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NLGI

India Chapter

ISSN : 0972-2742

GREASETECH INDIA

A Quarterly Journal of NLGI-India Chapter

Vol. XXIII, No. 2, Oct - Dec 20

GREASETECH INDIA

A Quartely Journal of NLGI-India Chapter

Vol. XXIII, No. 2, Oct - Dec 20

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"Developing Energy Efficient Manual Transmission Fluids (MTFs)"

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Keywords – Energy Efficiency, Low Viscosity, Traction coefficient, Mini Traction Machine, Durability.

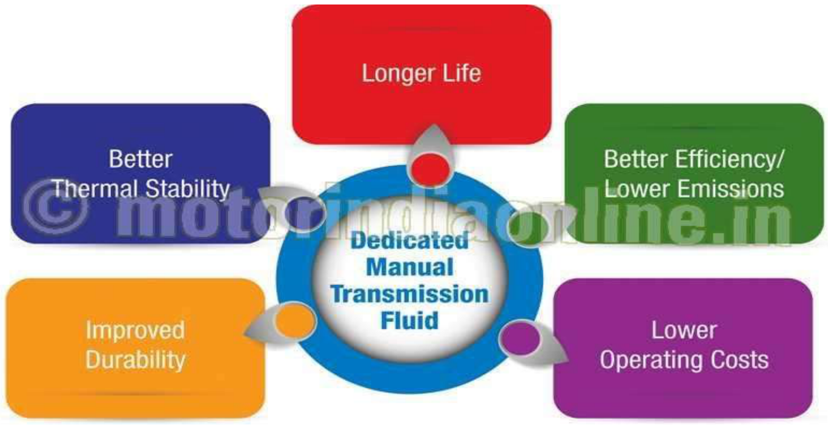
Abstract:
Stricter emission and fuel economy regulation has posed significant challenges on the original equipment manufacturers (OEMs) globally for adopting new technologies to meet the stringent targets stipulated by the government and policy maker. Indian auto industry is also trying to develop cost effective and affordable technologies especially for the indigenous market. Technological changes in engine and driveline especially in their size, designs, metallurgy and new configurations require specific high performance customized lubricants differentiated from conventional one.

In Indian passenger car market, wide span multi grade manual transmission fluids (MTFs) like SAE 80W-90, 75W-90,75W-80 etc are being used in manual transmissions of passenger cars. Strict CAFÉ norms especially for passenger car vehicles led the focus of OEMs on low viscosity manual transmission fluids like SAE 75W and SAE 70W for achieving extra efficiency benefits through transmissions. With this increasing focus towards low viscosity fluids, hardware durability (gears, bearings) and synchronizers friction is still a great concern for OEMs before adopting low viscosity MTFs.

In this paper experimental data is being presented for the development of low viscosity MTFs. Efforts were made to differentiate the fluids for efficiency characteristics by adopting various formulation approaches. Work has been done on SAE 75W manual transmission fluid with special focus on efficiency. Efforts have been made to develop energy efficient product with durability features(like bearing/gear pitting protection, enhanced oxidation and shear stability). This study provided some directions for developing new generation high performance dedicated lubricants meeting Indian OEMs needs.

1. Introduction

Traditional Manual Transmission Fluids(MTFs) are no longer able to satisfy the requirements of latest industry automakers and transmission suppliers. In addition to the key performance parameters set for any modern MTF such as ability to cope with higher power densities and promise of longer service life with enhanced efficiency consumer choice in terms of quality of the shift feel plays an important role in selection of MTF. These parameters are addressed in the lubricant by a combination of appropriate base fluid, an optimized package of additives and other necessary additive components. In terms of synchromesh performance, which is essential for shift quality and feel for a manual transmission, the synchronizer blocker ring is a key part that must have the correct fluid with the right frictional properties in order to engage and dis-engage efficiently. Using the correct fluid for factory fill as well as service fill applications are of paramount importance in order to ensure optimum transmission performance over the extended drain periods used today.



High quality transmission oils must lubricate, cool and protect geared systems, prevent wear, pitting, spalling, scoring, scuffing and other types of damage that result in equipment failure and downtime. Protection against oxidation, thermal degradation, rust, copper corrosion and foaming is also important. Following are the major requirements which should be addressed properly while developing dedicated MTF specific to OEM:

- ✓ Viscosity Profile and shear resistance
- ✓ Optimized synchronizer friction performance
- ✓ Excellent friction durability
- ✓ Reduced gear shift effort– especially at lower ambient temperatures
- ✓ High thermal and oxidative stability
- ✓ Optimized load carrying ability
- ✓ Improved anti-corrosion
- ✓ Bearing protection
- ✓ Improved Transmission Efficiency

Above requirements show that besides key lubricant properties, there is requirement of improved component protection over extended drain periods. The above requirements necessitate development of tailor-made energy efficient fluids for individual transmission types satisfying the need of various synchronizer metallurgies without sacrificing endurance. This paper highlights the development of such an energy efficient transmission fluid using a combination of screening tests carried out in various tribological test rigs for evaluation of efficiency, EP, antiwear and endurance characteristics followed by validation of efficiency in chassis dynamometer. This study has revealed that laboratory data provided good directions for selecting right candidate oil for validation in actual transmission.

2. Balancing Energy Efficiency vis-à-vis Endurance in MTF

Enhancing both energy efficiency and durability by use of suitable MTF is a challenge for oil formulators as they are just like the opposite sides of a coin. OEMs demand for MTF having higher energy efficiency over traditional oil without sacrificing endurance. This necessitates the development of newer chemistries which can offer a balance between these contrasting requirements.

2.1 Role of MTFs in Transmission Efficiency:

Power loss in an automotive transmission consists of gear, bearing, seal and auxiliary losses[1]. Gear and bearing losses can be further categorized to two types (i) no load losses and (ii) load dependent losses. No load losses are mainly related to lubricant viscosity, whereas Load dependent losses depend on transmitted load, coefficient of friction and sliding velocity which in turn demand for suitable additive systems to protect the contact surface. For nominal power transmission, the load dependent losses of the gear mesh are typically dominant. For part load and high speed conditions, no load losses dominate total losses. Thus, Load dependent losses and no load losses have to be addressed for an optimization of the whole operating range of a gearbox.

Oil churning, seal drag and friction accounts for most of losses in gear boxes. Churning losses are due to gear box components moving through the oil sump. The majority of efficiency loss in a vehicle's drive train results from friction generated in the manual transmission and differential gearing. So the oil churning (fluid’s internal friction) and boundary friction are two major parameters which need to be controlled while developing efficient fluids.

2.2 Fluid Friction:

The Stribeck Curve, shown in Figure 1, relates friction between load-bearing surfaces as a function of relative oil film thickness and lubrication regime. Relative oil film thickness is the ratio of film thickness to surface roughness. The thicker the film relative to surface roughness indicates a reduced likelihood of contact by surface asperities. Figures 2 through 4 illustrate the relationship between film thickness and surface roughness.

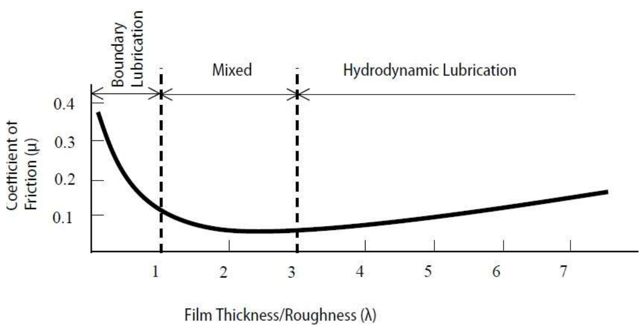


Figure 1. Stribeck Curve.

A gear transmission operates in a combination of Boundary lubrication and mixed lubrication regime when the contacts are in a combination sliding and rolling, as well as thin film elastohydrodynamic lubrication regime at the pitch circle where the surfaces are in rolling contact. Boundary lubrication occurs when the load-bearing surfaces come into contact. Boundary lubrication can occur when the relative speed between mating surfaces is low, there are high loads, or changes in direction. Anti-wear (AW) or extreme pressure (EP) additives can reduce friction and wear to acceptable levels by forming sacrificial solid-film barriers.

A lubrication film completely separates two load-bearing surfaces. With no metal-to-metal contact, machine life depends on oil cleanliness. Friction increases with increasing film thickness. Mixed film lubrication describes the condition where the asperities (peaks) of two surfaces come into contact though a lubricating film is present. The lubrication film is thicker than in boundary lubrication, and it is a combination of full film and boundary lubrication. Friction will be higher than thick film hydrodynamic lubrication, but mixed film lubrication requires AW additives to reduce wear. At the pitch circle, the elastohydrodynamic lubrication regime occurs where the viscosity of oil is important to be able to support the concentrated loads at the counter conformal contacts.

Figure 2. Boundary Lubrication.

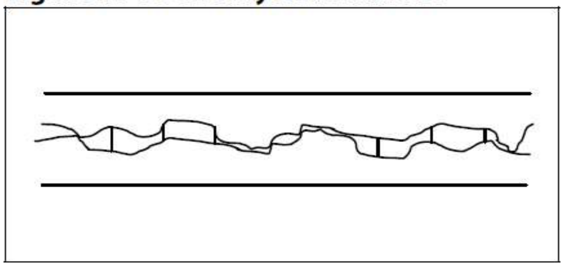


Figure 3. Hydrodynamic Lubrication.

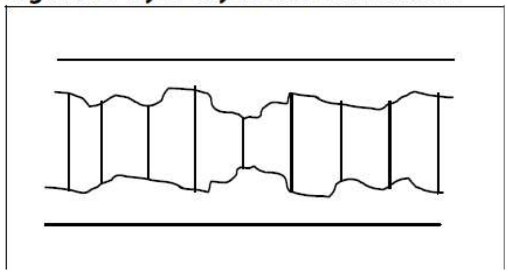
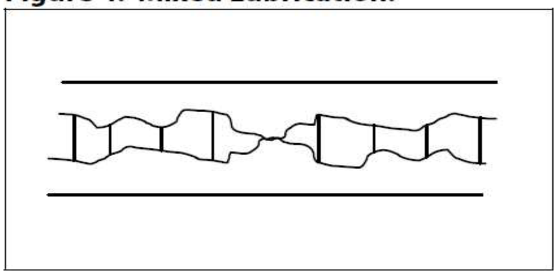


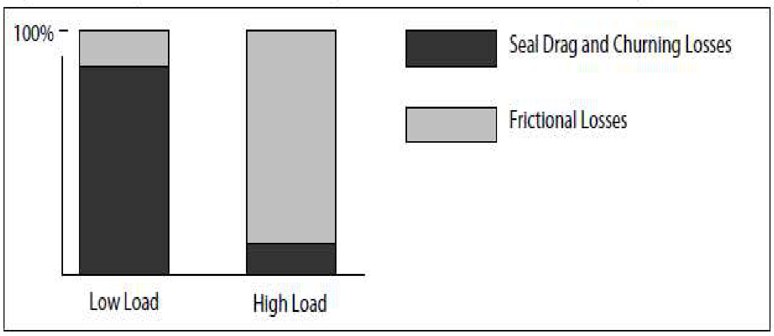
Figure 4. Mixed Lubrication.



