elgi

EUROGREASE

2021

April-May-June

2

ELGI "Business as Unusual"

Spring Virtual Events / Autumn Events

Call for 2022 Papers Hamburg

Position Paper LiOH

New Members

Company Profile:

- HL Blachford
- Cocan Graphite

Technical Papers:

- The economic situation of the air transport industry in times of the Corona pandemic.
- Composition of calcium sulfonate grease: Improvement of performance through different additive systems
- Shear Stability of Polymer-Modified Greases
- Maintaining safe and quiet railways with " Top of Rail Materials

grasa
greix
мастило
koipea
schmierfett
smeervet
plastické maziva
smørefett
grasso
γρασσο
grusu
mazive masti
voitelurasva
شحم
смазка
mazacie tuky
smørefedt
gres
graisse
únsoare
smurfeiti
smar plasticzny
kenozsir
משחת סיכה
smörjfett
lubricating grease

EUROGREASE MAGAZINE 2

2021 April-May-June

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Terry Dicken
ELGI Chairman

"Business as unusual"

Under normal circumstances I would be writing this podium letter following our Annual General Meeting, stating how good the paper presentations were, how we had a large number of delegates and exhibitors, how the annual dinner had been a great success with good food, fine wine and an excellent band which allowed dancing till the early hours and I would have offered a big thank you to all the sponsors.

But, as we all know, circumstances are far from normal and it will have been two years since I have been able to write any of the above, but maybe we are seeing a light at the end of the tunnel, with a vaccine programme being rolled out and, with a better understanding of this terrible virus, we could be seeing the first stages of returning to normality. I appreciate that some parts of the world are still struggling with the pandemic but I hope they will soon overcome their struggles.

So, I am not able to write about an AGM, but I can write about our Virtual Spring Events which have just taken place.

The events ran from April 19th to April 27th and, since this was the very first time we have hosted such an event, there was a learning curve to be overcome, especially regarding the technical aspects of the programme. Following a lot of hard work, (especially from our office manager, Carol Koopman, and board members Manfred Jungk and Rolf Quermann) the event was very successful and I wish to thank the sponsors and exhibitors for supporting us during this event.

The programme started on the 19th with a two-day joint ELGI/STLE tribology exchange workshop with 44 delegates and lecturers. This workshop was vibrant and active with a large amount of technology discussed and exchanged.

The exchange workshop was followed by 3 days of working group meetings, Food Grade Lubricants, Test Methods, Railway Lubricants, Grease Particulate Evaluation and Bio-Based Greases. These groups were well attended and delegates were able to continue their programmes successfully.

The final two days were dedicated to a "Virtual Grease Symposium ". 125 delegates attended with 13 papers being presented including the keynote presentation by Norbert Lübben from the Federal Association of the German Aviation Industry (BDL) who discussed the future of air transport and also highlighted the devastation the air travel industry has experienced over the last year. In addition, the symposium received reports from the working group chair persons and also a presentation from Chuck Coe regarding the Grease Production Survey.

It was also a pleasure to receive updates from NLGI (President Mr Jim Hunt), NLGI India Chapter (President Mr SSV Ramakumar) and CLGI (Representative Mr Ray Zhang). I think we all agreed that we hope that the next AGM's will be face-to-face meetings and all our organisations have tentative plans in place for this to happen. Talking of which, look out for our Autumn Events 2021 to be held 25th-29th October in Amsterdam. Further details can be found in this edition of Eurogrease and on our website www.elgi.org

I also want to remind you that, hopefully, it will be third time lucky for our 2022 AGM, scheduled to be held in Hamburg Germany between April 30th and May 3rd 2022 at the Grand Elysee Hotel. I can then revert to using this podium slot for reviewing an actual face-to-face AGM!

Take care and stay safe
Terry Dicken
ELGI Chairman

Autumn Events 2021

Save the dates 25th - 29th October 2021
Amsterdam

Working Group Meetings
25th October 2021

Grease Training Course
26th & 27th October 2021

ELGI-STLE Tribology Exchange Workshop
28th & 29th October 2021

stle Society of Tribologists and Lubrication Engineers

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Call for Papers
32nd ELGI AGM
30th April – 3rd May 2022

"The Music of Greases, it's in the Composition"

Would you be interested in presenting a paper on the above mentioned theme at our 2022 AGM Hamburg, Germany?

Our 2022 themed topics of interest can include, but are not limited to:

- Performance of Greases
- Composition of Greases & Food Grade Greases
- Instruments used in the measurement & evaluation of Greases
- Conduction surveys, field tests; application

If yes, do send your paper proposal by completing author information form (download from the ELGI website). Papers that tie in with the 2022 theme will be given high priority. Other topics of interest to the grease industry are welcome and will be considered too. We have space for 14 papers only, so do submit paper abstract by the 20th October 2021.

For information, please contact
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Joint position paper on Lithium salts harmonised classification and labelling proposal

Following the proposal by ANSES (on behalf of the French MSCA) in June 2020 for harmonised classification (CLH) for reproductive toxicity category 1A – H360FD, for lithium carbonate, lithium chloride and lithium hydroxide, ATIEL, the Technical association of the European lubricants industry, together with the UEIL and the ELGI, is raising some concerns about the appropriateness of the proposed dossier.

The three associations are supporting the technical arguments that have been submitted during the consultation, notably by FUCHS the lead registrant of lithium 12-hydroxystearate and by Lithium REACH consortium. We indeed question the selection of the studies and the read-across performed.

This classification would have a severe impact on the lithium-based greases which are of major importance in multiple applications in Europe, such as the operation of all wind turbines, ensuring the performance of most of the industrial machines that use electrical motors, aircraft lubrication (including landing gear), and numerous automotive parts such as wheel bearings, which are just a few applications that are key to society. Replacement of Li-based greases would result in performance loss, lower levels of machine availability, higher greenhouse gas emissions and a substantial volume increase of resource-demanding grease type products helping to meet the goals of the new green deal.

Lithium hydroxide is used as raw material, acting as a starting material together with a fatty acid to manufacture the thickener in the production of greases.

The fatty acids used for the reaction are normally derived from natural sources with typical compositions but a certain range of variability from batch to batch. This may result in a slight excess of lithium hydroxide after the reaction in certain batches to meet the desired grease properties.

If the final concentration of Lithium Hydroxide exceeds the classification threshold for toxicity for reproduction Cat. 1A or 1B, the final grease will be classified accordingly. The application of the conservative calculation method (assuming presence of Lithium Hydroxide above 0.3% in general) would lead to an over-classification in most of the cases.
Lithium based greases are the most widely used greases in Europe, at approximately 70 % of the grease used (2019 NLGI Annual production Survey).

Lithium based greases have been used safely over the last decades, and we question such a proposal that could be detrimental for investments in the sector.

We would appreciate a careful review and assessment of the current French proposal by ECHA and other member states' competent authorities with these considerations being brought forward.

About the signatories:

- **The Technical Association of the European Lubricants Industry (ATIEL)** is a non-for-profit association (ASBL) representing the combined knowledge and experience of leading European and international engine oil manufacturers and marketers. By drawing on the technical know-how of its membership, ATIEL promotes consensus on key technical, product stewardship and sustainability issues, ensuring that engine oils continue to contribute to improved wear protection, deposit control, lower emissions, and fuel economy C02 emissions efficiency.

- **The Union of the European Lubricants Industry (UEIL)** is the umbrella association representing more than 35 associations and 450 companies covering the entire lubricants' value chain, from manufacturing and distribution to recycling. Our mission is to support informed policymaking by educating EU stakeholders about the value and benefit of the lubricants industry for Europe's economy, society, and the environment.
- **The European Lubricating Grease Institute (ELGI)** established in 1989 is an independent, self-governing institute, financed solely by members. It is a group of scientific and technical institutes active in the field of tribology dealing with lubricating greases and associated products.

New ELGI Member

The ELGI is pleased to welcome our new member

LPC SA
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Grease Production Unit
124 Megaridos Avenue
19300 Aspropyrgos, Attica Greece
Tel: +30 21 08093898 Mob: +30 6983292223 achatzaki@lpc.gr www.lpc.gr

LPC is a member of the Motor Oil Group, founded in 1981 and is active in the industrial production of base oils, the production and marketing of packaged lubricants and greases as well as the marketing of paraffins and other petroleum products.
The company is charting a constantly upward and dynamic course in Greece, Europe and many other countries in the world.
Specifically, LPC is the largest exporter of packaged lubricants in Greece with exports to more than 55 countries, while operating subsidiaries in Bulgaria, Romania and Serbia.

The Brand portfolio of LPC consists of internationally strongly developing brands such as Cyclon, Avin, Nipco, and is ideally complemented in Greece by the representation of one of the most historic and highest quality brands in the category of lubricants internationally, Valvoline.
LPC has developed and marketed over 200 different types of lubricants for automotive, industrial, shipping, aviation and all types of industrial use. The basic and final lubricants produced are approved by international organisations and cover all lubrication needs. All chemical substances in LPC products are registered and approved according to REACH Regulation (EC 1907/2006).

