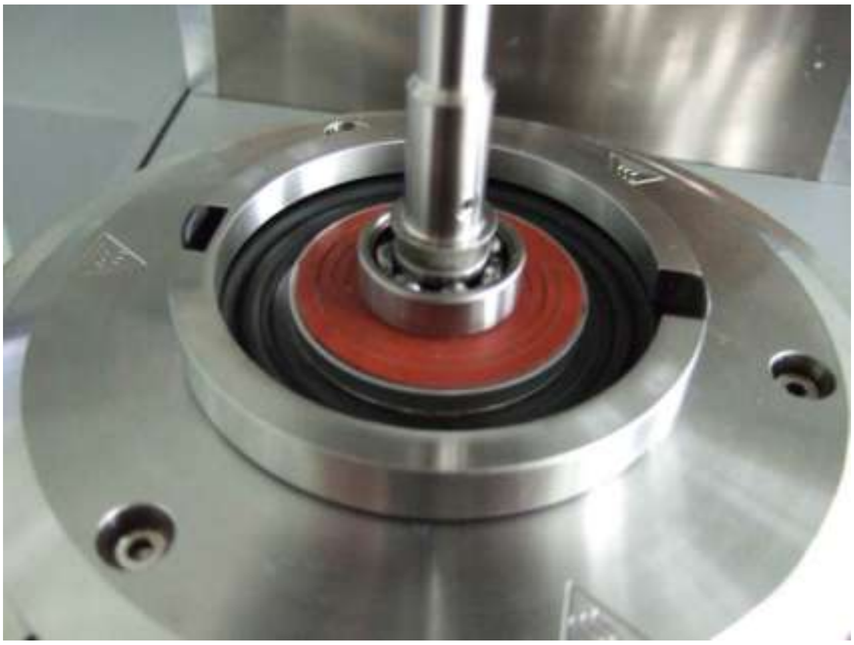

Figure 4: The ball bearing fixture mounted on the tribology cell.

4 Samples

The performance check of the tribology accessory was conducted with plates made of PC (Polycarbonate - Macrolon^{TM}) and POM (Polyoxamethylene). The ball material was stainless steel for all tests. Commercially available Diesel motor oil (0W-40) and penetrating oil were used for lubrication. The Newtonian viscosity values measured at +25 °C were 140mPas for the motor oil and 5.7 mPas for the penetrating oil, respectively. Grease measurements were performed with three different mineral oil based greases using the classification of the American National Lubrication Grease Institute (NLGI): NLGI 0, 1, and 2. The small bottom plates for tribological testing on greases were made of Polycarbonate.

For testing with the bearing fixture ball bearings from SKF/Germany with an outer diameter of 22 mm and inner diameter of 8 mm were used.

5 Measurements and Discussion

5.1 Performance tests of the tribological accessory

In Figure 5 Stribeck curves are plotted in dry and lubricated conditions using a steel ball on POM plates. At the measuring temperature of +25 °C the applied normal load was set to 4.7 N. The speed ramp started from 0.1 and was increased logarithmically up to 3000 rpm. The curve representing the friction partners at dry conditions shows a significant increase of the friction coefficient at low speeds and reaches a COF of 0.27 at larger speeds. Citations of friction factors are rare and vary in case of the steel/POM material combination from 0.2 up to 0.4 [11]. Thus the measured value is in good agreement with literature. Motor oil between the friction partners is decreasing the friction at low speeds showing a good lubrication performance over large speed ranges. The low-viscosity penetrating oil reduces the friction continuously from the beginning finally reaching a similar friction coefficient as motor oil at high speeds.

The different tribological behavior is related to the application of the products. Motor oils are optimized for lowest friction whereas an excellent flow behavior is more important for penetrating oils. Therefore from low to intermediate sliding speeds the motor oil exhibits a much lower friction coefficient compared to the penetrating oil.

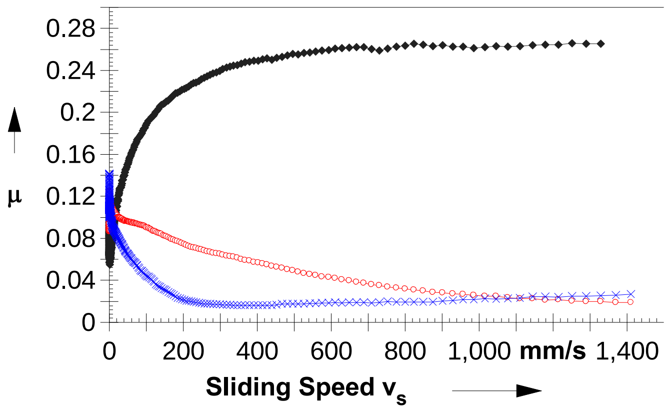

Figure 5: Stribeck curve from 0.1 to 3000 rpm at +25 °C. Steel ball on POM plates, dry (black), lubricated with penetrating oil (red) and with motor oil (blue).