2.3 Oil Churning and seal drag:

Churning losses are a function of viscosity. Thicker oil requires more energy to move gears and bearing rollers through the oil. When changing from a SAE 75W oil to a 75W-80, film thickness and viscosity will increase. Seal drag depends on seal material, seal design, and the force imparted onto the shafting by the seal itself. For a gearbox not experiencing shaft deflection, seal drag is independent of load. Seal drag and churning losses are independent of load and as load is increased, these fixed losses make up as smaller portion of losses. Figure 5 visually shows the relationship.

Figure 5. Proportional Relationship of Frictional Losses vs. Seal Drag and Churning Losse



It is evident from above that by reducing viscosity of the oil churning effect of fluid can be reduced but based on literature following are the key factors to be taken care while developing energy efficient MTFs by reducing viscosity of the oil.

1. Low viscosity oils prevent oil churning, thicker oils increase losses through internal fluid friction.
2. Adequate film thickness is required to provide traction benefits.
3. A good Anti-Wear or Extreme Pressure additive package is needed when transient conditions(hydrodynamic to boundary and mixed film lubrication) do not provide an adequate oil film.
4. Synthetic oils and oils with exceptionally low traction coefficient[2] are capable of reducing internal friction losses.

Above is true as far as efficiency is concerned but for durability following are the key facts from the literature which also are required to be considered while developing a balanced fluid for efficiency as well durability:

1. Optimized friction is required for smooth functioning of synchronizers.
2. Lower wear improves durability of transmissions.
3. Appropriate friction modifiers are required in new generation transmissions lubricants where variety of synchro materials (brass, moly and carbon based) is used.
4. Additives play their role based on their surface activation energies under various combinations of speed, load and temperature.
5. It is the synergy among base oil, additives and friction modifiers which deliver an efficient and durable manual transmission fluid.

Experimental:

Traditional oil (Oil 1) of SAE 75W having a good endurance characteristic was developed against reference oil (Oil 5). There was a need to develop energy efficient oil without sacrificing endurance. Three candidate oils Oil 2, 3 and 4 with different additive systems and FMs were developed. All the above candidates of SAE 75W (KV min 4.1 cSt as per SAE J306) transmission oil were developed for efficiency and durability studies by various laboratory methods. Blends were developed using API Group III base oil and MTF additive technology.

3.0 **Physicochemical Data**

Physicochemical data of the above candidate oils vis-à-vis reference oil is shown in Table 1.

Table 1 Comparative Physicochemical Data of Candidates

	Oil 1	Oil 2	Oil 3	Oil 4	Oil 5
KV 100, cSt	4.78	5.17	5.14	5.56	5.23
KV 40, cSt	23.09	24.65	24.50	28.09	26.51
VI	131	146	145	141	132
PP, deg C	-48	-45	-45	-45	-48
Copper corrosion	1A	1A	1A	1A	1A
Rust Protection, D665A @ 60°C x 24 h Distilled Water	PASS	Pass	Pass	Pass	Pass
ISOT Viscosity Change rate (%) @ 100°C	1.00	1.00	1.61	0.9	Not done
ISOT, TAN Increase	0.05	0.07	0.68	0.07	Not done
ISOT, Lacquer Degree	NIL	Nil	Clear	Nil	Not done
BOV @ 100 C, cSt	4.42	4.16	4.16	4.3	Not known

Viscosity Modifier	Nil	Yes	Yes	Yes	Not known
FM2, %	Nil	2X	X	X	Not known
FM1, %	X	X-1	X-1	X-1	Not known

3.1 Tribological Test Data:
A combination of following tribological test rigs was used to evaluate EP, AW, efficiency and endurance characteristics of the candidate oils.

1. Four Ball Test (ASTM D4172), Wear scar dia
2. KRL Shear Stability CEC L 45-A-99
3. Traction coefficient - Mini Traction Machine(MTM)
4. FZG Gear Test(Scuffing Load stage)
5. FZG Pitting Test (Pitting Damage Intervals)

The test results of the above tests are depicted in Table 2.

Table 2 Tribological Data of various Candidates

	Oil1	Oil 2	Oil 3	Oil 4	Oil 5
Four Ball Test (ASTM D4172), @ 40 kg, 1500 rpm, 80 deg C, 1 hour -0.47 mm	0.45	0.50	0.47	0.47	0.50

KRL Shear Stability (CEC L 45-A-99), % viscosity loss	0.6	1.2	1.2	1.2	Not done
FZG Gear Test(Scuffing Load stage)	11th Pass	11th Pass	11th Pass	11th Pass	11th Pass
FZG pitting failure at load stage 9 @ 120 C, hours	64 (Initiation)	32 (initiation)	64 (initiation)	64 (initiation)	60 (initiation)
Traction coefficient by Ball on disc tribometer (Mini Traction Machine MTM)	Graph 1-4	Graph 1-4	Graph 1-4	Graph 1-4	Graph 1-4

4.0 Results and Discussion on Key Tests:

4.1 EP and Endurance Test results

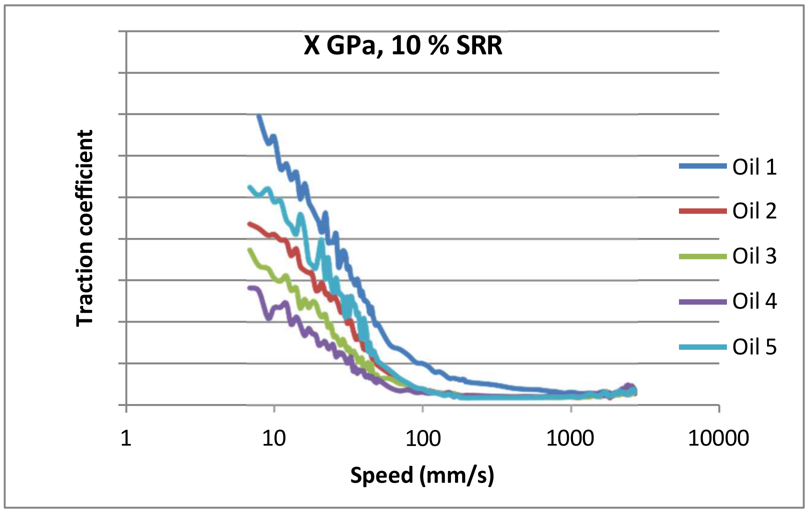
Test results in Table 2 depicts that FZG scuffing load stage does not differentiate the load carrying capacity of the candidate oils. However, long duration pitting tests shows a clear differentiation of the endurance characteristics of the lubricants with Oil 1, 3 and 4 showing better endurance characteristics than reference oil(Oil 5), whereas oil 2 showing inferior endurance characteristics at high temperature. Thus, Oil 1,3 and 4 are expected to give better endurance characteristics in field. MTM traction tests was used to see the energy efficiency credential of these candidate oils vis-à-vis reference oil for short listing of EE transmission oil.

4.2 Traction Studies by Mini Traction Machine (MTM) for evaluating EE

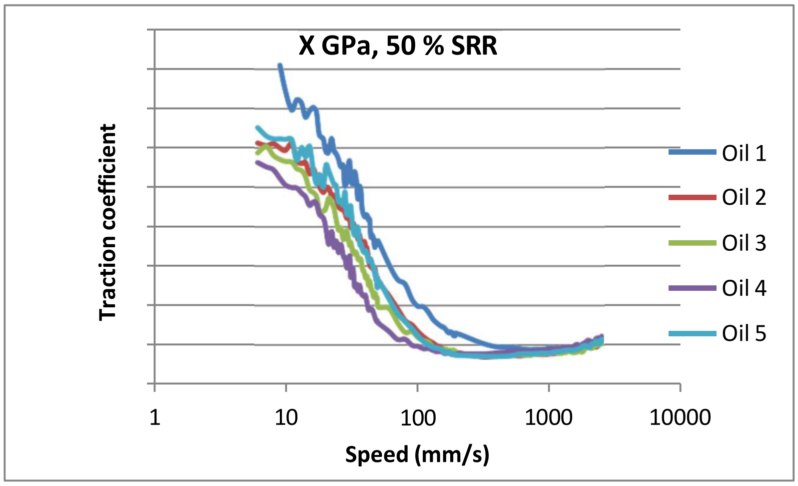
MTM which is a ball and disc tribometer is widely used technique for the traction mapping of lubricants under conditions commonly found in internal combustion engines. In this set up traction coefficient can be measured and a complete picture of all lubrication regimes can be obtained at various slide roll ratios, various temperature range and at different load conditions. In the standard configuration the test specimens are a 19.05mm (3/4 inch) steel ball and a 46 mm diameter steel disc. The ball is loaded against the face of the disc and the ball and disc are driven independently to create a mixed rolling/sliding contact. The frictional force between the ball and disc is measured by a force transducer. Additional sensormeasure the applied load, the lubricant temperature and (optionally) the electrical contact resistance between the specimens and the relative wear between them.

Test had been conducted at 60 deg C (10% slide roll ratio+ X GPa load, 50% slide roll ratio + X GPa load, 10% slide roll ratio+ 2X GPa load, 50% slide roll ratio + 2XGPa load). Comparative traction coefficients of oil 1 to 5 have been plotted in Graph 1-4.

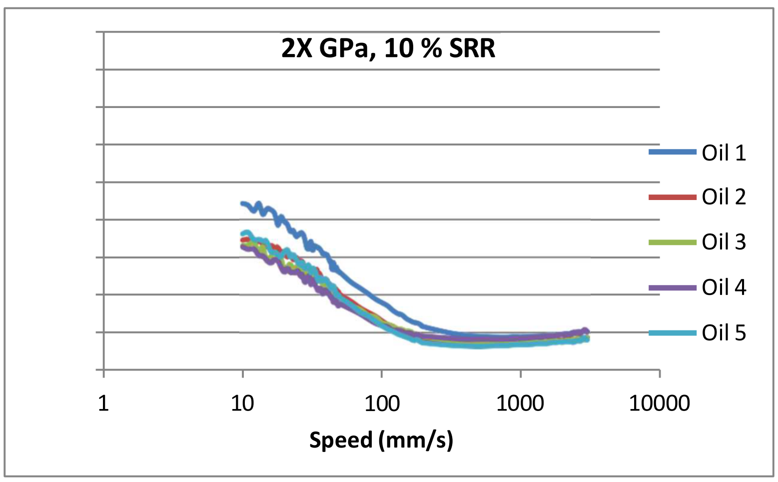
Graph 1 MTM Traction coefficient at 60 deg C



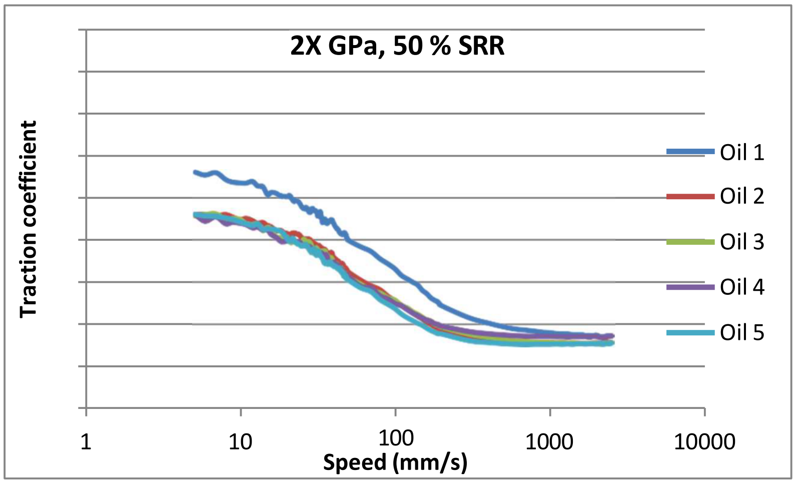
Graph 2 MTM Traction coefficient at 60 deg C



Graph 3 MTM Traction coefficient at at 60 deg C



Graph 4 MTM Traction coefficient at 60 deg C



From the above graphs following may be inferred:

1. Oil 1 is having highest traction coefficient.
2. Under boundary conditions (low speeds) traction coefficient is decreasing in the following order.
   Oil 1 > Oil 5 > Oil2 > Oil 3 > Oil 4
3. At slightly higher speeds Oil 2 ,3, 4 and Oil 5 behave same and Oil1 still has higher traction coefficient.