The five international management standards applied in LPC (ISO 9001: 2015, EMAS: EL00051, ISO 14001: 2015, ISO 50001: 2018, ISO 45001: 2018) ensure the consistent delivery of improved products and services and enhance the company's commitment to people and the environment.

At the LPC production facilities in Aspropyrgos, Attica, there are three distinct production units:

- The LPC refinery, which has been operating since 1981
- Lubricants Blending and Packaging Unit with fully automated packaging lines, which produce the full range of lubricants.
- In 2020, LPC in collaboration with ABB Cellier completed the set-up of a state-of the-art, flexible, highly automated and modern grease production unit capable of producing various types of grease. The new production unit launch was coupled with the newly launched brands, Cyclon Innova & Cyclon Premus, products that are specially engineered to provide protection and cover the needs of a multitude of applications from the simplest to the most demanding ones.

Starting with our in-depth knowledge of market trends and requirements and subsequent technical developments, LPC is constantly investing in technological innovation and research in order to develop advanced technology products with a reduced environmental footprint, while actively supporting the circular economy in all its activities.


ENGINEERED TO
PROTECT
INNOVA
COMPLEX/FG-GL
CYCLON
CYCLON developed a full range of technologically advanced greases, specially engineered to protect your equipment in any demanding condition.
INNOVA: Premium, top-tier products for specialized applications.
PREMUS: High performance products for demanding applications.
www.cyclon-lpc.com
CyclonLPC

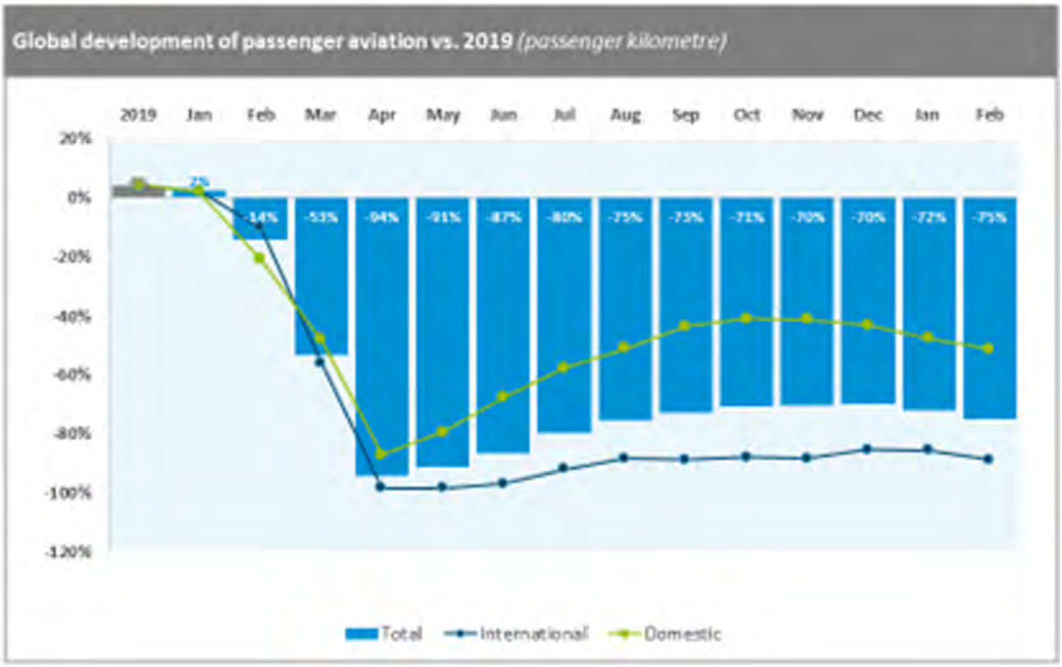
Key Speaker: Norbert Lübben (BDL)
Bundesverband der Deutschen
(Federal Association of the German Aviation Industry)

After his studies in Geography at the universities of Cologne and Rome Norbert Lübben worked for more than 20 years at Lufthansa German Airlines and Air Berlin in the management of network development, airline alliances and distribution. During his work at Lufthansa, he completed studies at various international business schools, finalising them with a master's degree in business from the University of McGill in Montreal. He is currently an independent consultant in the aviation business. Since 2019 he is the head of the department of economics at the German Aviation Association (BDL).

Keywords: Air traffic, Covid19 pandemic, green deal

The presentation reflects on the development of the German aviation industry in the year 2020 and the first month of 2021. The impact on passenger traffic, cargo traffic and the development of airports in the extraordinary crisis will be analysed. Coming from the current situation there will be also an outlook on how the path to recovery could look like and which future challenges – namely in the area of sustainability – the industry has to tackle.

Airlines: Global traffic more or less broke down and is not recovering



Source: IATA

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Also in Germany: Traffic at the airports does not recover at all

Source: Flughafenverband ADV (Januar bis Dezember), Internationaler Flughafenverband ACI (Januar bis November)