In Figure 6 the curves from Figure 5 are plotted in semi-logarithmic scale. This presentation illustrates the speed performance of the rheometer. Sliding speeds as low as 0.01 mm/s and up to 1.41 m/s can be set and measured. The curves obtained with the lubricated systems are slightly higher at very low speeds compared to the dry contact data. A possible explanation could be adhesive forces of the oil in the boundary region in which a lubrication film is not present.

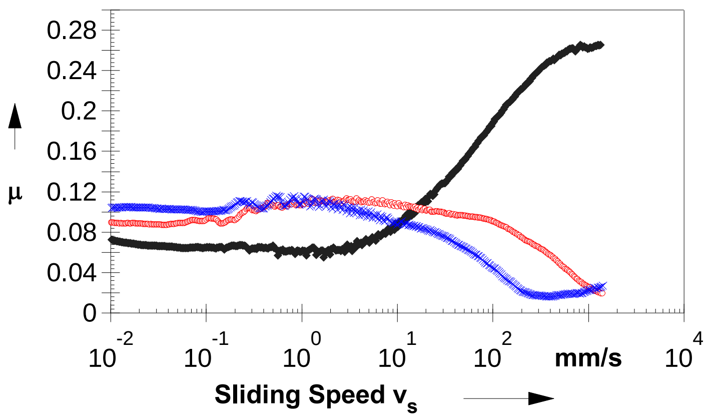

Figure 6: Stribeck curve one a semi-logarithmic scale from 0.1 to 3000 rpm at +25 °C. Steel ball on POM plates, dry (black), lubricated with penetrating oil (red) and with motor oil (blue).

5.2 Rheology of greases

Compared to mineral oils greases are showing a more complex flow behavior because they have a paste-like structure making them suitable for applications where the lubricant should stay in the tribological contact area. In Figure 7 rheological strain sweeps of three greases of the NLGI classes 0, 1 and 2 at +25 °C are shown. In order to show the excellent reproducibility of the method, data of two measurements are plotted for each sample. Shown are the storage modulus G' representing the elastic part, and the loss modulus G'' representing the viscous portion of sample response to the oscillation.

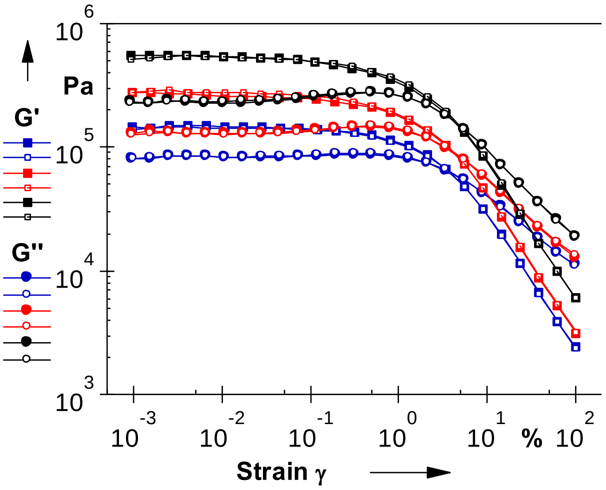

Figure 7: Strain sweeps from γ = 0.001 up to 100 % at +25 °C. Two measurements for each sample:. NLGI 0 (blue) lowest values, NLGI 1 (red) medium values, NLGI 2 (black) highest values.

At small strains G’ is higher than G’’ for all three samples, indicating a solid-like structure. As expected both moduli are increasing for increasing class specifications NLGI 0, NLGI 1 and NLGI 2, respectively.

Figure 8 shows the same type of measurement at -40 °C. Again data of two measurements are shown for each of the three samples. The general behavior and the obtained order according the sample type is the same as at +25 °C, with NLGI 0 having the lowest values of the moduli and NLGI 2 having the highest values, respectively. However, the absolute values of the moduli are significantly higher at -40 °C.