The above results depict that Oil 3 and 4 are expected to give better energy efficiency as compared to Oil 1 as well as reference oil (Oil 5) without compromising endurance characteristics.

In order to further check the additive-additive interaction of two types of FMs (FM1 and FM2) at different temperature and load stage, a 4 ball wear test was carried out at a severe condition.

Standard ASTM test result of all the oils @ 40 kg, 1500 rpm, 80 deg C, 1 hour - showed wear scar dia values varying from 0.45 to 0.50. In order to further differentiate and understand the effect of temperature and load, Four ball studies conducted at higher load (60 kg); higher speed (1800 rpm) and elevated temperature (80 and 120 deg C) for the optimized oils i.e. oil 3 and oil 4. Figure 1 represents the comparative data of oil3 and Oil 4 at 80 and at 120 deg C at various FM dosages.

Fig 1 Four Ball Test at 80 and 120 deg C results. From the above data following can be inferred:

At higher temperature(120 deg C) WSD is low and at lower temperature(80 deg C) WSD is higher. This shows both types of FMs are competing for the surface at lower temperature whereas at higher temperature probably FM1 is getting activated providing a protection to the contact surface.

Further tests on Oil 1,2,3 and 4 in actual transmission system vis-à-vis reference oil correlated well with the MTM traction result over an entire operating range of test conditions.

Conclusions:

Above study offers a clear direction for the development of new generation dedicated manual transmission fluids. Some of these points are as below:

1. Reducing viscosity always does not lead to improvement of efficiency. Oil 1 in spite of having lowest viscosity was inferior in traction performance (efficiency). This may help in reducing no load loss and improvement of efficiency in a narrow range of operating conditions.
2. Film forming properties of the lubricant play key role in maintaining traction performance.
3. Both base oil with low traction properties and suitable anti wear additives are required to maintain the film thickness under various operating conditions of temperature, speed and load.
4. Viscosity modifiers are needed for improving film formation. Suitable friction modifiers are further needed to again enhance the performance.
5. Surface competition among surface active additives plays important role in maintaining anti wear performance.
6. More data may be required to be generated for evaluating transmission efficiency performance in actual vehicles.

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2. Impact of Viscosity Modifiers on Gear Oil Efficiency and Durability SAE Int. J. Fuels Lubr. 5(1):470-479, 2012, doi:10.4271/2011-01-2128

Performance evaluation of nano additives in gear oil applications

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Abstract:
Nano fluids formulated with suitable nano additives to improve the tribological properties of lubricants. The performance benefits include increased thermal properties, oxidation life and tribological performance of the lubricant in terms of friction reduction and improved anti-wear. In the present study metallic nano additives were synthesized and dispersed into industrial and automotive gear lubricants. The dispersion stability of the nano fluids is established by suitable UV visible spectroscopy method. The tribological properties of synthesized metallic nanoparticles compared with commercially available nano additives (moly-graphene additives) in Industrial and Automotive gear oils by using four ball tribo tester under ASTM D4172 B standard. The tribological data indicate the improved performance of gear oils compared to existing gear oils.

Keyword: Nano additives, Gear Oil, Tribology

1.0 Introduction

In industries, every mechanical system required proper and efficient lubricant for lubrication. Thus, lubricant additives help in enhancing the performance of lubricating oils. The combination of different type of additives and their doses are determined by type of lubricants used. The conventional lubricant additives, containing sulphur or phosphorous plays an important role in gear lubricants. As gear oils in general are used for longer periods of operation in automotive applications and in challenging environments for industrial application, anti- wear and friction reduction are prime improvement and any improvement factors is always a welcome challenge. Metallic or non-metallic nano-particles can be used in synergy with existing as lubricant additives in gear oils to enhance the tribological properties. In this regard, metallic and non-metallic nano-particles have been evaluated for its tribological performance along with lubricant additives in gear fluids. A large number of papers have reported that the addition of nano-particles significantly reduced the friction and anti-wear property in industrial lubes . Various nano-additives such as Graphene, MoS_2, SiO_2, CuO, TiO_2, Al_2O_3 and others nano-particle used as nano-additive in various lubricating oils. Most of these nano particles have been dispersed in base oils and formulated lubricants using variety of dispersants like Tween 20, Tween 60, Span 60, Sodium Dodecylsulphate, Sodium Oleate Oleic acid etc. K. Prabu et al. [1] used Tween-20, Tween 60 and Span 20 dispersant to improve the dispersion stability of Al_2O_3 nano-particles in gear oils and reported that 0.2% Al_2O_3 significantly improve the anti-wear property of gear oil. Cai-xiang et al. [2] used Tween-20, Tween 60, Span 20 and sodium dodecylbenzenesulfonates dispersant to improve the dispersion stability of CeO_2 and TiO_2 nano-particles in 500SN base oil and investigate the anti-friction and anti-wear properties. Zhang et al. [3] used oleic acid functionalized graphene nano-additive in gear oil and studied the friction, wear and load carrying behaviour of gear lubricant. Guo et al. [4] used non-functionalized graphene nano-additive in polyalphaolefin (PAO) and studied the friction, wear and load carrying behaviour. Kole et al [5] used Oleic acid functionalized Cu nano-additive in gear oil to measure the thermal conductivity of gear oil. S. Bhaumik et al. [6] used oleic acid functionalized CuO nanoparticles and Graphite micro powder in 460 cSt mineral base oil to friction and wear behaviour. F. Ilie et al. [7] used oleic acid functionalized TiO_2 nano-particles in mineral base oil and investigate the friction and wear behaviour of TiO_2 nano-lubricants.

On the basis of literature review it has been observed that dispersed nano-particles reduces friction and wear properties of lubricating oils. Significantly in variety of base oils and lubricants CuO, MoS_2 and Graphene nano-additive have shown promising results in enhancing the tribological properties of lubricants. However, the studies on CuO, MoS_2 and Graphene additives have still needed to optimize concentration in commercial available gear oils and with improved the dispersion stability of nano-particles in lubricating oils. The present paper focusses on the improved dispersion stability of nano-particles by the surface functionalization technique in gear oils and comparing the tribo-performance in terms of anti-friction or anti-wear properties.

2.0 Experimental Section

2.1 Synthesis of CuO nano-additive

The CuO nanoparticles were synthesized using co-precipitation method [8]. For the synthesis of CuO nano-particles, $CuSO_4.5H_2O$ and NaOH was used as a precursor. The synthesized CuO nano-particles were further chemically treated with oleic acid with molar ratio 1:5 at 180°C for 6 hours. The oleic acid functionalized CuO nano-particles in terms of oleate were blended as such in selected lubricating oils.

2.2 Selection of lubricant and nano-additive

In present study, an automotive gear Oil (GL-4 80W90) and an industrial gear oil (ISO-VG-320) were selected to investigate the performance behaviour of oleic acid functionalized CuO nano-particles and commercially available Moly-graphene (Moly-Gr) base additive (recommended dose: 3w/w%). The GL-4 80W90 is a multigrade oil and used in regular and turbo-charged vehicles while ISO VG-320 oils used in heavy duty enclosed gear drives under heavy/shock load conditions.

2.3 Preparation of nano-lubricant

The concentration of nano-additive was selected on the basis of tribo-experiments. The tribo result optimized the concentration of nano-additive to prepare the nano-lubricants. An ultrasonic shaker was used for homogeneous dispersion of nano-additive in lubricating gear oils. The concentration of nano-additive used in the selected gear oil was tabulated in table 1.

S. No.	Nano-lubricants	Coded as
1	GL-4 80W90	G-1
2	GL-4 80W90 + 0.005% CuO	G-2
3	GL-4 80W90 + 0.01% CuO	G-3
4	GL-4 80W90 + 1.5% Moly-Gr	G-4
5	GL-4 80W90 + 3.0% Moly-Gr	G-5
6	ISO VG-320	S-1
7	VG-320 + 0.005% CuO	S-2
8	VG-320 + 0.01% CuO	S-3
9	VG-320 + 1.5% Moly-Gr	S-4
10	VG-320 + 3.0% Moly-Gr	S-5

2.4 Dispersion Stability

The dispersion stability of prepared nano-lubricants was determined by UV-visible spectro-photometer. The UV-visible technique provides quantitative assessment of dispersion stability as there exists linear relationship between the particle concentration and the absorbance of suspended particles. UV-visible spectra were recorded with UV-2600 spectrometer in the region of 250-500 nm wavelengths.

2.5 Physico chemical properties of prepared nano-lubricants

The physicochemical properties of prepared nano-lubricants were determined using ASTM standard test methods such as (i) Density ASTM D-1298, (ii) Kinematic viscosity ASTM D-7042, (iii) Viscosity Index ASTM D-2270 (iv) Flash Point ASTM D-92, and (v) Copper Corrosion ASTM D-130. The blending of nano-additive (functionalized CuO nanoparticles and commercially available Moly-Graphene additive) into selected gear oils show very negligibly affect in physic-chemical properties. The density, viscosity, viscosity index have almost negligible change. Similarly the flash point and copper corrosion almost unchanged. The result of physico-chemical properties of prepared nano-lubricants are tabulated in table 2 & 3.

Table 2: Physico-chemical properties of prepared Automotive Gear Oil

S. No.	Characteristic Properties	G-1	G-2	G-3	G-4	G-5
1.	Kinematic Viscosity at 40°C	159.4	159.22	158.27	160.6	161.1
2.	Kinematic Viscosity at 100°C	15.66	15.69	15.69	16.22	16.71
3.	Density at 29.5°C	0.882	0.882	0.882	0.883	0.883
4.	Viscosity Index	100.1	100.5	101	105.3	110.2
5.	Flash Point, °C	240	230	230	232	232
6.	Copper Corrosion	1a	1a	1a	1a	1a

Table 3: Physico-chemical properties of prepared Industrial Gear Oil

S. No.	Characteristic Properties	S-1	S-2	S-3	S-4	S-5
1.	Kinematic Viscosity at 40°C	319.5	319.18	317.52	307.68	305.51
2.	Kinematic Viscosity at 100°C	24.34	24.43	24.38	24.28	24.41
3.	Density at 29.5°C	0.888	0.888	0.888	0.889	0.889
4.	Viscosity Index	97.1	97.6	97.8	99.6	101.3
5.	Flash Point, °C	226	234	226	228	228
6.	Copper Corrosion	1a	1a	1a	1a	1a

2.6 Tribological Testing

The tribo testing in terms of coefficient of friction (COF) and wear of prepared nano-lubricants were done by using four ball tribo tester under ASTM D-4172 B test procedure.