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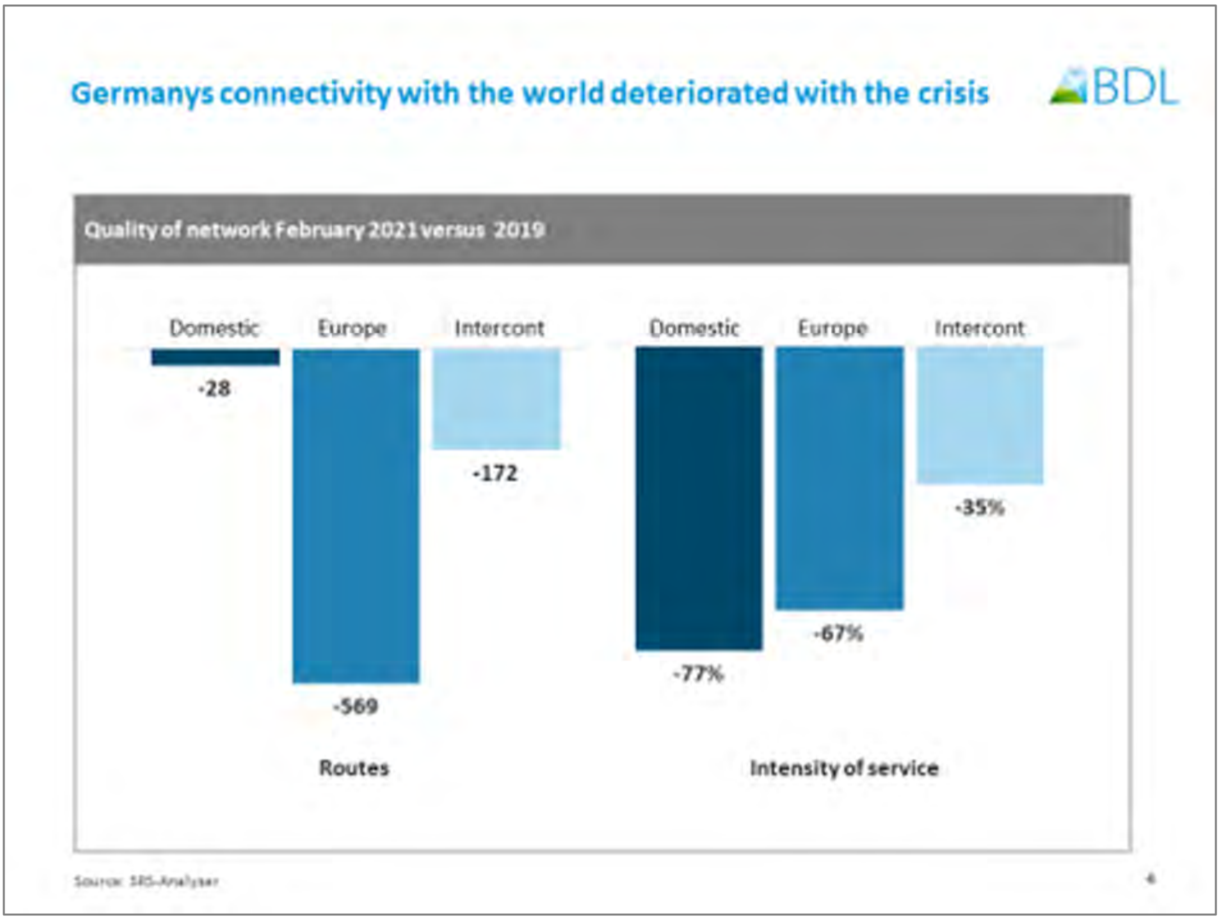
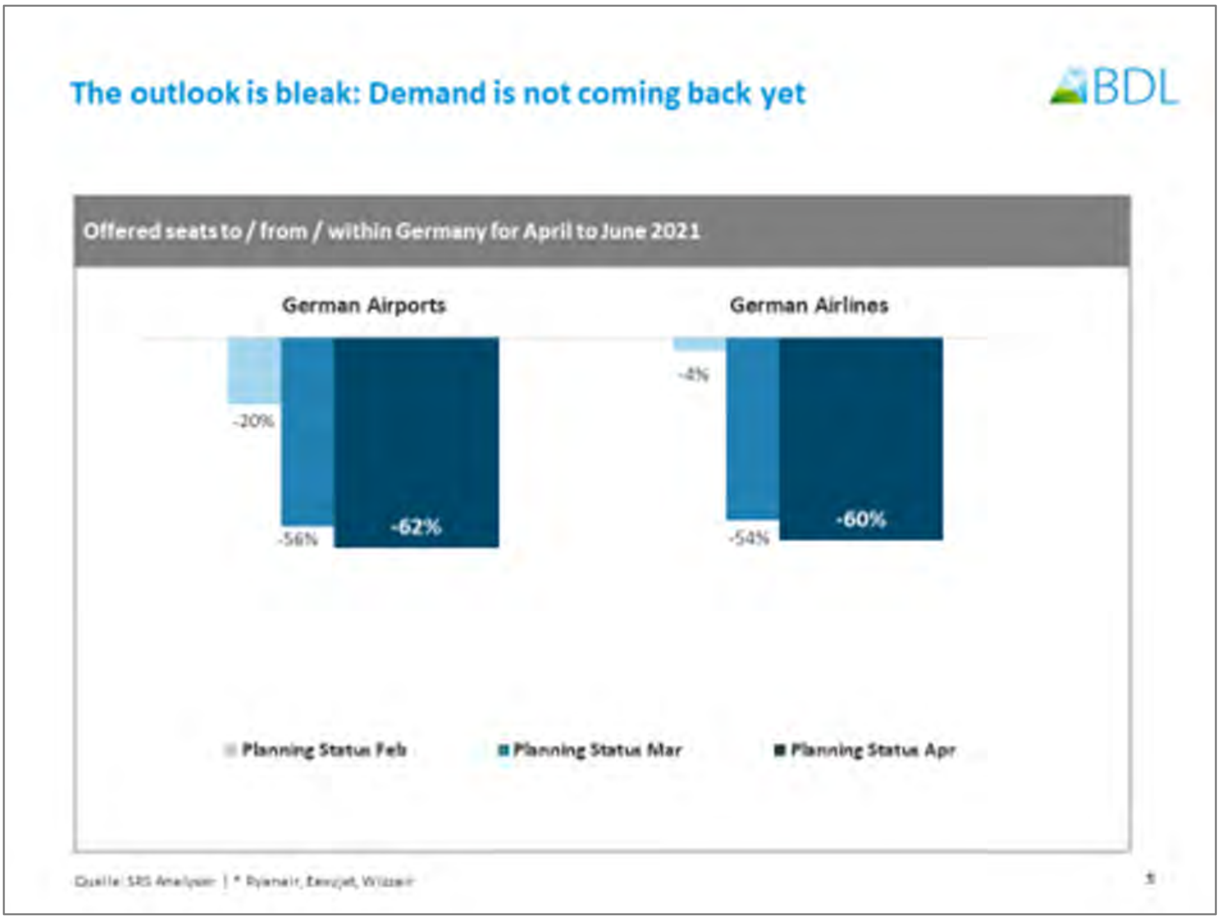
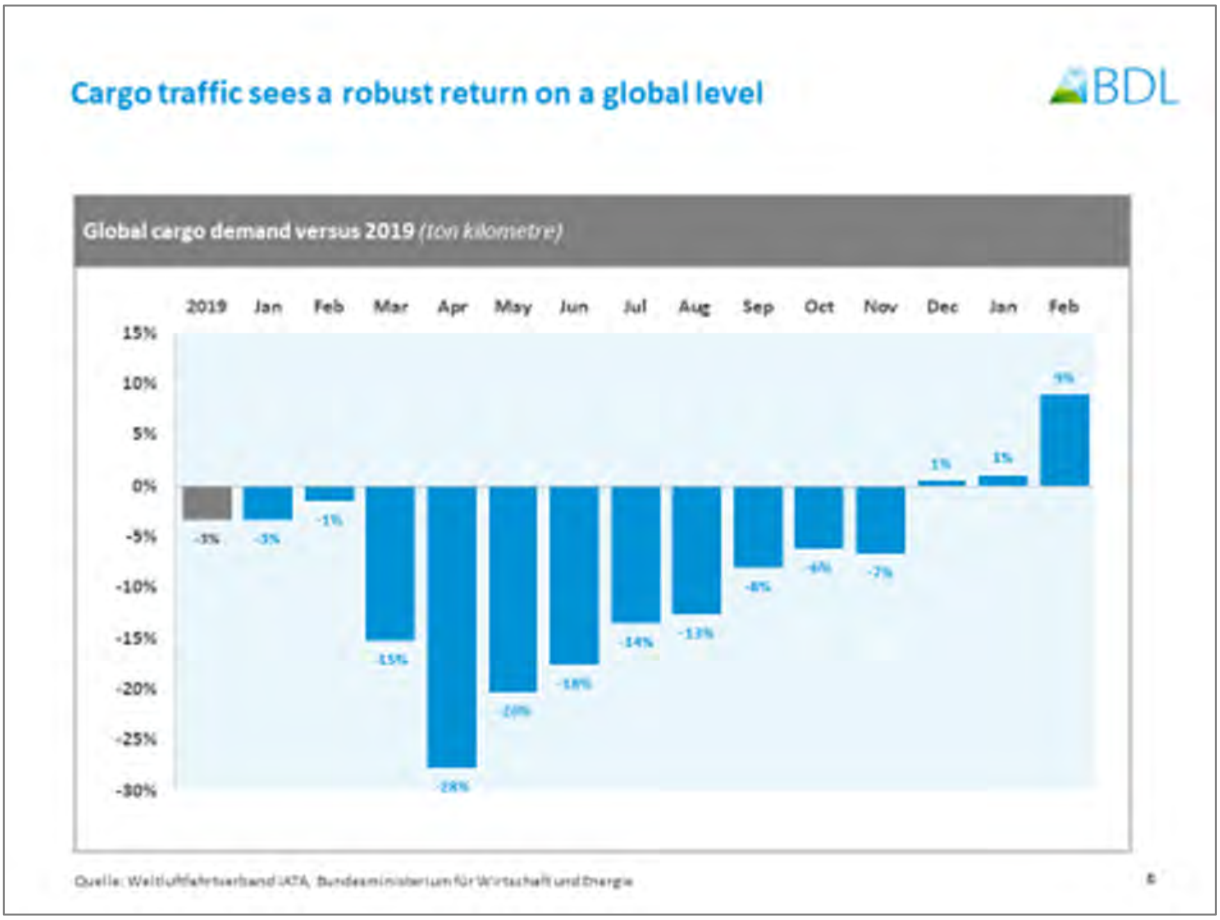

Germanys connectivity with the world deteriorated with the crisis
BDL
Quality of network February 2021 versus 2019
Domestic
Europe
Intercont
Domestic
Europe
Intercont
-28
-569
-172
-77%
-67%
-35%
Routes
Intensity of service
Source: SRS-Analyser
4


The outlook is bleak: Demand is not coming back yet
BDL
Offered seats to / from / within Germany for April to June 2021
German Airports
German Airlines
-20%
-56%
-62%
-4%
-54%
-60%
Planning Status Feb
Planning Status Mar
Planning Status Apr
5


Cargo traffic sees a robust return on a global level
BDL
Global cargo demand versus 2019 (ton kilometre)
2019 Jan Feb Mar Apr May Jun Jul Aug Sep Oct Nov Dec Jan Feb
15% 10% 5% 0% -5% -10% -15% -20% -25% -30%
-3% -3% -1% -15% -28% -20% -18% -14% -13% -8% -6% -7% 1% 1% 9%
Quelle: Weltluftfahrtverband IATA, Bundesministerium für Wirtschaft und Energie
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Cargo recovered as well substantially at German airports
BDL
Cargo (loaded / unloaded) at German airports versus 2019
2019 Jan Feb Mrz Apr Mai Jun Jul Aug Sep Okt Nov Dez Jan Feb
20% 15% 10% 5% 0% -5% -10% -15% -20%
-3% -7% -5% -12% -15% -11% -8% -7% -3% 0% 1% 4% 13% 6% 9%
7

The economic impact is massive and an unprecedented shock

BDL

The year 2020

- Revenue all airlines -68 per cent
- Ebit all airlines -119 bn. USD*
- Ebit European airlines -27 bn. USD*
- Revenues German aviation -71 per cent

Effects on staff

- German aviation -6% in 4. quarter
- Roughly 70 per cent on short time
- 60.000 jobs in Germany at risk

The industry is in survival mode

- Fast and continuous capacity adjustment (flights, aircraft, terminals) to reduce variable cost
- Staff to be laid off or send to short work on long-term basis
- Immediate fix cost control and control of liquidity (projects, investments etc.) and debt
- Renegotiations with deliverers, shareholders, banks, unions, and governments
- No trends of consolidation yet

Catalyst damages

- Tourism, business travel / acquisition, event management, industry fairs, gastronomy, local transport, culture etc. suffer from tremendously from reduced travel options.