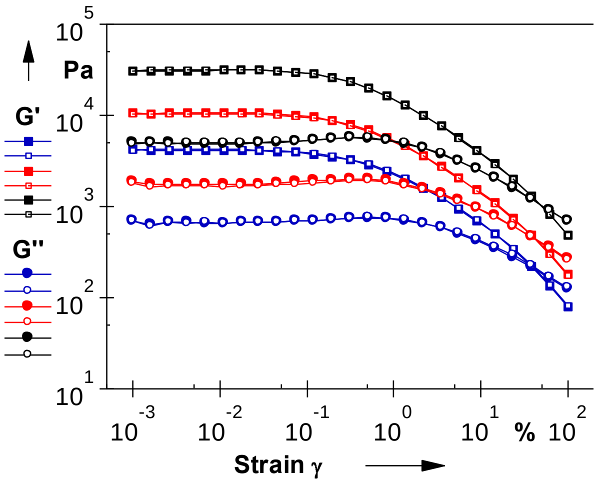

Figure 8: Strain sweeps from γ = 0.001 up to 100 % at -40 °C. Two measurements for each sample: NLGI 0 (blue) lowest values, NLGI 1 (red) medium values, NLGI 2 (black) highest values.

For both temperatures the moduli are decreasing at higher strains. The point where G’ starts to decrease is generally defined as the end of the so-called linear visco-elastic (LVE) range.

In order to evaluate the behavior of the greases during the practical application it is useful to evaluate the moduli as a function of the shear stress. In Figure 9 data from Figure 7 and Figure 8 are plotted versus the shear stress. For clarity reasons only one measurement is shown for each sample and temperature. At both temperatures the stresses are lowest for the NLGI 0 sample and the highest for the NLGI 2 sample, respectively. As can be seen in

Figure 9 the stress values at which the moduli are starting to decrease are lower at +25 °C compared to -40 °C. In terms of shear stress these values can be attributed as the apparent yield point. At higher shear stresses a cross over of the G' and G'' curves can be seen. At strains higher than the cross over point the viscous part, i.e. G'', is higher compared to the elastic part, i.e. G', indicating the samples are liquid-like now showing flow behavior. Therefore the cross over point is also called flow point. The stress values at the flow point are clearly higher compared to the stress value at the end of the LVE-range.

The rheometer software enables the determination of both the yield and the flow point. For the practical use in the automotive industry the stress value at which the lubrication greases are starting to flow is an important property. In Figure 9 the stress values at the flow point (cross over of G' and G'') as given by the analysis method are displayed in addition to the G' and G'' curves. The stress values at the flow point are nicely ordered from 120 Pa for NLGI 0 at +25 °C to 9350 Pa for NLGI 2 at -40 °C.

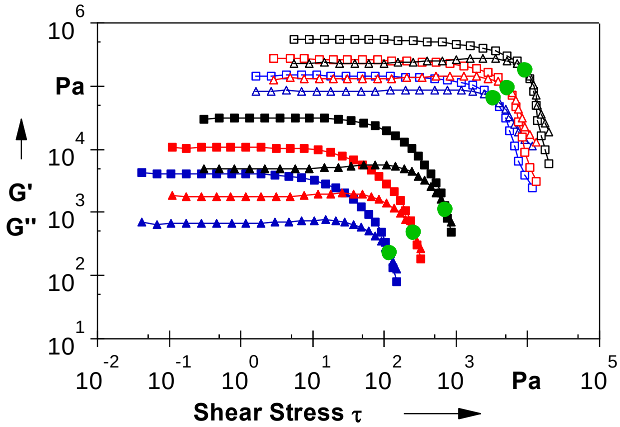

Figure 9: Strain sweeps from $\gamma = 0.001$ up to 100 % at +25 °C (full symbols) and -40 °C (open symbols). Data from Figures 8 and 9 plotted versus the shear stress. The big dots mark the respective flow points of the three greases at the two temperatures.

5.3 Tribology of greases

In Figure 10 the COF as a function of the sliding speed for two greases measured at +25 °C and -40 °C is shown. All data was measured at least 3 times. Good reproducibility was observed. For clarity only one measurement curve per sample and temperature is shown. For both greases, at low sliding speeds up to 5 mm/s the friction values are lower at -40 °C (squares) compared to +25 °C (circles). At higher sliding speeds up to 100 mm/s the friction coefficient is lower at +25 °C compared to -40 °C. A possible explanation of these effects might be given by the rheology data from Figure 9, which shows that at -40 °C the structure of the greases is stronger compared to +25 °C. It seems that due this stronger structure and higher yield stresses a thicker film is present at lower temperatures, resulting in smaller friction values at low speeds. At higher speeds part of the grease in the contact area might be moved out and the stronger yield behavior restricts the flow of grease into the gap. Therefore the film thickness is reduced and the friction increases. This would also explain that at both temperatures the friction value for the NLGI 2 sample increases already at lower speeds compared to the NLGI 0 sample. The NLGI 2 has larger yield and flow stresses compared to NLGI 0.