3.0 Result & Discussion

3.1 Dispersion stability of CuO nanoparticles

The evaluation of dispersion stability of oleic acid functionalized CuO or Moly-Graphene based nano-lubricant were measured by recording the UV-visible spectra of the prepared nano-dispersion. The respective absorbance Max for the two nano dispersions as evaluated in the suitable UV region and the dispersion stability of the nano dispersions were recorded over a period of time. The results were reported in Figure 1(a) and 1 (b) shows the absorbance of nano-dispersion with respect to concentration of nanoparticles and over the time period respectively. The figure 1(a) show the linear relationship between the concentrations of nano-particles, absorbance increase the concentration of nano-particle absorbency increases. It is clearly observed that the absorbency decreases with decrease the concentration of dispersed nano-particles. The figure 1(b) shows the maximum UV absorbency of prepared nano-fluids over the time period of 28 days. It is clearly observed that the absorbance decrease over the time. However, the decrease is very nominal which indicate that the oleic acid functionalized CuO nanoparticles are stable in gear oil and can be used as lubricant additive.

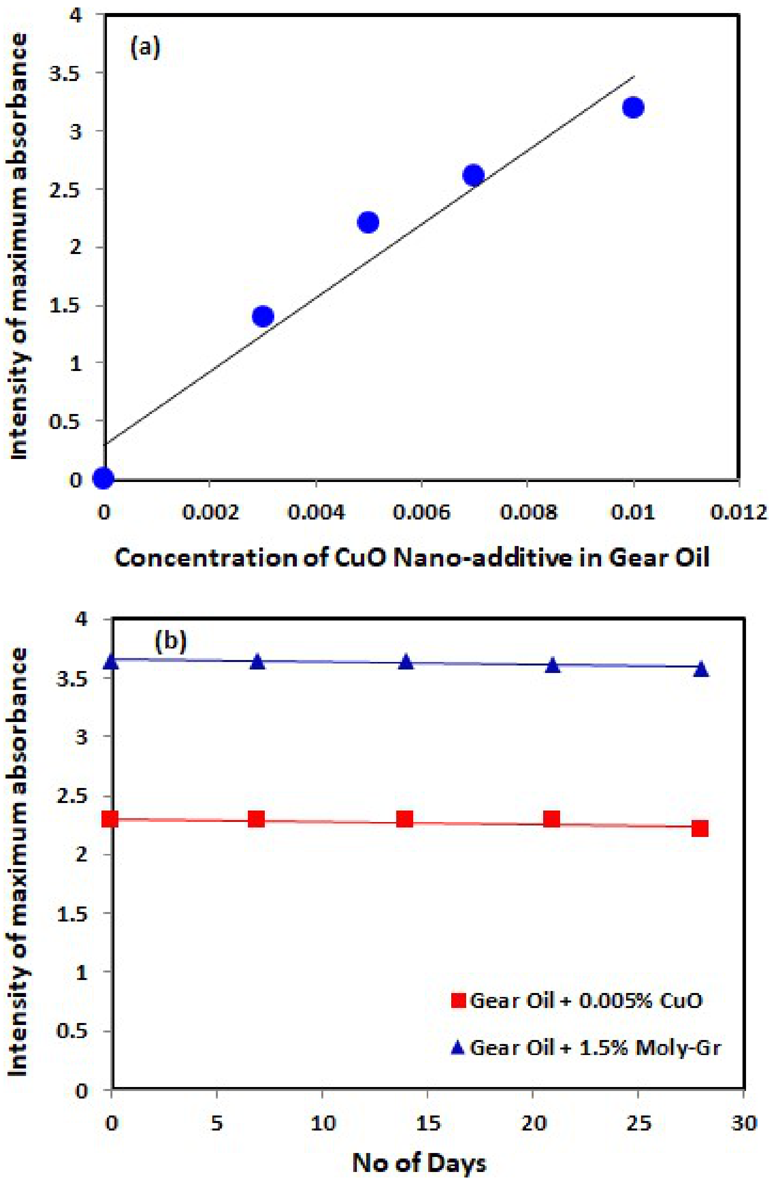

Figure 1: Dispersion stability of blended nano-lubricants in terms of Intensity of maximum absorbance with respect to (a) concentration of nano-particles and (b) number of days.

3.2 Tribo-performance of prepared nano-lubricants

3.2.1 Anti-friction Property

The anti-friction property of prepared CuO and Moly-Gr nano-lubricants in terms of coefficient of friction (COF) is shown in figure 2(a) & 2(b) respectively. The figure 2(a-b) shows the variation of average coefficient of friction observed at the end of the test. The results clearly observed that the COF of selected gear oils without containing nano-additive is higher than prepared nano-lubricants. The friction and wear properties of 0.5% oleic acid blended gear lubricant showing negligible change when compare with existing gear lubricants. The 0.005% CuO nano-lubricant improves the anti-friction property of automotive and industrial gear oil by 18.1% and 35.8% respectively. While 1.5% Moly-Gr nano-lubricant improves the anti-friction property of automotive and industrial gear oil by 25.4% and 41.1% respectively. The further addition of CuO and Moly-Gr additive in gear oil is showing less improvement in anti-friction property. Therefore, on the basis of tribo results, the optimum dose of CuO and Moly-Gr additive in gear oil is 0.005% and 1.5% respectively.

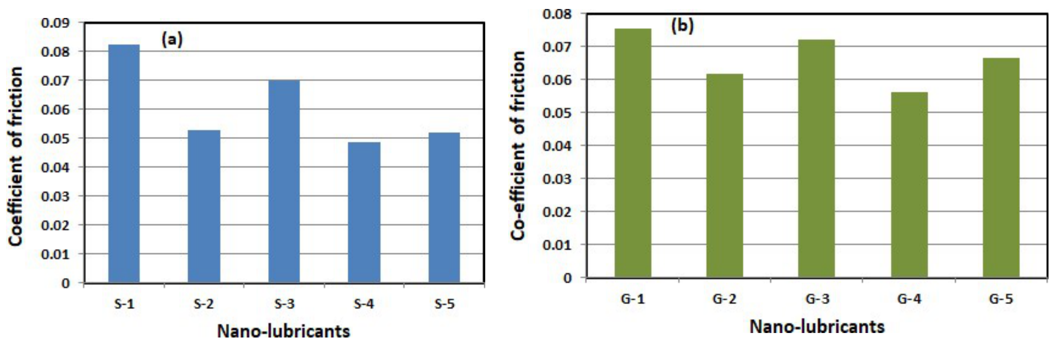

Figure 2: The variation of coefficient of friction of prepared nano-lubricants (a) Industrial gear oil (b) Automotive gear oil

3.2.2 Anti-wear Property:

The anti-wear property of prepared CuO and Moly-Gr nano-lubricants in terms of wear scar diameter (WSD) is shown in figure 3(a) & 4(b) respectively. The figure 3(a-b) shows the variation of average WSD observed at the end of the test on test specimen steel balls. The results clearly observed that the WSD of selected gear oils without containing nano-additive is higher than prepared nano-lubricants. The 0.005% CuO nano-lubricant improves the anti-wear property of automotive and industrial gear oil by 14.9% and 25.6% respectively. While 1.5% Moly-Gr nano-lubricant improves the anti-friction property of automotive and industrial gear oil by 4.97% and 17.8% respectively. The further addition of CuO and Moly-Gr additive in gear oil is showing less improvement in anti-wear property.

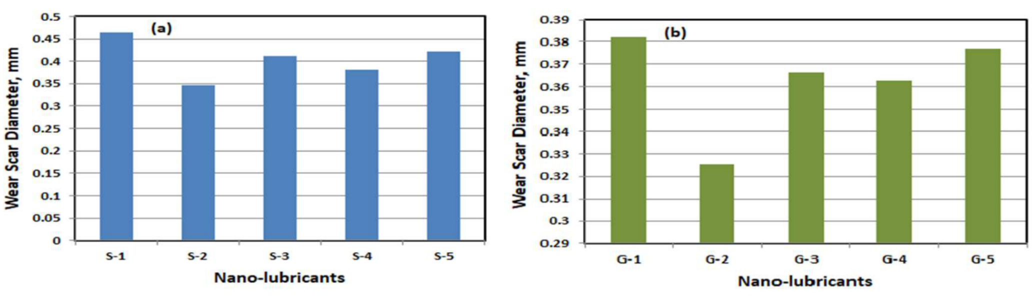

Figure 3: The variation of WSD of prepared nano-lubricants (a) Industrial gear oil (b)

Automotive gear oil

The images of wear scar diameter observed on the used test specimen steel balls are shown in figure 4 (a) & (b). It is observed from figure that the specimen lubricated with nano-additive containing gear oils have relatively smaller wear scar as compared to without nano-additive containing gear oils. At the end of the tribo test the observed average value of COF and WSD are tabulated in table 3.

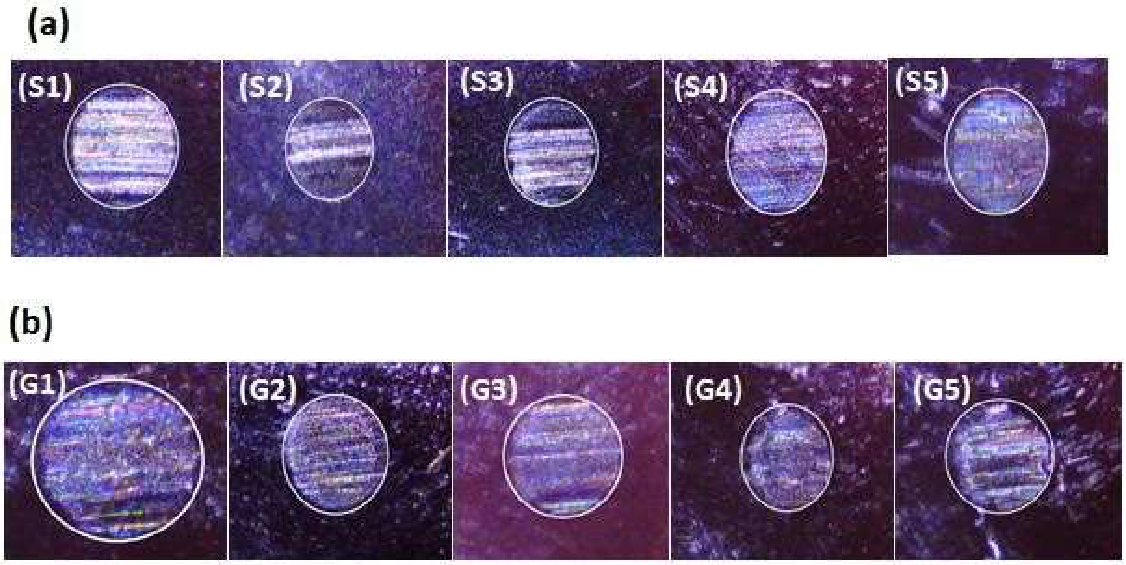

Figure 4: Wear Scar diameter observed on the used ball test specimens (a) Industrial gear oil (b) Automotive gear oil

Table 4: The variation of average value of COF and WSD as per standard ASTM D 4172 B

S. No.	Nano-lubricants	COF	WSD	% Changes	
				COF	WSD
1	G-1	0.0755	0.382	-	-
2	G-2	0.0618	0.325	18.1	14.92
3	G-3	0.0721	0.366	4.5	4.18
4	G-4	0.0563	0.363	25.4	4.97
5	G-5	0.0667	0.377	11.65	1.3
6	S-1	0.0823	0.465	-	-
7	S-2	0.0528	0.346	35.8	25.6
8	S-3	0.0699	0.411	15.1	11.6
9	S-4	0.0485	0.382	41.1	17.8
10	S-5	0.0521	0.421	36.7	9.5

3.0 Conclusion

The present paper evaluated the tribo-performance of Nano-additive in automotive and industrial gear oil lubricants using 4-ball tribo tester under point contact geometry. In the present paper oleic acid functionalized CuO and commercially available Moly-Graphene nanoparticle used as nano-additive in different concentrations. The dispersion stability of CuO and Moly-Gr nano-lubricants was assessed by the UV-visible technique. The optimum concentrations of CuO and Moly-Gr nano-additive on tribo-performance of nano-lubricants have been investigated. The results reveal the following:

- The functionalization of CuO nano-particles with oleic acid results in stable dispersion of CuO nano-particles in gear oil lubricants.
- In automotive gear oil, the blending of 0.005% CuO nano-additive reduces the COF and WSD by 18.1% and 14.92% respectively.
- In industrial gear oil, the blending of 0.005% CuO nano-additive reduces the COF and WSD by 35.8% and 25.6% respectively.
- In automotive gear oil, the blending of 1.5% Moly-Gr nano-additive reduces the COF and WSD by 25.4% and 4.97% respectively.
- In industrial gear oil, the blending of 1.5% Moly-Gr nano-additive reduces the COF and WSD by 41.1% and 17.8% respectively.
- The reduction of COF and WSD of CuO and Moly-Gr nano-additive can be attributedto the formation of efficient boundary films.
- The formed boundary film separate the surface in contact and results in the smaller wear scar as compared to larger wear scar observed in case of gear lubricants.