Quelle: IATA * Forecast base November 2020

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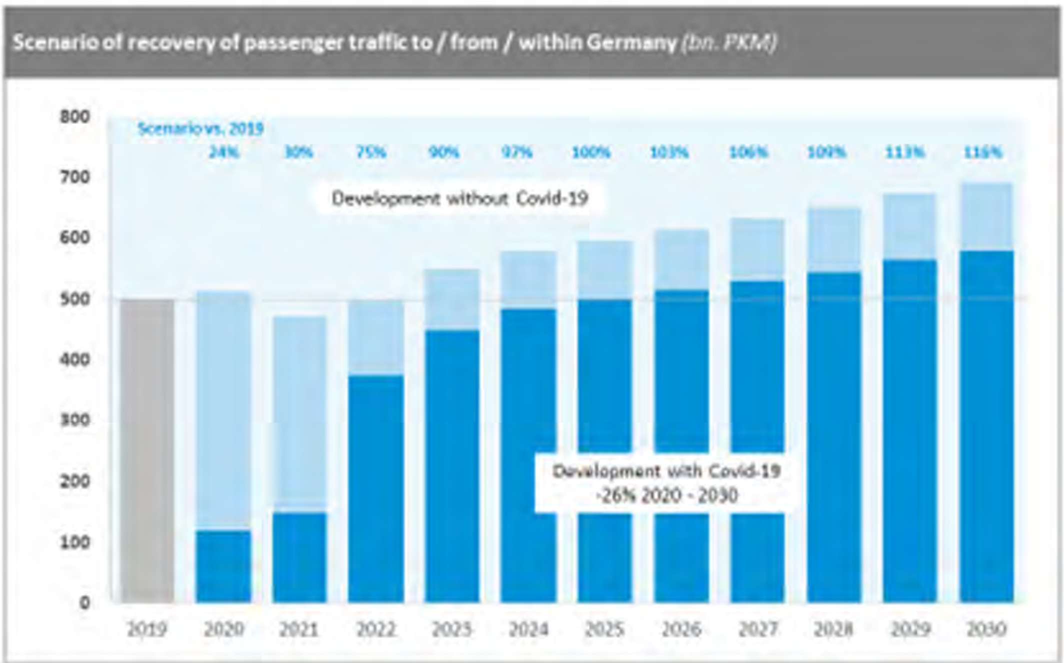
Recovery? It will take some time.....

BDL



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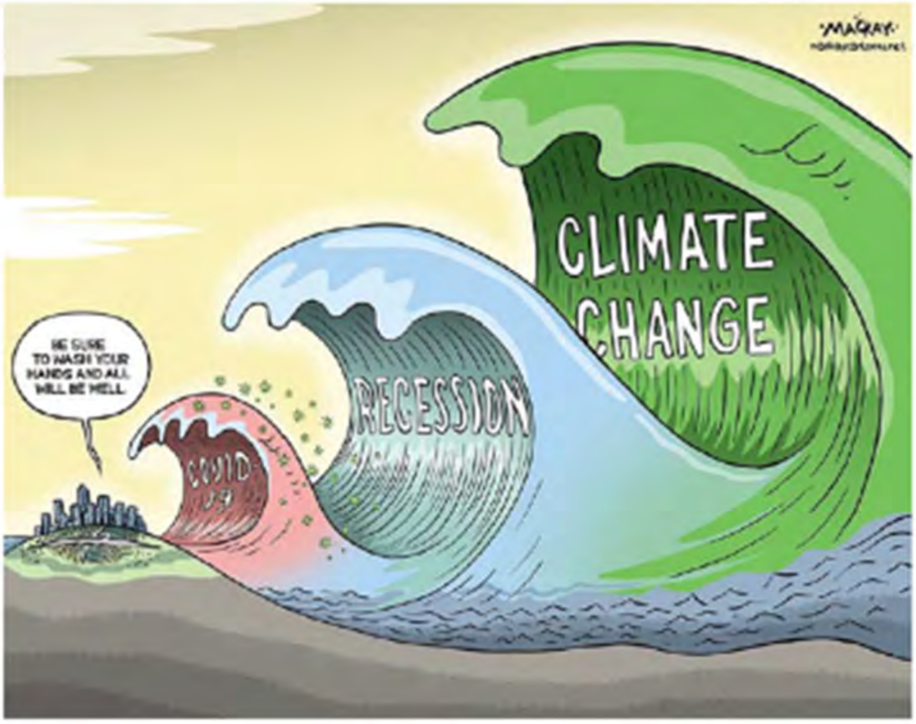
More challenges ahead!



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When Corona is done... the next challenge: The Green Deal

The target is a climate neutral Europe in 2050

Green Deal as a growth strategy

- Transform the EU in a
  - modern
  - resource-efficient
  - competitive

  economy
- Turning climate and environmental challenges into opportunities and
- Making the transition just and inclusive for all.

Planned investments: ca. 1.000 bn. € in 10 years

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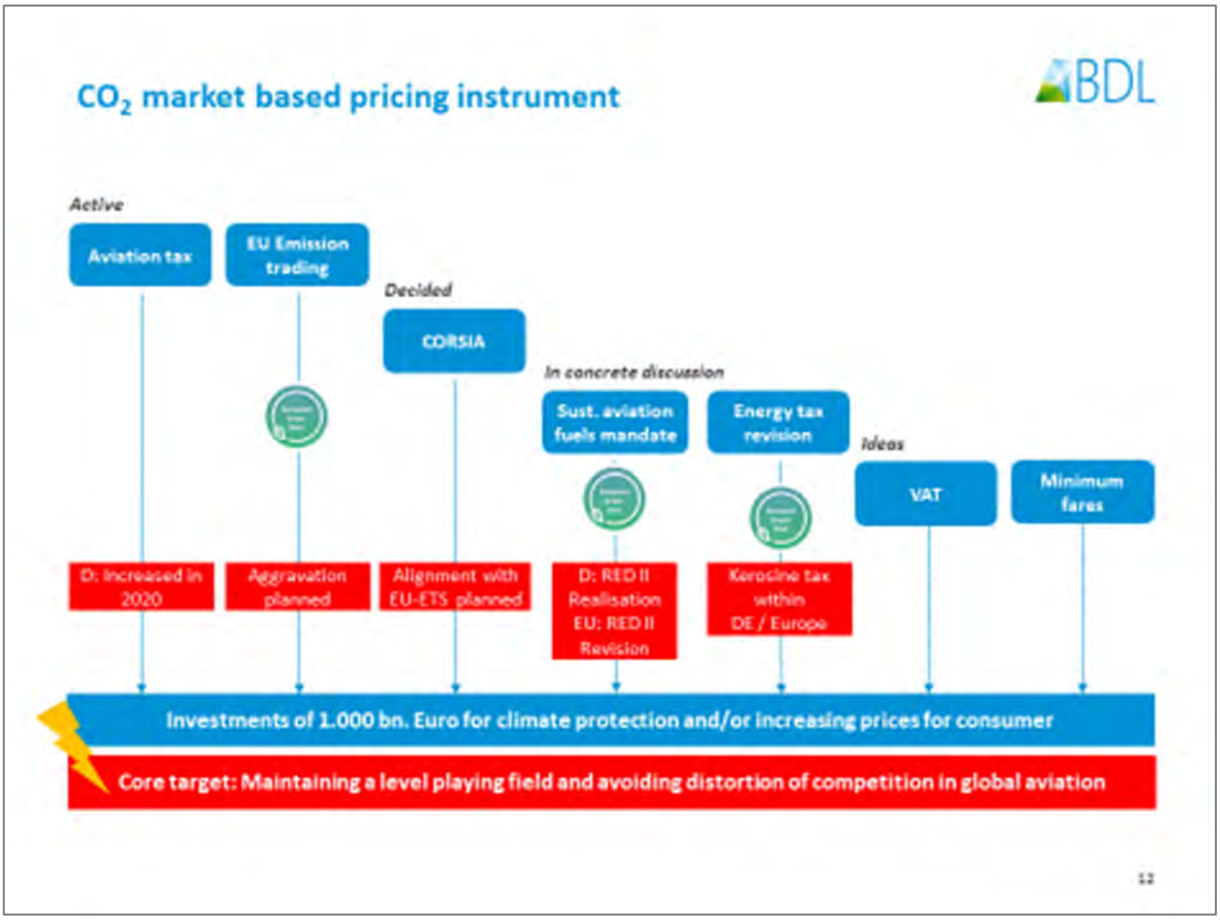