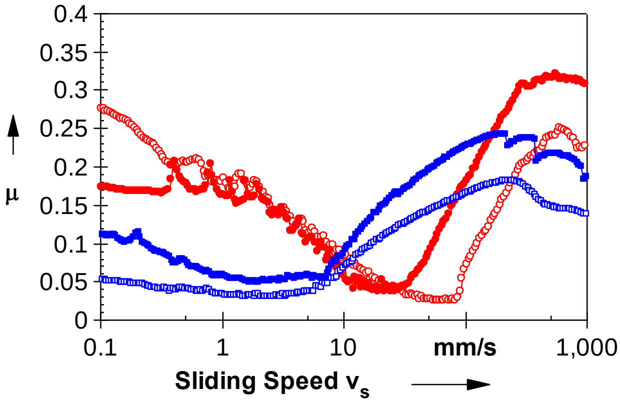

Figure 10: Friction coefficient as function of sliding speed for 2 lubrication greases (NLGI 0: open symbols; NLGI 2: closed symbols) at +25 °C (circles) and -40 °C (squares).

In order to investigate the static friction behavior, measurements were performed. Again a normal load of 4.7 N was used. Before the actual measurement a run-in at a speed of 10 mm/s was performed for 10 min followed by a rest period of another 10 min. In Figure 11 the results are shown. The friction coefficient is plotted versus the sliding speed. At small forces there is practically no effective movement noticeable and the data is scattering around a zero speed, represented by very small speed values. If the force is large enough to overcome the static friction the speed is jumping from zero (or very small values) to large values. At +25 °C (circles) the friction coefficient remains more or less constant up to the maximum plotted speed. However, at -40 °C (squares) a step in speed can be seen as well, and at higher speeds the friction is increasing again. This is in accordance with the increase of the friction coefficient at smaller speeds in Figure 10 at -40 °C compared to +25 °C.

The static friction, which can be taken as the friction value when the big step in the speed is occurring, is smaller at -40 °C compared to +25 °C and it is also smaller for the NLGI 2 sample compared to the NLGI 0 sample at both temperatures. A possible reason might by the larger yield and flow stresses at lower temperatures, which might result in a larger film thickness at the beginning of the test. The same explanation can be used when NLGI 0 and NLGI 2 are compared at the same temperature. NLGL 2 has a larger yield stress and lower static friction value, i.e. a larger film thickness.

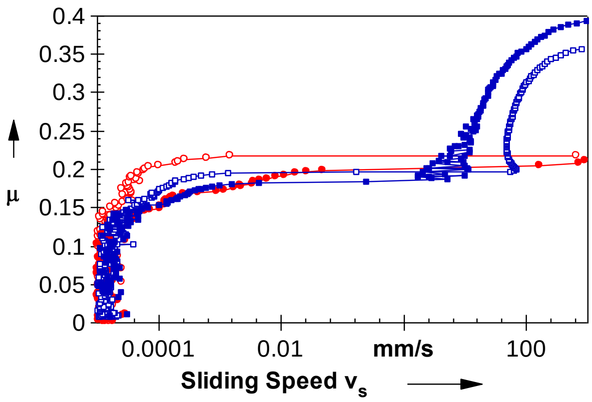

Figure 11: Static friction measurement for 2 lubrication greases (NLGI 0: open symbols; NLGI 2: closed symbols) at +25 °C (circles) and -40 °C (squares).

5.3 Measurements of ball bearings

Ball bearings are often lubricated with greases ensuring lowest possible friction under working conditions. Starting torque, running torque and roll out time after an applied speed are the most interesting parameters.

The settings for the starting torque measurement are the same as for the static friction determination. The torque is logarithmically increased and the occurring speed is measured. Figure 12 shows starting torque measurements at -40 °C, +25 °C and +60 °C. Due to the influence of the yield point of the greases on the starting torque the breakaway torque required is higher at lower temperatures. It has been shown before that the shear stress necessary to overcome the yield point is strongly temperature dependent. The lower the temperature, the higher the yield point and the more force is required to induce flow. Therefore greases for bearings need to be designed carefully as on one hand a good structure is beneficial to keep the grease at the desired position, whereas on the other the yield point prevents the ball from rolling by increasing the starting torque.