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Dispersion In Bentonite And Cas Solids In Lithium Complex Greases

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Abstract

Today's highly competitive market demands automotive and heavy-duty industries to increase efficiency, reduce downtime and emission. Core components of various mechanisms are exposed to extreme conditions: temperature, load, and vibration. To meet industry requirements the use of the high-performance additive for lubricant and grease production, that can protect equipment under extreme conditions, has significant importance.

Industries widely use solid particles to enhance its tribological properties such as extreme pressure (EP), wear and friction. Micron size and platelet/lamellar structure particles of WS2, MoS2, and PTFE have been known as a good lubricious solid and widely used in industrial applications. Novel fullerene-like inorganic nanoparticles of tungsten disulfide (IF-WS2) have a close caged (spherical) structure and are considered to be excellent Antiwear, antifriction and EP additives. Due to particle's nanosize the treat rates are the order of magnitude lower than for conventional platelet particles. The unique spherical shape of IF-WS2 nanoparticles with a hollow core allows withstanding high impacts (up to 35 GPa) by absorbing shock, increase EP properties and reduce wear and coefficient of friction up to 2 times.

This paper shows a comparative study of IF-WS2, MoS2 and PTFE particles in mineral and full synthetic Lithium complex greases. Extreme pressure (EP), Antiwear (AW) and antifriction (AF) properties, as well as other physical properties of greases, have been reported.

Introduction

Tribological properties such as antifriction, anti-wear and EP (Extreme Pressure) properties in greases play a very important role in industrial applications such as mining, automotive, steel production, etc. There are many additives in the market that are reducing wear and friction (i.e., MoDTP, MoDTC, ZnDTP, 2H-MoS2) and increasing EP properties (i.e., heavy metals, sulfur, phosphorous).

Anti-wear characteristics of ZnDTP (Zinc dialkyl-dithiophosphate) are attributed to the formation of phosphate films that can react with abrasive iron oxides [1]. On the other hand, MoDTC (molybdenum dialkyl-dithiocarbamate) has been used primarily as a friction modifier due to the formation of MoS2 at high Hertzian pressure points. MoDTP (molybdenum dialkyl-dithiophosphate) possesses combined anti-wear and friction reduction properties [2]. Both MoDTP

and MoDTC require the presence of temperature and friction in order to start the generation of MoS2 layers. 2H-MoS2 particles (Figure 1a [3]) are platelet shape micron size particles that in the presence of shear stress exfoliate layers of MoS2 thus reducing friction.
IF-WS2 nanoparticles (Figure 1b) are novel spherical particles that were invented in 1992 by Professor Reshef Tenne in the Weizmann Institute of Science. These particles currently are produced on a commercial scale and are available in various forms of dispersions (water, paste, oil). In this paper, the commercially available paste dispersion of *IF*-WS2 nanoparticles has been tested in LiX grease. Wear, friction, and EP characteristic along with other physical properties have been evaluated.

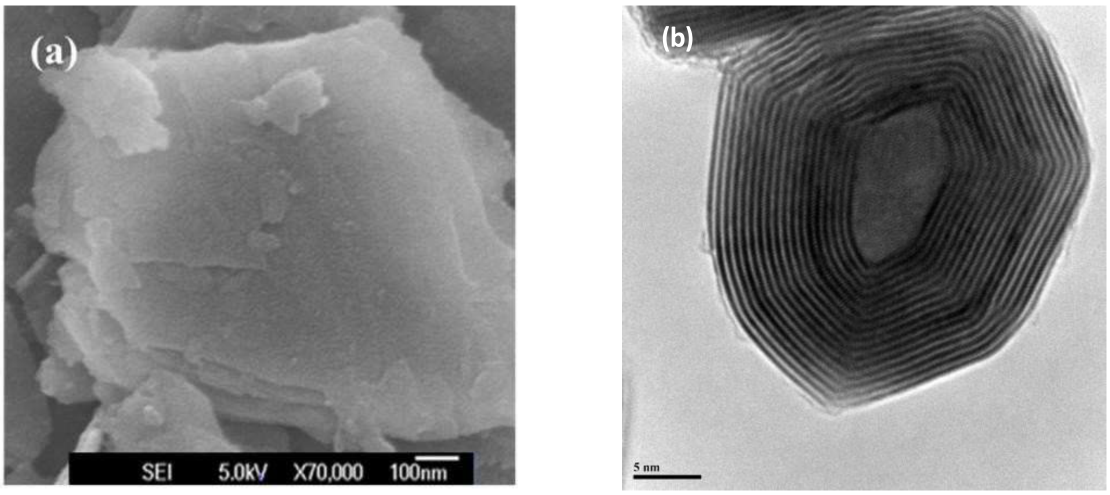

Fig. 1. SEM image of 2H-MoS2 micron size particle [3] (a) and closed-caged nanoparticle of IF-WS2 (b)

Due to their morphology and size, *IF*-WS2 nanoparticles provide excellent shock absorbing properties along with antiwear, anti spalling and pitting, [4] friction reducing, and extreme pressure properties. Figure 2 depicts the mechanisms of IF-WS2 nanoparticles and their behavior under various conditions: static and dynamic loads, shock, shear, etc. Due to the Nano range of the primary particle size (30-280nm), the surface area is significantly large thus the treat rate of *IF*-WS2 in applications varies from 0.15% to 1%. In comparison 2H-MoS2 is mostly used between 3% - 10% accompanied with active sulfur to get decent EP characteristics (above 315kgf ASTM D2596).

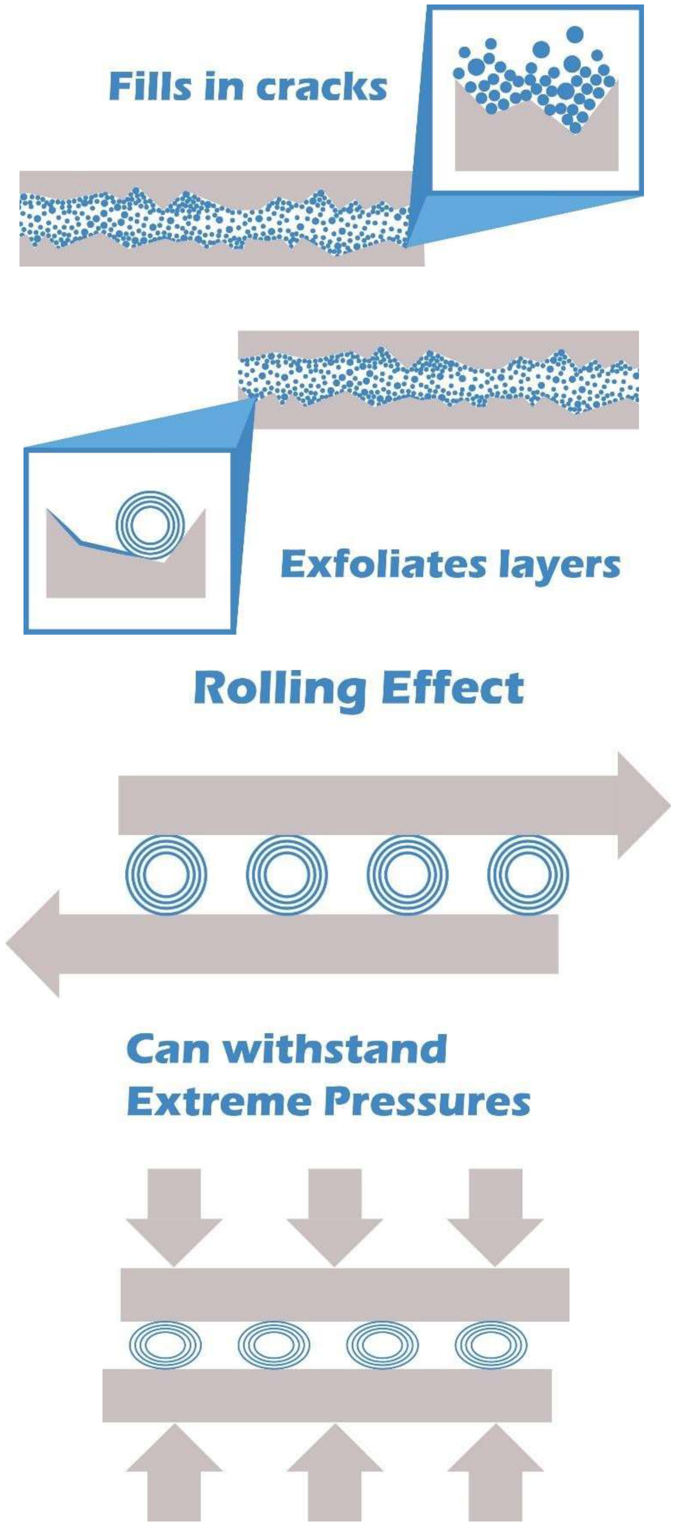

Figure 2. Mechanism of *IF-WS2* performance

Below is the summary of mechanism that describes the operation of IF-WS2.

- Spherical nanoparticle with a hollow core act as a damper at applications with high loads and impact
- Nano-sized particles smooth a surface, covering roughness irregularities and asperities
- The spherical geometry allows nanoparticles to play a role of nano ball bearings, creating a roller friction
- Multi-layer onion-like structures of IF WS2 nanoparticles start exfoliating under high pressures and create a thin protective film on the friction surface.

The study conducted by O. Tevet et al. shows mechanisms of *IF* –WS2 nanoparticle's operation [5]. For the experiment a golden nanoparticle was deposited on a surface of *IF* –WS2 nanoparticle, to play a role of the marker. The study showed that rotating mechanism predominant under loads up to 0.7 GPa, sliding under uniaxial compression between 0.6 and 1.1 GPa and exfoliation at compressions above 1.1 GPa. All the tests were conducted under low shear rate: low speed (quasi-static) 10^{-7} m/sec.

Remarkable tribological results of IF –WS2 particles results shown in some earlier tests were not always repeatable while testing the materials by various matrices. It was found that proper/stable dispersion was not the only requirement to achieve excellent tribological. Stable dispersion provides a homogeneous distribution of particles and shelf life. However, certain surfactants may restrict IF –WS2 particles to be delivered to metal surface thus reducing performance [4].