CO_2 market based pricing instrument
BDL
Active
Aviation tax
EU Emission trading
Decided
CORSIA
In concrete discussion
Sust. aviation fuels mandate
Energy tax revision
Ideas
VAT
Minimum fares
D: Increased in 2020
Aggravation planned
Alignment with EU-ETS planned
D: RED II Realisation EU: RED II Revision
Kerosine tax within DE / Europe
Investments of 1.000 bn. Euro for climate protection and/or increasing prices for consumer
Core target: Maintaining a level playing field and avoiding distortion of competition in global aviation
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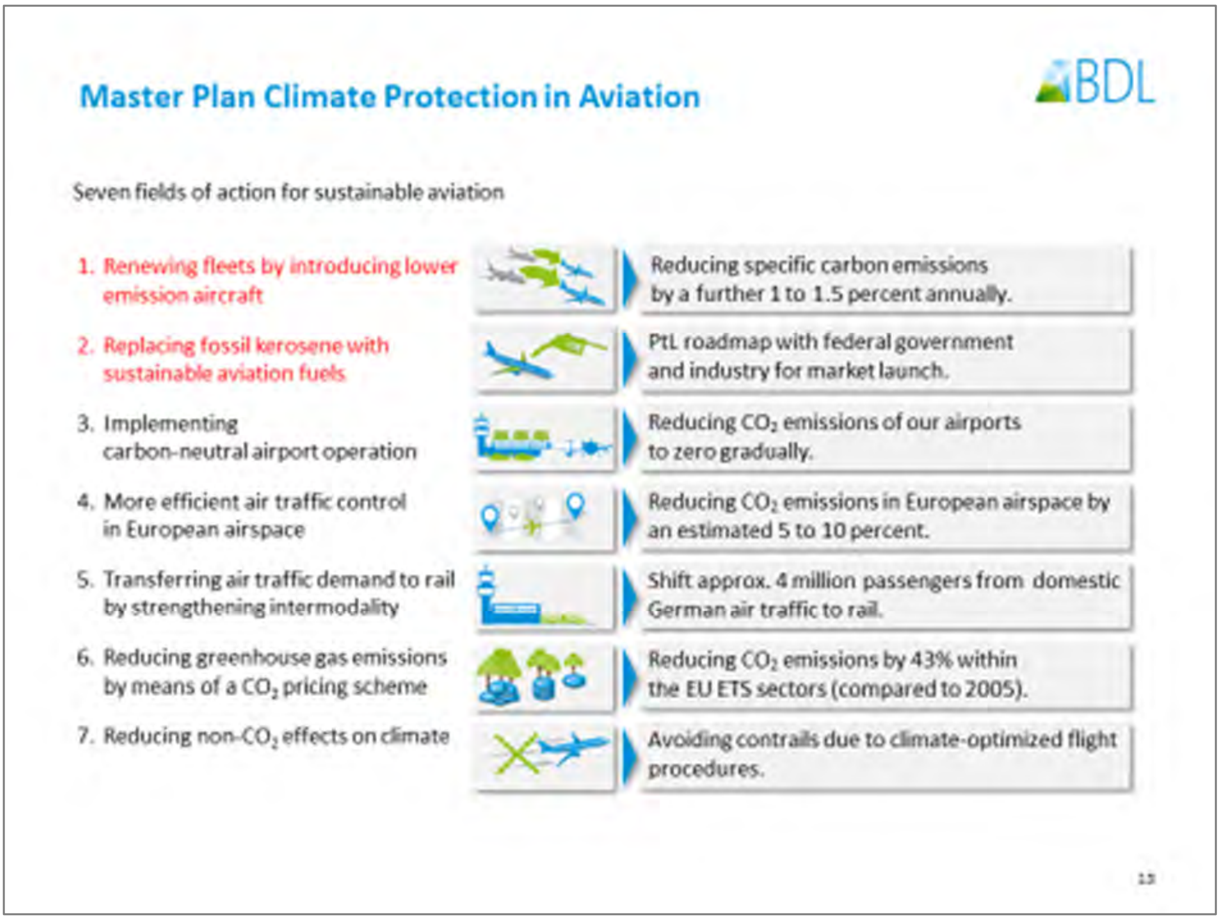

Master Plan Climate Protection in Aviation
BDL
Seven fields of action for sustainable aviation
1. Renewing fleets by introducing lower emission aircraft
Reducing specific carbon emissions by a further 1 to 1.5 percent annually.
2. Replacing fossil kerosene with sustainable aviation fuels
PtL roadmap with federal government and industry for market launch.
3. Implementing carbon-neutral airport operation
Reducing CO_2 emissions of our airports to zero gradually.
4. More efficient air traffic control in European airspace
Reducing CO_2 emissions in European airspace by an estimated 5 to 10 percent.
5. Transferring air traffic demand to rail by strengthening intermodality
Shift approx. 4 million passengers from domestic German air traffic to rail.
6. Reducing greenhouse gas emissions by means of a CO_2 pricing scheme
Reducing CO_2 emissions by 43% within the EU ETS sectors (compared to 2005).
7. Reducing non-CO_2 effects on climate
Avoiding contrails due to climate-optimized flight procedures.
13

Composition of calcium sulfonate grease: Improvement of performance through different additive systems

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Lorraine Segreto holds a Master's Degree in "Materials and Processing" where her main focus was on ceramics. Between 2007 and 2013, she worked as Project Manager for abrasives and lubricants at Tribotecc in Austria. She then became head of their lubricant department until 2016 when she took over her new job, still at Tribotecc, as "New Business & Innovative Technology Manager". In May 2018, she gained her CLS Certification

Fabian Hofmann obtained his Master's Degree in chemistry from the Friedrich-Alexander University Erlangen-Nürnberg in Germany with focus on inorganic metal-complex synthesis. Between 2015 and 2020 he had been completed his PhD-thesis in the field of surface applications and electrochemistry. Since 2020, Fabian works as a Research Scientist at Tribotecc in Austria and took over the technical support for lubricant business.

Andreas Dodos received a Master's degree in Chemical Engineering and Environmental Technology from the University of Manchester, UK. He has spent the last 18 years at Eldon's SA, with duties mainly focused on industrial and marine lubricants development. Since 2007 he has been actively involved in product stewardship with focus on global regulatory developments. He is a member in a number of professional bodies such as the ELGI, the Hellenic Maintenance Society and National Tribology Centre. Since 2010 he has been the Chairman of the European REACH Grease Thickener Consortium and he is currently serving on the ELGI Board of Directors. Andreas has extensive experience in formulation and application of lubricating grease and since 2018 he is CLGS certified by the NLGI – Certified Lubricating Grease Specialist.

Abstract
Calcium Sulfonate based greases are the fastest growing grease type as reported in the 2018 NLGI grease production survey. In a global lubricating grease market that is growing by less than two percent per year, calcium sulfonate is growing by 13 percent. This means that this particular grease type is finding uses in more and more applications. Calcium sulfonate complex greases have some inherent, distinct advantages over soap-based greases but, in some cases, the performance characteristics need to be improved.

In this study, we intend to investigate how the performance of calcium sulfonate complex grease can be improved, particularly in terms of the wear characteristics, by looking at a number of additive technologies and systems that can be used in particular types of products. Two mineral oil versions of base greases are considered with main variations on the base oil viscosity as well as the base additive package. The reason for this is to underline the versatility of overbased calcium sulfonate greases for a large number of applications.

Due to the recognised high resilience of overbased calcium sulfonate grease in terms of anti-wear and anti-seizure properties, standard test methods have been modified in order to differentiate the obtained results and highlight performance differences in high load applications.
The additivated greases are further evaluated in order to confirm that overall performance characteristics are met in the base grease as well as in the enhanced versions.

Keywords: Calcium sulfonate, overbased, solid lubricants, sulphides, additives, wear, extreme pressure

Introduction
Calcium Sulfonate based greases are the fastest growing grease type as reported in the NLGI 2018 grease production survey [10]. In a global lubricating grease market that is growing by less than two percent per year, calcium sulfonate is growing by 13 percent. First traces of this type of grease are dated from the 50's [1]. Important volumes were initially applied in steel plants and paper mills. During the last years, application volumes are observed in sectors such as marine, food and mining industries including heavy mobile equipment. A real advantage for the end user of this particular technology is the reduction of grease consumption as well as the lower bearing failures, which can be seen in the field.

Calcium sulfonate complex greases have some inherent, distinct advantages in comparison to soap-based greases. They exhibit excellent extreme pressure properties and high dropping points. When formulated with suitable components they can also be used in H1 lubricants for incidental food contact applications. Nonetheless, for most applications, the performance characteristics need to be improved.