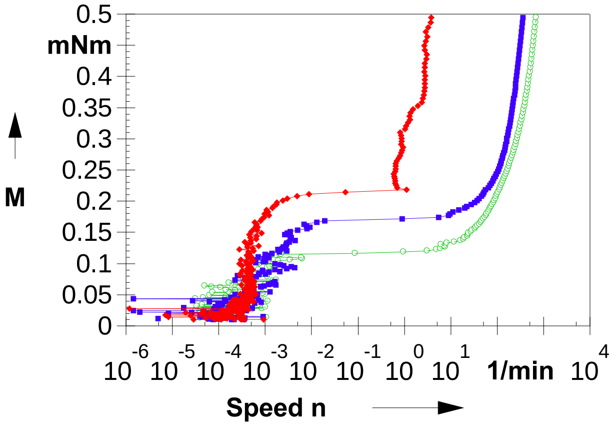

Figure 12: Starting torque measurement for a ball bearing at -40 °C (red), +25 °C (blue) and +60 °C (green).

The rolling friction test presented in Figure 13 is showing a rotational speed ramp from 0.1 up to 3000 rpm. The curves measured at +25 and +60 °C look similar even though the measured values are lower for +60 °C. For both curves the torque increases slightly in the speed range from 0.1 to 100 rpm, whereas at higher speeds a steep slope can be observed indicating a lubrication problem. At -40 °C the lubrication is insufficient right from the beginning since the running torque increases continuously. At 200 rpm the lubricant's viscosity drops due to friction heating resulting in an improved lubrication.

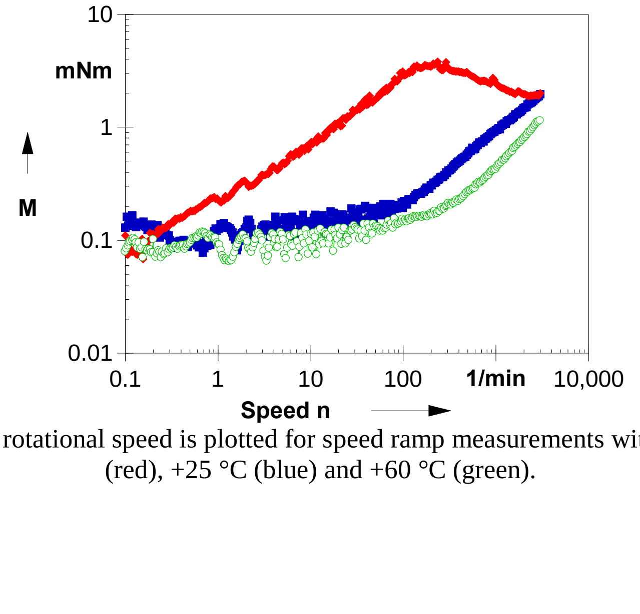

Figure 13: Torque versus rotational speed is plotted for speed ramp measurements with a ball bearing at -40 °C (red), +25 °C (blue) and +60 °C (green).

6 Conclusions

A state of the art rheometer is obviously not limited to the determination of flow behavior. Due to the excellent speed and torque characteristics and the capability of setting and reading normal forces, it can be used for applications it was initially not designed for. This statement is underlined by the presented accessories for tribology and the additional holder for ball bearings. The results obtained in the performance tests of the tribological accessory on dry and oil-lubricated friction partners were reproducible and in good agreement with data from literature. It could be shown that a single instrument can measure Stribeck curves as well as static friction data.

A rheological and tribological study was performed on greases from NLGI class 0, 1 and 2. Significant differences were obtained in strain sweeps in terms of yield point and elastic/viscous moduli between the specified classes. In addition the influence of temperature on the structural strength could be observed as well using a Peltier temperature control system down to -40 °C. It has been shown that structure properties had significant influence on the lubrication behavior. The grease having a high yield point was showing low static friction but the lubrication properties were reduced at high sliding speeds. Therefore for a good static friction performance a high structural strength is beneficial but it also leads to a low speed collapse of the lubrication film and vice versa.

The temperature dependency of the starting friction and the running friction of ball bearings were studied. The grease/bearing combination used had a good friction performance in the range from 0.1 to 100 rpm but at higher speeds as well as at -40 °C the lubrication was insufficient. The importance of both tribology and rheology is obvious for the lubricant development and characterization. The presented Rheo-Tribometer covers both tribometry and rheometry and is therefore a powerful and cost-saving tool for investigations on lubricants.

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