A new challenging task faced the researchers: developing a way of converting the powder into liquid-type material with enhanced stability and performance.

The efforts of researchers and engineers resulted in 2^{d} generation of dispersions of *IF*-WS2 nanoparticles combining the best knowledge of nature of nanoparticles with advanced manufacturing techniques, resulting in a new level of stability and performance of lubricating materials.

In addition, the available data and experience in developing lubricating formulations made it possible to build dispersions of Nanopowders in liquids targeting specific operating conditions. The difference in formulation and way of manufacturing of this ready to use additives is a result of careful study of operation conditions and environment specific to the particular application.

Basically, IF –WS2 particles, compare to other solids, have multiple mechanisms of operation. Layer exfoliation/release, that is mono or multilayer WS2 that provides antiwear protection, occurs due to shear force, shock/impact and normal load. During WS2 layer exfoliation/release, friction decreases and released layer/layers adhere to the surface, by chemo-mechanical interlock, and creates a protective layer on a surface.

It is very important to note that in order to utilize IF –WS2 particles' benefit, it is required to provide particle to surface delivery mechanisms. It is important to use proper surfactants and synergistic additives that can provide: stable dispersion, surface delivery and avoid surface competition.

Experimental

The grease selected for our current study was a lithium complex soap (LiX) obtained from a commercial batch of a grease manufacturer. *IF*-WS2 based concentrate in paste form (EMX) was used at various treat rates between 0.5% and 3%. 2H-MoS2 micron-sized platelet-shaped particles of MoS2 were used at concentration range 3-10%. PTFE submicron solid dispersion in PAO30 oil was used in treat rates: 3wt%, and 5 wt% respectively. EMX, 2H-MoS2 and PTFE were mixed in the LiX grease followed by homogenization in the FlakTec Speed Mixer shown in Figure 3. Wear scars were measured in an optical microscope, and 3D images were obtained via RTec profilometer (Rtec Instruments).

All samples after milling were evaluated for grease consistency following ASTM D217 standard test procedures and dropping point following ASTM D2265. Table 1 shows a description of each sample evaluated.

Table 1. Sample description

Sample #	**Description**
Sample 1	LiX soap
Sample 2	LiX soap+1% IF WS_2 dispersion
Sample 3	LiX soap+1.5% IF WS_2 dispersion
Sample 4	LiX soap+2% IF WS_2 dispersion
Sample 5	LiX soap+3% IF WS_2 dispersion
Sample 6	LiX soap+3% 2H MoS_2
Sample 7	LiX soap+5% 2H MoS_2
Sample 8	LiX soap+10% 2H MoS_2
Sample 9	LiX soap + 3% PTFE
Sample 10	LiX soap + 5% PTFE

Figure 3. High-Speed grease mixer

Since *IF*-WS_2 powder after synthesis process comes in agglomerated form, where agglomerates are several microns in size, the de-agglomeration and dispersion procedures have been conducted. Figure 4c shows TEM micrographs of *IF*-WS_2 agglomerates, aggregates, and primary particle. Where on the edge of the aggregate can be seen the spherical shape of *IF*-WS_2 primary particles? In order to make sure that all *IF*-WS_2 particles were in the primary size range (30-280nm), ready to use IF-WS_2 based concentrate (EMX) has been used for this study.

Table 2 shows the list of standard tests that been conducted for each sample and parameters monitored.

Table 2. List of tests conducted on samples

#	Test Name	Description
1	ASTM D2266	Four-Ball Wear
2	ASTM D2266	Coefficient of Friction
3	ASTM 2596	Four Ball EP
4	ASTM D2509	Timken EP test
5	ASTM D217	NLGI grade
6	ASTM D2265	Dropping Point

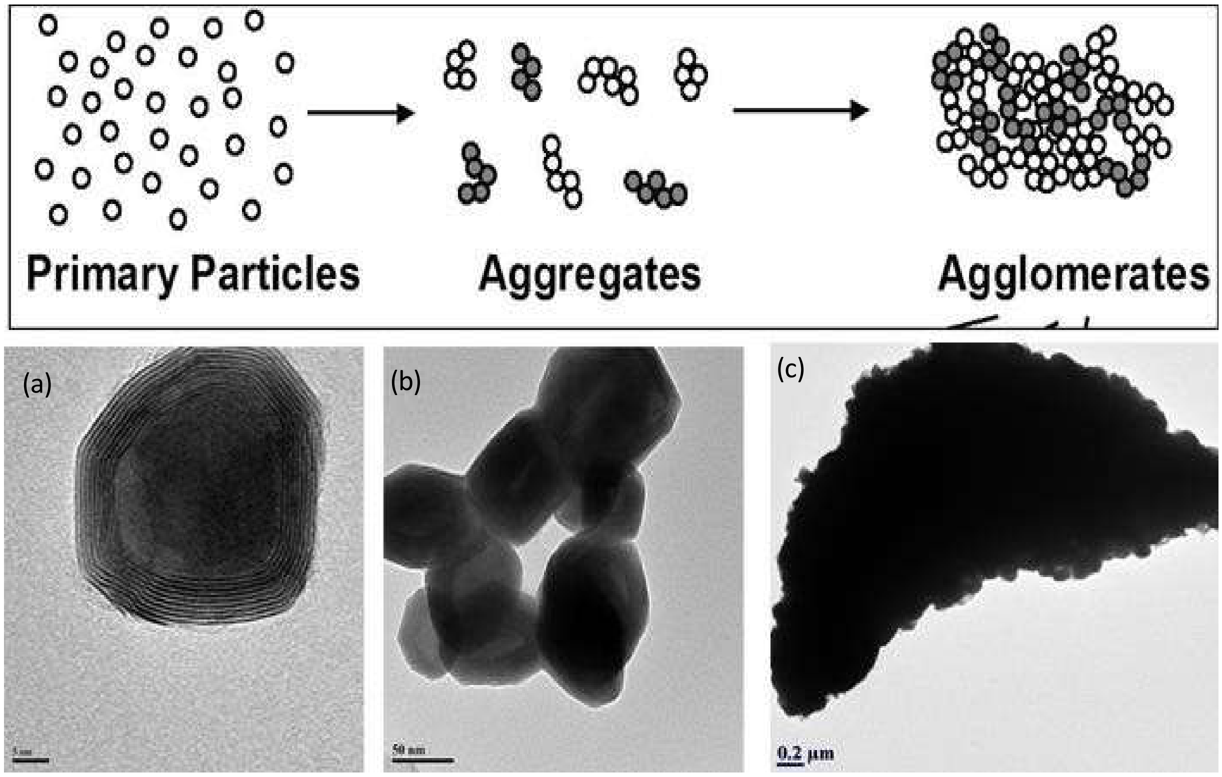

Figure 4. *IF*-WS_2 primary particle (a), aggregate (b) and agglomerate (c)

Results and Discussion

The grease formulations evaluated in this study showed excellent tribological properties. Results in table 3 below show that the LiX greases with *IF*-WS_2 particles even at low percentages increase the performance in extreme pressure and wear. 2H-MoS_2 particles showed good antifriction properties and increased EP properties, however, increased wear at higher treat rates. PTFE solids showed good antifriction properties however no EP performance and some antiwear properties.

Low friction, wear and increased EP properties of *IF*-WS_2 based dispersion could be explained by the multi-functional mechanism of *IF*-WS_2 particles. Due to spherical morphology of IF-WS_2 they behave differently under various tribological conditions. The mechanism can change from rolling, to sliding and to exfoliation under various loads, shear and shock. On the other hand 2H-MoS_2 platelets reduce friction predominantly under shear/sliding motion. And due to platelet structure and big particle size (about 1.5-3 μm) could be abrasive, especially high-speed applications.

Figures 5-8 shows wear, friction and weld point data for all tested samples.

Table 3 Property summary of LiX grease samples

Test ame	Description	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10
ASTM D217	Cone Penetration of Lubricating Grease	263	267	273	273	276	271	275	277	270	272
ASTM D2266	Four Ball wear, mm	0.57	0.515	0.465	0.425	0.4	0.47	0.54	0.655	0.46	0.51
ASTM D2266	oefficient of Friction	0.0704	0.0718	0.0723	0.0569	0.0525	0.063	0.0635	0.0671	0.045	0.048
ASTM 2596	Four Ball EP, kg weld point	160	315	500	620	800	315	400	620	160	160
ASTM 2596	Four Ball EP, LWI	N/A	N/A	N/A	122.77	N/A	N/A	92.94	N/A	N/A	N/A
ASTM D2509	Timken EP test, lbs	N/A	N/A	55	65	N/A	N/A	N/A	65	N/A	N/A
ASTM D2265	Dropping Point, C	245	242	253	255	255	253	251	248	N/A	N/A

Figures 5, 6 and 7 show wear, CoF and weld point data for all tested samples.