One of the interesting aspects of sulfonate greases is that base oil viscosity is seen to have a bigger influence on the properties of the grease than it does with soap thickened grease [2]. This means that the base oil viscosity will significantly affect not only the lubricating oil film thickness in the contact zone but also significantly impact the production yield. Considering the high thickener content of calcium sulfonate complex greases compared to soap-based greases, this can have adverse effects on parameters such as pumpability and low temperature performance as well as the EP properties for lower consistency grades.

In this study, we intend to investigate how the performance of calcium sulfonate complex grease can be improved by looking at a number of additive technologies and systems that can be used in particular types of products. Synthetic as well as mineral oil versions of a base grease are considered. Also, the resulting calcium carbonate content and structure that are attributed to different structures of the overbased alkylarylsulfonic acid used as a starting material and its effect on the load and wear characteristic of the grease is evaluated.

The additivated greases are further evaluated in order to confirm that overall performance characteristics are met in the base grease as well as in the enhanced versions.

Sulfonate greases and structure
Sulfonates are metal salts of long-chain alkylarylsulfonic acids. Beside their excellent anticorrosion properties, neutral and so called overbased sulfonates with colloidally dispersed metal oxide or hydroxide additionally exhibit an excellent detergent and neutralisation potential that makes them very cost-effective multifunctional dispersant / detergent additives for engine oil [3]. Structure of overbased sulfonate is showed below.

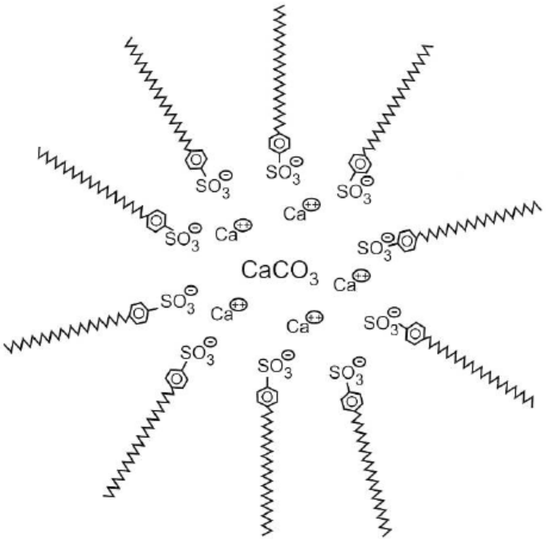


Figure 1: Model of structure of overbased sulfonate [3].

In general, the manufacturing process involves converting liquid overbased calcium sulfonate to grease containing calcite particles. Depending on the process applied during the production stages, different structures of calcium carbonate are formed. Of particular interest are two structures, calcite and vaterite, with the former being favoured due to the desirable performance characteristics that the final grease exhibits [2]. In order to produce a calcium sulfonate complex grease, various complexing agents including organic and inorganic acids can be used as a further production stage, thus improving the performance of the final product.

According to Kobylansky et. al. the lubricating mechanism of overbased sulfonate complex greases is somewhat different to overbased sulfonate additives for engine oils. While micellar nuclei of overbased additives for engine oils are characterised by the amorphous structure of $CaCO_3$, the carbonate nuclei of micelles in overbased calcium sulfonate grease have a polymorphic modification of calcite. Calcite particles having nanoscale size have a great surface activity owing to the high ratio to surface area to volume. They cover a friction surface with a certain degree of orientation forming a fish-scale layer [4] Figure 2 shows an example of the micelles of a complex overbased calcium sulfonate grease.

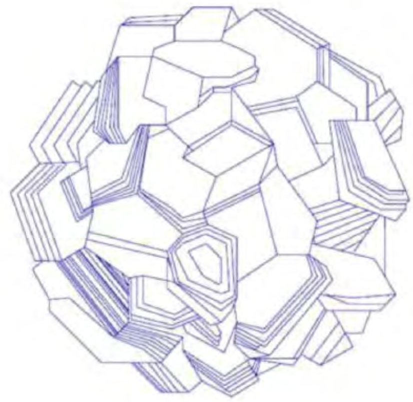


Figure 2: Micelles of overbased complex calcium sulfonate after Kobylanski, et al. [4].

Calcium sulfonate complex greases show a variability in performance, depending on the raw materials used, the manufacturing method, thickener content and additives. If solid additives are added, generally EP performance will be boosted. However, due to the lubricating properties of the calcite particles, performance additives containing sulphur, phosphorous or zinc may not be needed [5]. The aim of this study is to review a variety of additives and see if an improvement of performance can be achieved.

Grease properties

Two different base greases have been used for the preliminary evaluation of the additive systems. These greases are based on two different sources of over-based calcium sulfonate resulting in a different concentration of calcite in the final products. The main difference in the two types of calcium sulfonate starting materials relates to their carbon chain length and structure as well as their total base number. Additionally, the base oil viscosity of the two greases is different with the base grease having a viscosity equivalent to ISO VG 220 and the base grease EP having a viscosity equivalent to ISO VG 320. The typical properties of the base greases used for the study are listed in Table 1 below

The theoretical amount of calcium carbonate in the formulation is calculated based on the amount of calcium in each starting material. Table 2 gives a quantitative indication of the amounts in the formulated grease versus the TBN of the starting material.

Table 1: Base grease performance characteristics.

Performance Characteristics	Method	Base Grease	Base Grease EP
Worked Penetration, 0.1 mm^{-1}	ASTM D217	291	287
NLGI Consistency	Classification	2	2
Dropping Point, °C	ASTM D2265	338	342
Roll stability, Δ0.1 mm^{-1}	ASTM D1831 ASTM D1403	+3	-6
Extended Worked Penetration 100.000 strokes + 20% water Δ0.1 mm^{-1}	ASTM D217	+11	+17
Water washout, % wt.	ASTM D1264	1.14	0.89
Oxidation stability, min	ASTM D8206		
Oil Separation, % wt.	ASTM D1742	0.45	0.31
Cu corrosion	ASTM D4048	1b	1b
Base Oil Viscosity, mm^2/s at 40 °C	ASTM D445	220	320
Flow Pressure at – 20°C, hPa	DIN51805	1230	1690

Table 2: Theoretical amount of Calcium Carbonate in each base grease.

Performance Characteristics	Method	Base Grease	Base Grease EP
TBN of starting material, mg KOH gr^{-1}	ASTM D2896	311	403
Theoretical $CaCO_3$ in formulated grease, % wt	Calculated	8,41	10,89

Solid lubricants

Several additive systems have been tested in calcium sulfonate greases, in particular solid lubricants. Solid lubricants are necessary for improved emergency running properties where grease starvation might occur. They are also used for extreme pressure and high temperature applications, due to their high load carrying capacity and thermal stability. They effectively reduce friction and wear between contacting surfaces in relative motion. The most important property for metal sulphides remains the high load-carrying capacity.

Molybdenum disulphide, tungsten disulphide and tin disulphide fall under the category of structural lubricants. They adhere readily to most substrates and the lamellae structure provides good friction properties. Figure 3 shows an example picture of the layer-latticed structure of SnS_2.

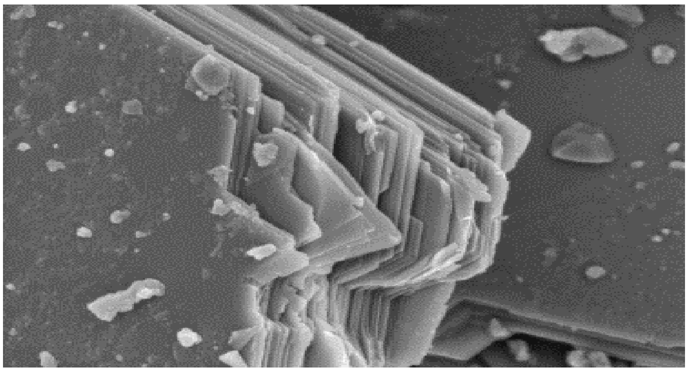


Figure 3: SEM picture of SnS_2.

Bismuth sulphide has very interesting properties as well. The face-centred orthorhombic crystal structure of the bismuth sulphide allows direct contact of the bismuth atoms with the metal surfaces making the formation of the “intermetallic compound” bismuth-iron easier. This intermetallic layer protects the surfaces from wear. Another property of bismuth sulphide is its extremely high load carrying capacity [5].

Another sulphide that has already been mentioned as solid lubricant in literature is zinc sulphide. This substance shows lubricating properties but it needs a supporting medium to ensure the transport to the contact point where friction is occurring. It acts as supporting agent or catalyst by producing friction and wear reducing layers [3].

Phosphates, hydroxides or carbonates are reactive substances which generally need a supporting medium. The mechanism of action is based on the reactivity of the product with the iron from the metallic surface to create wear resistant layers. For example, calcium hydroxide supports the production of a layer of Fe_3O_4 iron oxide on the rubbing surface of steel. This oxide has better tribological properties than the more common α-Fe_2O_3 ferric oxide, possibly because of its more favourable close-packet cubic lattice structure compared with the corundum like lattice structure of ferric oxide [3]. Some pyrophosphates also react with the surface and promote the creation of Fe_3P and FeO layers. Similarly to Fe_3O_4, FeO layers allow a significant reduction of friction and wear in comparison to Fe_2O_3, which promotes wear [6], [7].

The necessity of solid lubricants in calcium sulfonate raises some questions as, generally speaking, through the formation of calcite particles a certain amount of solid is already present. The micelle structure of calcium sulfonate grease and the mechanism of action may prevent the lubrication properties of solid lubricants and the substrate adhesion. Solid lubricants generally help in improving the extreme pressure properties however one particularity of calcium sulfonate greases is that they exhibit extremely good extreme pressure properties by nature. The aim of this study is to evaluate the potential of specially developed solid lubricant in calcium sulfonate base grease.

Test methods

Mechanical-dynamic testing has become an essential element in the development of modern lubricants. Test rigs and test methods play a decisive role in the development of lubricants and additives. To compare the friction and wear reducing properties as well as the extreme pressure properties of the new generation of solid lubricants, standardised tests have been performed with different test rigs.

The **four ball apparatus** is one of the oldest and best-known simple test rigs for liquid and solid lubricants. The test consists of a roller bearing ball rotating at constant speed and constant load on three fixed steel balls. The three-point contact is lubricated with the lubricant of interest. It allows the determination of the wear preventive properties by measuring the average wear scar worn into the fixed balls (ASTM 2266). The standard test method ASTM D 2596 describes how to determine the load carrying properties like weld point or load-wear index (LWI) of lubricants. The parameters of the standardised test methods used in this study are summarised in

Table 3.

Table 3: Parameters of the four ball test methods used in this study.

Method	Result	Temperature [°C]	Rotating speed [rpm]	Duration	Load [kg]
ASTM D 2266	Wear	75	1200	1 h	40
ASTM D 2596	Weld Point	27 ± 8	1770	10 sec	Load steps

In order to increase the precision of the results, at least two repeating measurements are carried out for each standard test method and the average results are described in the following section.

The high frequency linear oscillation test machine (SRV) is designed to simulate very small displacements under well-defined conditions of load, speed and environmental control [3]. It is used to investigate applications, where high-speed vibrational or start-stop motions are present for extended periods of time under initial Hertzian point contact pressure. The standard test method ASTM D 5706 is used to quickly determine the extreme pressure properties of lubricating greases. The pass load is defined as the highest load where no seizure occurs [8]. ASTM D 5707 is a standard testing method used to determine the coefficient of friction and the wear properties of lubricating greases [9]. The testing parameters used in this study are listed in Table 4.

Table 4: Parameters of SRV test methods applied in this study.

Method	Result	Temp. [°C]	Freq. [Hz]	Stroke [mm]	Duration [min]	Load
ASTM D 5706	Pass load	50	50	1	~40	+100N/2min
ASTM D 5707	Coefficient of friction Wear	50	50	1	120	200N

In this study, the following additives have been investigated in the calcium sulfonate base grease.

Table 5: Description of the tested solid lubricants.

Solid lubricant	Composition	Density [g/cm³]	d_{50} [µm]	d_{90} [µm]
WS_2	Pure tungsten disulphide	7.5	3	7
W/MoS	Complex sulphide based on molybdenum and tungsten	5.5	3	8
SnS2	Pure tin disulphide	4.5	3	9
Sn/ZnS_xF_y	Synergistic mixture Zn, Sn + S, fluoride	4.3	2	5
$ZnSP_xO_y$	Synergistic mixture ZnS, phosphates	2.8	2	6

Results four ball wear tests:

Standard Test Method

Solid lubricants have been added at different treat rates of 1%, 1.5% and 3%.

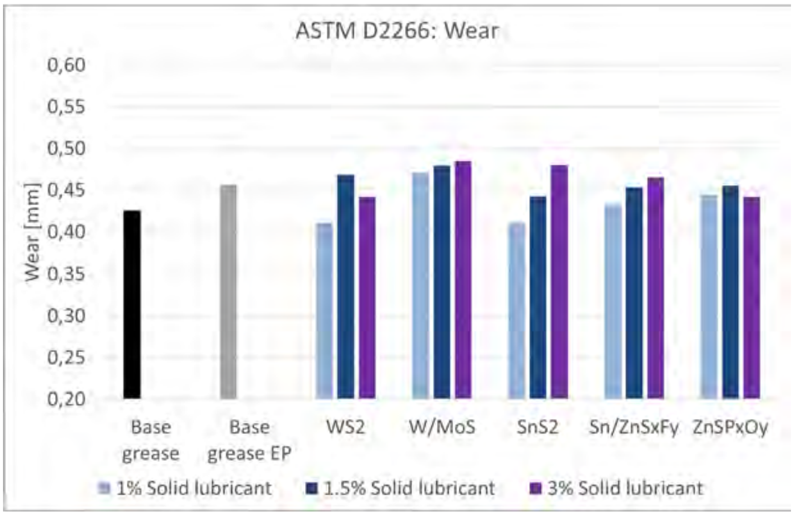


Figure 4: Results of the ASTM D 52266.

The screening tests showed that the effectiveness of solid lubricants can be limited in calcium sulfonate complex grease under the testing condition of the ASTM 2266. Tin disulphide and tungsten disulphide show a reduction of the wear at a lower treat rate. When the concentration of solid lubricant is increased to a 3 % treat rate, the wear is slightly higher. Generally, solid lubricants only have a very small influence on the performance. It was demonstrated that in some cases, performance is reduced with the addition of solid lubricant compared to the results obtained by the base grease. Additional tests at higher load confirm the findings. Results are summarised in the Table 5.

Table 6: Summary of test results.

	ASTM D 2266 Wear (mm)			ASTM D 2596
Base grease	0,43			> 800 kg
Base grease EP	0,46			> 800 kg
	1%	1,50%	3%	
WS2	0,41	0,47	0,44	> 800 kg
W/MoS	0,47	0,48	0,49	> 800 kg
SnS_2	0,41	0,44	0,48	> 800 kg
Sn/ZnS_xF_y	0,43	0,45	0,46	> 800 kg
$ZnSP_xO_y$	0,44	0,46	0,44	> 800 kg

Independently from the solid lubricant used and the concentration, the greases all succeeded to pass a weld point of 800 kg. The last non-welding load of the base grease and base grease EP is at 160 kg as shown in Figure 5.

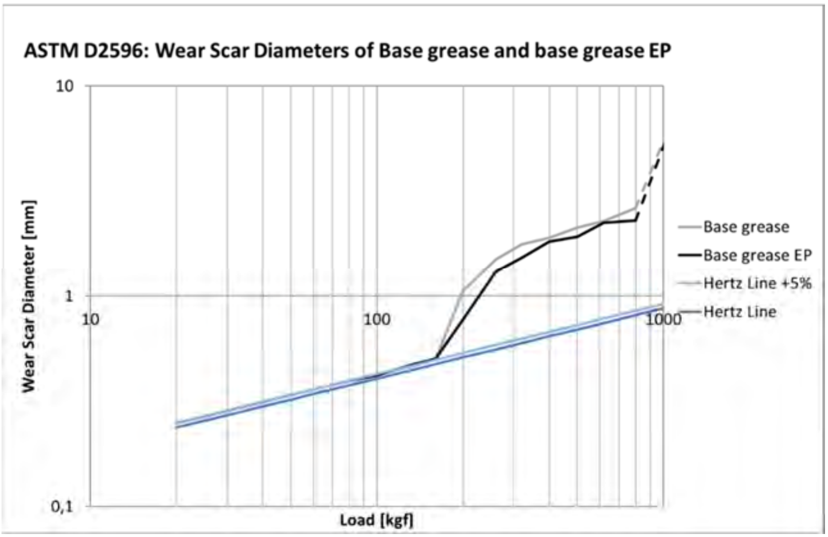


Figure 5: ASTM D 2596: wear scar diameter at different loads.

The difference between the base grease and the base grease EP is marginal in this test. Base grease and base grease EP both show extremely good extreme pressure properties with load wear indexes exceeding 140 (respectively 142 and 160).

Modified test method

As mentioned previously, calcium sulfonate grease has a recognised high performance in terms of wear resistance partly due to the inherent properties of the thickener system. This is particularly obvious when performance of a base grease is compared to that of a standard lithium grease and how a number of performance additives affect the test results.

This does not allow for a clear differentiation between the performance improvement using an additional additive package in the formulation at standard test loads as these are mainly designed to cope with the performance level of lithium or calcium-based greases.

A clear example of this is the welding point; we observed that none of the tested formulations welds even at the maximum design load of 800 kgf of the test rig. Additionally, it has to be noted that even in the relevant ASTM method (D2596) no repeatability results can be ensured with step loads higher 700 kgf. With more and more high-performance greases being designed and used in industrial applications this needs to be addressed.