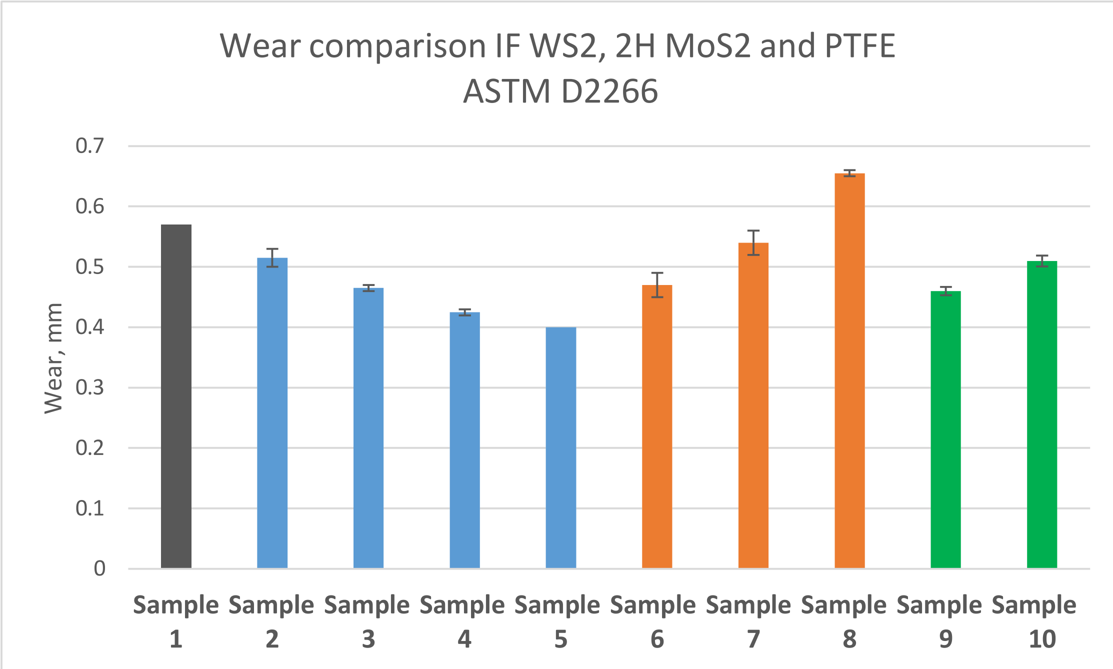

Figure 5. Wear scar comparison of tested samples

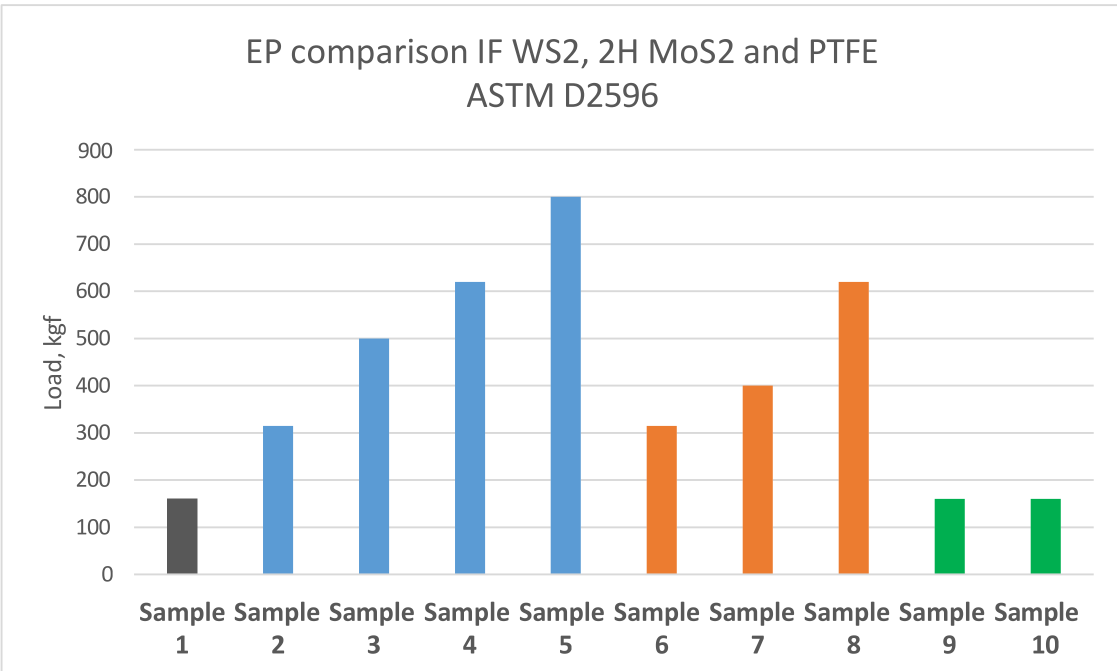

Figure 6. EP properties comparison of tested samples

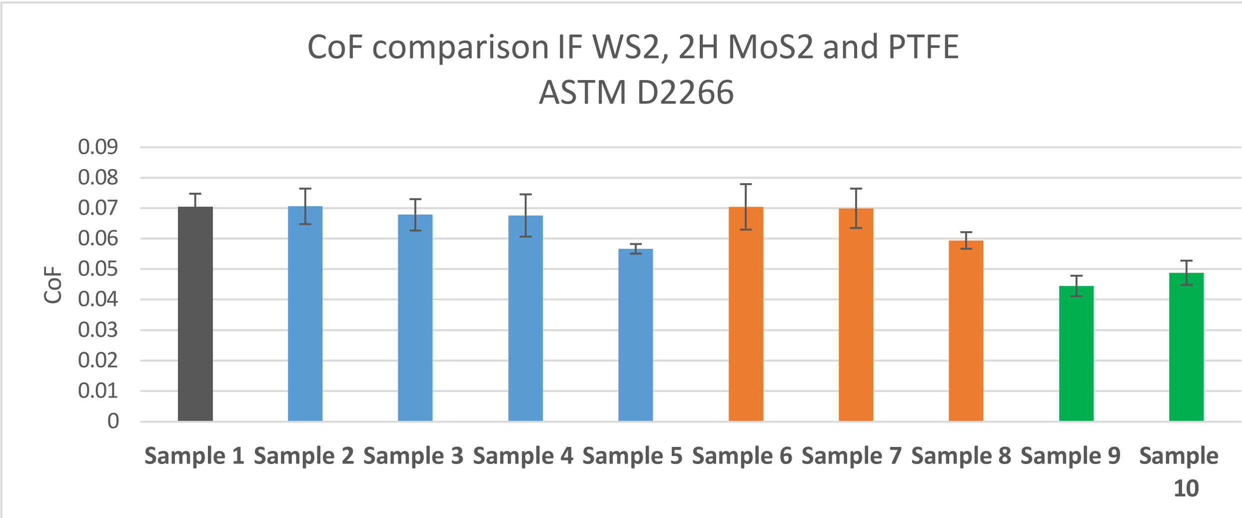

Figure 7. antifriction properties comparison of tested samples

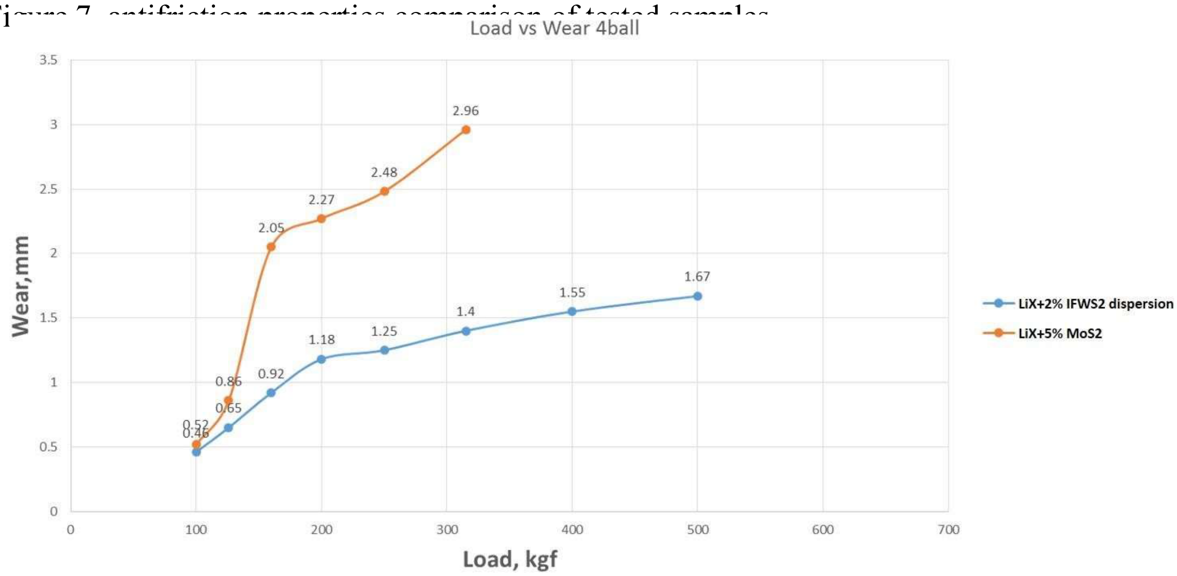

Figure 8. Wear as a function of load

Conclusion

The work done here has compared IF-WS_2 spherical particles' based concentrate, micron-sized platelet form 2H-MoS_2 particles and submicron PTFE particles in LiX (lithium complex) grease formulation. By using some basic tribological tests (4-ball EP (ASTM D2596), 4-Ball Wear (ASTM D2266), and Timken (ASTM D2509)), it has been shown that *IF*-WS_2 dispersion significantly improves tribological properties of greases compare to platelet *2H*-MoS_2 and PTFE particles. Other physical properties of the grease such as dropping point or cone penetration were not affected.

It was noticed that surfactant chemistry is important to improve properties and provide particle to surface delivery mechanisms. Also, experiments showed the synergistic effect of IF –WS_2 particles with MoS_2, sulfur and organo phosphate (ZnDDP, MoDDP)

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Low-temperature tribological testing of greases

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Abstract:

The current study focusses on a newly developed setup which enables tribological testing of greases over a broad range of temperatures, including sub-zero temperatures. The greases were tested with the roller bearing attachment for the MCR Tribometer, equipped with a convection temperature device (CTD) for temperature control. Speed ramps starting from $5x10^{-5}$ rotations per minute (rpm) to 500 rpm, break-away torque measurements, and rollout tests were carried out to determine the static/limiting frictional resistance of the system. In addition to the tribological tests, rheological investigations were also carried out on the greases to find a correlation between rheological and tribological properties, especially in the static regime and during the transition of the system into the kinetic regime.

Keywords: Low-Temperature Grease Testing, Boundary Lubrication, Static Friction, MCR Tribometer

1 Introduction

Lubricating greases find their use in various applications such as gears, bearings, chains, guides, etc. Selection of grease for a particular application depends upon various factors ranging from the contact pressure, lifetime, temperature, etc. With ever increasing demand for improvement in the efficiency of systems, selection criterions have also become quite stern. Under such conditions, having knowledge about the characteristics of the grease beforehand is highly beneficial in the selection process. This however is only possible for its inherent properties such as thixotropy, density, oxidation stability, rust protection, etc., and some specific tribological properties like extreme pressure properties, loadability, etc. There are of course various other parameters such as its resistance towards corrosion, long term stability, friction behaviour, etc., which need to be characterized individually for each given tribological system.

The principal aim of this paper is to present a test methodology to characterize frictional properties of grease over a range of temperatures using real life bearings. The uniqueness of this methodology lies in its low-torque and low-speed capabilities. These are extremely important while characterizing the breakaway torque characteristics of a system. Breakaway force required to overcome static frictional resistance of the tribological system and set it into macroscopic motion. Typical applications for the sub-zero temperature conditions are space, aerospace industry, etc. While in most cases a low breakaway force is wished for, it must also be noted that a certain amount of resistance is still required to inhibit involuntary movements. This parameter is also important in tribological systems with dynamic motion profiles involving start-stop events, which are often detrimental to the system.

2 Test Methodology

The tests were carried out on an MCR Tribometer from Anton-Paar, equipped with a convection temperature device (CTD600) with a bearing test setup. A rendering of the bearing test setup is shown below in **Fehler! Verweisquelle konnte nicht gefunden werden.**. The CTD chamber is capable of reaching temperatures from -160 to 600°C. This setup uses real life bearings for the tests.

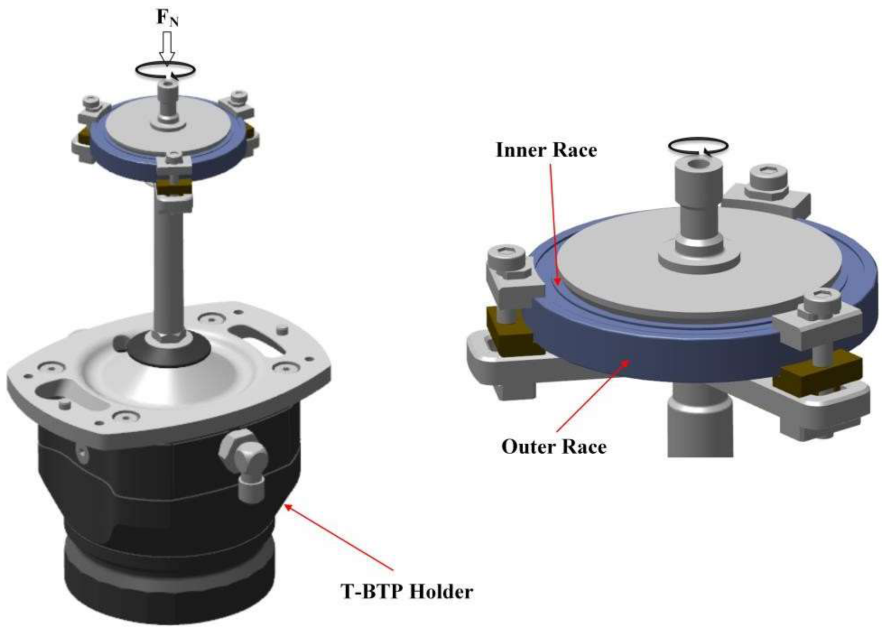

Fig. 1. Test Setup – Bearing setup for the CTD

2.1 Test Profile

To study the influence of temperature and the rotational speed on the greases, rotational speed ramp tests were carried out 20°C, -10°C, and -40°C. The normal force was maintained at 15 N during the entire test duration. During the test, the said load was applied to the contact and the system was allowed to stabilize for 3 minute (*Interval I*), see **Fehler! Verweisquelle konnte nicht gefunden werden.**. This was followed by *Interval II,* wherein the rotational speed was logarithmically increased from $5x10^{-5}$ to 500 rotations per minute (rpm) in a span of 6 minutes. This sequence is repeated three times for each set of specimen.

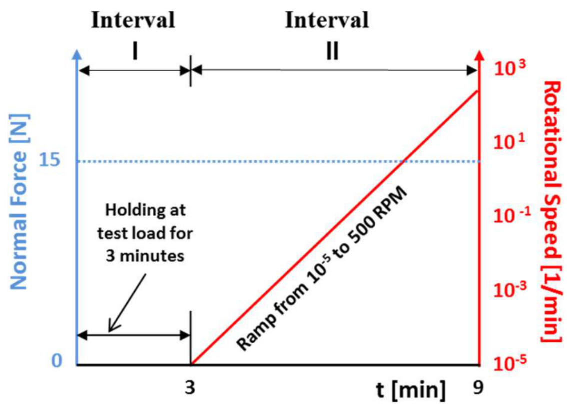

Fig. 2. Test profile showing logarithmic ramp of the rotational speed w.r.t. time.

Additionally, rollout tests were carried out to ascertain the frictional resistance of the system leading to a standstill when the external rotational torque is turned off. Here, the rotational speed was slowly increased from 0.1 to 500 rpm in 60 seconds and the speed was maintained for 5 minutes at 500 rpm. Thereafter, the rotational torque was turned out and the time taken to reach 0.1 rpm was observed.

2.2 Specimen

Commercially available ball bearings were used in these tests with an inner and outer diameter of 11 and 25 mm respectively.

3 Results

The results are presented in two parts. The first part deals with the rheological data, followed by the tribological data.

3.1 Rheological Data

Shear dependant viscosity measurements were carried out with the two greases at 5 and 35°C. The curves below in Fig. 3 show that Grease 1 has a lower viscosity overall compared to Grease 2. This is especially stronger at higher shear rates. The up and down curves for Grease 2 are not overlapping as they are with Grease 1. This indicates that Grease 2 suffers from irreversible structure breakdown during the upward run.

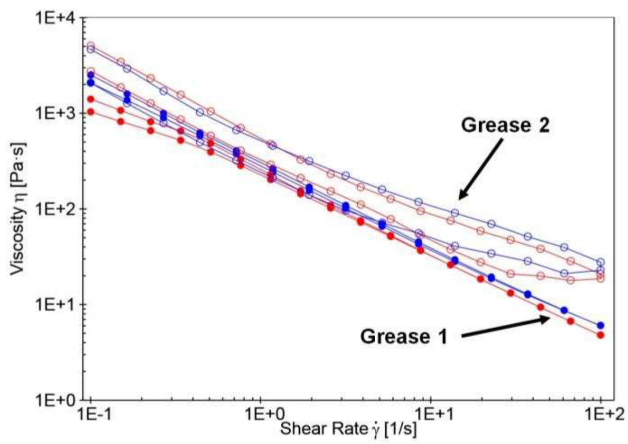

Fig. 3. Viscosity as a function of shear rate for Grease 1 (solid symbols) and Grease 2 (hollow symbols). Curves in blue are obtained at 35°C and those in Red at 5°C [1].

3.2 Tribological Data

The tribological data is depicted in the form of graphs wherein the frictional torque is shown as a function of rotational speed. In Fig. 4, the graph on the left contains data from Grease 1 and that on the right from Grease 2. The general trend observed is the significant increase in the frictional torque

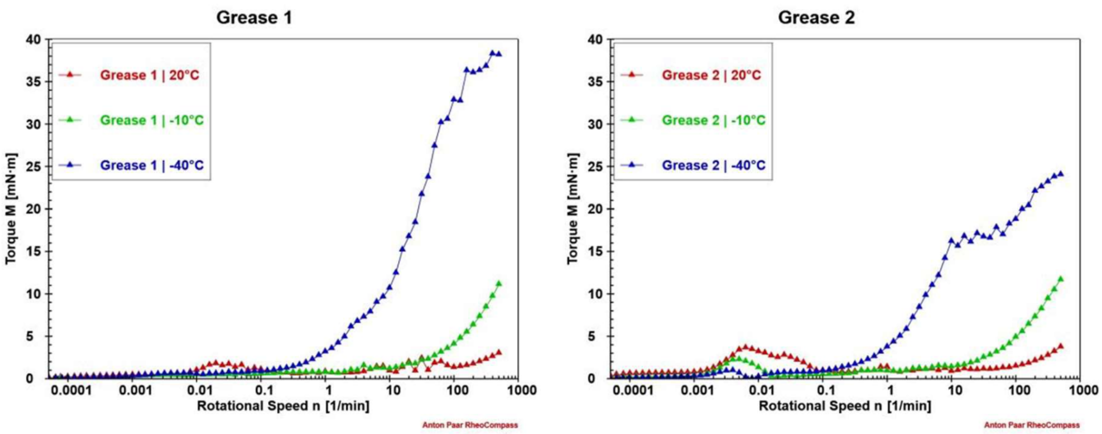

Fig. 4. Friction torque as a function of the rotational speed for Grease 1 (left) and Grease 2 (right).

at higher rotational speeds and the shift of transition into fluid friction regime towards lower rotational speeds with decreasing test temperature. This is due to the increase in the viscosity with decreasing temperature which in turn increases the fluid drag at higher rotational speeds. At lower rotational speeds, as shown in Fig. 5, the initial peak represents the transition from static to kinetic state of motion and is known as the breakaway point and the corresponding torque the breakaway torque. The higher the value of the breakaway torque, the greater is the force that needs to be applied to bring in relative motion at the contact interface. In the case of Grease 1, there is no significant difference between tests carried out at -10°C and -40°C. However, with Grease 2, the breakaway torque is seen to increase with increasing test temperature. In general, Grease 2 has a higher breakaway torque, probably due to higher viscosity.

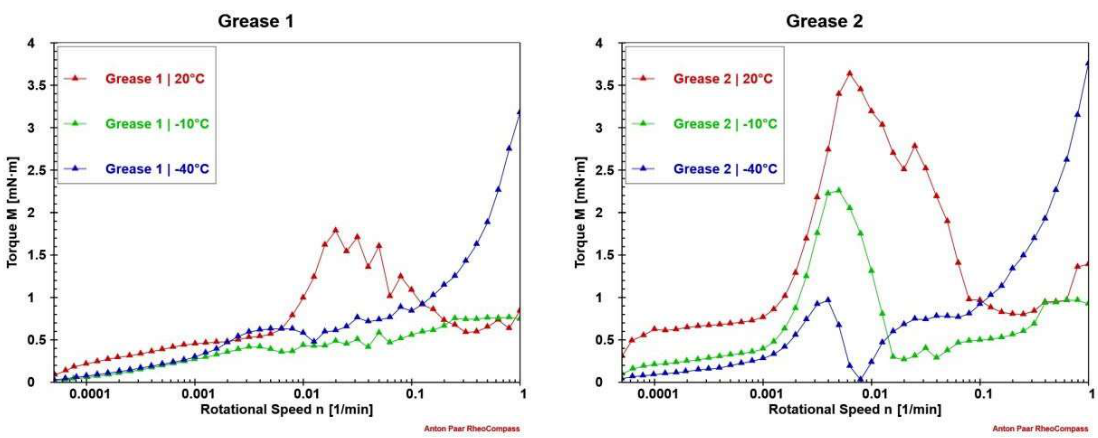

Fig. 5. Friction torque as a function of the rotational speed for Grease 1 (left) and Grease 2 (right) highlighting the low-speed regime of the data presented in Fig. 4.

3.3 Rollout tests

The results of the rollout tests are presented below in Fig. 6. Here, graphs with the solid symbols represent Grease 1 and those with the hollow symbols, Grease 2. With decreasing temperature, both greases show a decrease in the rollout time, and this can be attributed to the increase in viscosity. The test methodology needs to be further modified to obtain stronger temperature and sample based differences.

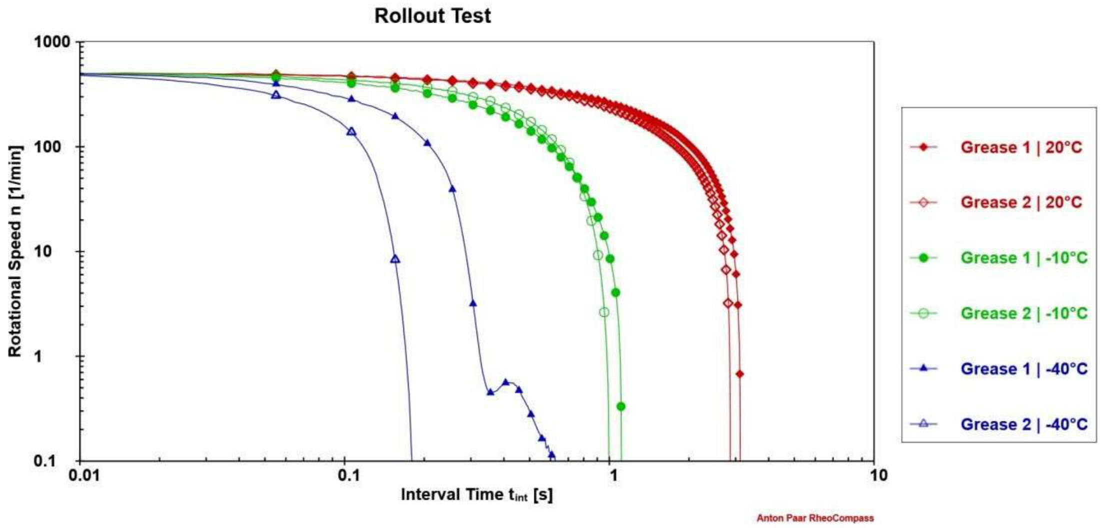

Fig. 6. Graph depicting the rotational speed as a function of the interval time for the rollout tests.

4 Summary

The work presented here showcases rheological and tribological tests carried out on two different greases at different temperatures, including sub-zero conditions. Differences were observed between the performance of the greases at different temperatures, and this difference was much pronounced at high speed regimes. However, even in the low speed regime, grease with higher viscosity showed a marked dependence of the breakaway torque on test temperature. This again correlates to the change in the viscosity of the grease. However, in the case of the grease with lower viscosity, the changes in the low speed regime were not that prominent, except at -40°C. The results presented here are the first part of a series of investigations that are being carried out to study the rheological and tribological behaviour of greases at sub-zero temperatures, extending to -80°C.

5 References

1. Läuger, J., Rummel, F., Pondicherry, K.S. Interdependency between Rheology and Tribology of Lubricants. World Tribology Conference, Beijing, Sep 2017

6 Acknowledgement

The authors would like to acknowledge that the samples were supplied by Mosil Lubricants Pvt. Ltd.