For the purpose of this study, the test method for wear (ASTM D2266) was modified in terms of the applied load for the duration of the one-hour test. The standard load of 40 kg was increased to 100 kg. It has to be noted that this performance parameter is extreme for standard lithium greases. When this test was carried out on a standard lithium grease, the four balls welded after running the test for less than 45 minutes.

For the calcium sulfonate greases under evaluation, the results are listed in Figure 6 below.

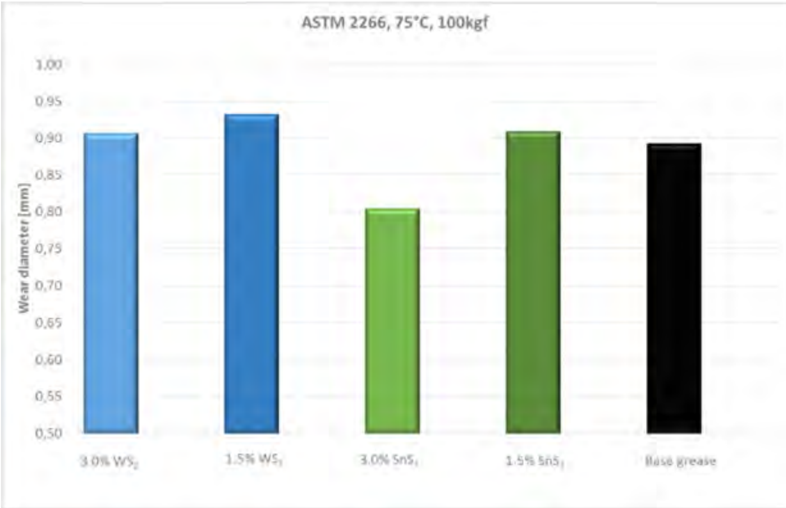


Figure 6: Results of the modified ASTM D2266 at 75°C with 100 kgf load.

With the increased load of 100kgf, the wear diameter could be decreased by about 10% using 3.0 wt. % SnS2 compared to the base greases. Moreover, this shows that it is crucial to adapt test parameters to ensure a proper comparison of additives in high performance greases.

Results of SRV wear test ASTM D 5707

The greases have been tested under the conditions of the ASTM D5707. Two repeating measurements have been carried out and the results are shown below.

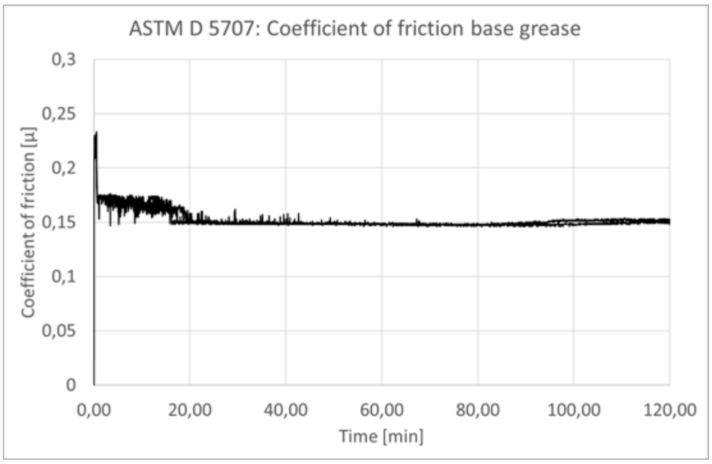


Figure 7: Coefficient of friction of the base grease.

A running-in effect is observed in the first 20 min. Indeed, the coefficient of friction is higher and less stable.

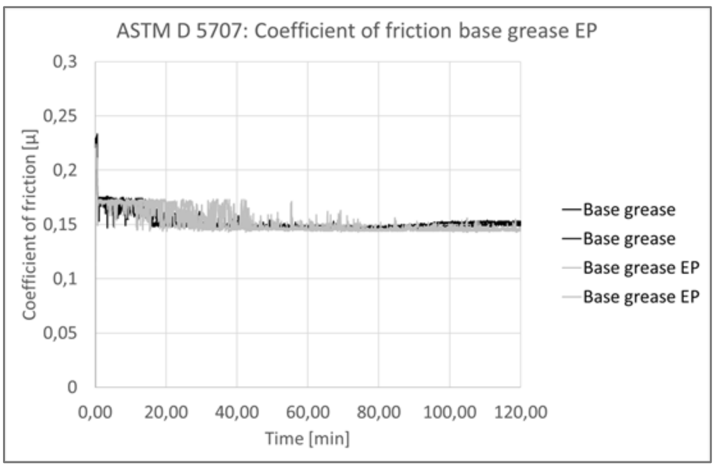


Figure 8: Coefficient of friction of the base grease EP.

The base grease EP shows a very similar behaviour to the base grease. The running-in phase lasts slightly longer, around 40 min.

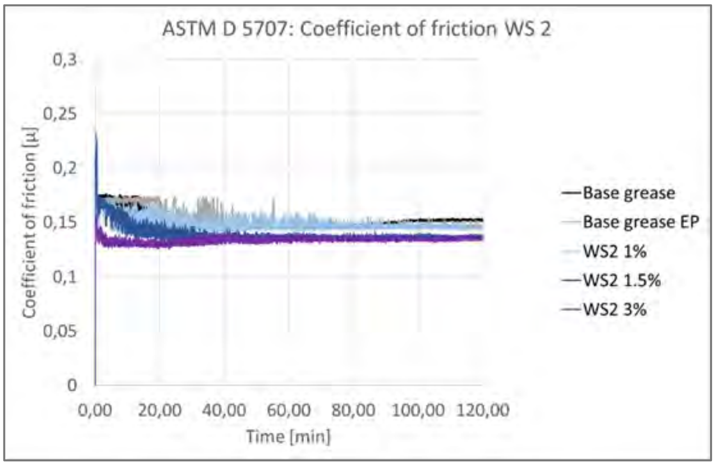


Figure 9: Coefficient of friction of WS_2.

The best results in terms of coefficient of friction are obtained at higher concentrations. The grease containing 3% WS_2 shows a stable behaviour from the beginning of the measurement and a lower coefficient of friction.

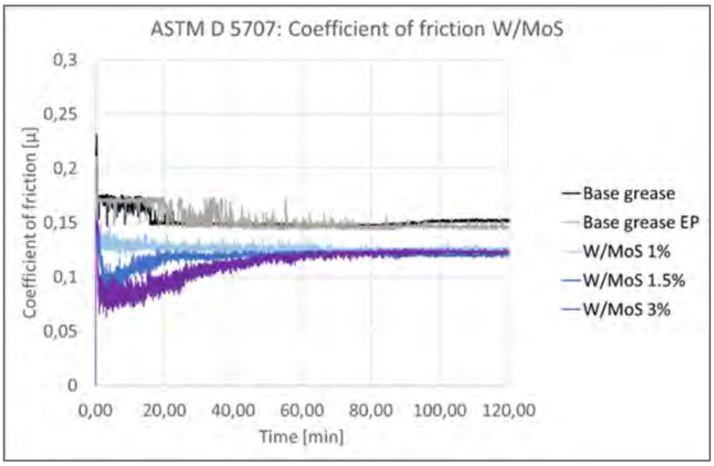


Figure 10: Coefficient of friction of W/MoS.

A similar behaviour as with WS_2 is observed and the higher the concentration in solid lubricant the better the coefficient of friction. The main effect is observed in the running-in phase. The additive lowers the energy needed to run the system until a stable state. At the end of the measurement, the coefficient of friction of the different additives at different concentration stabilises at the same value.

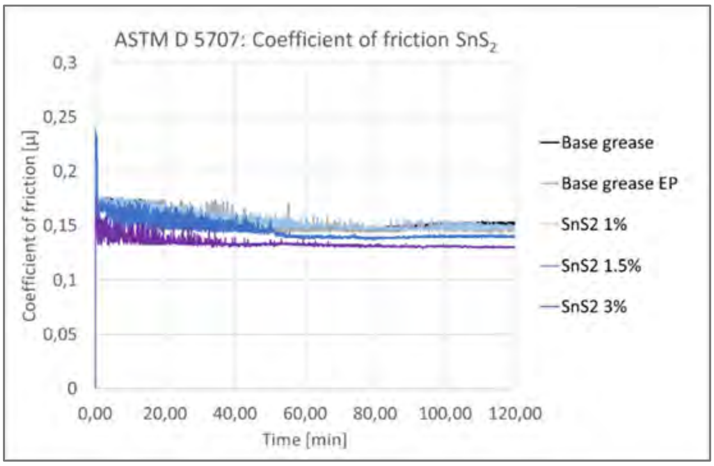


Figure 11: Coefficient of friction of SnS_2.

Here again the coefficient of friction is reduced with the increasing concentration of solid lubricant.

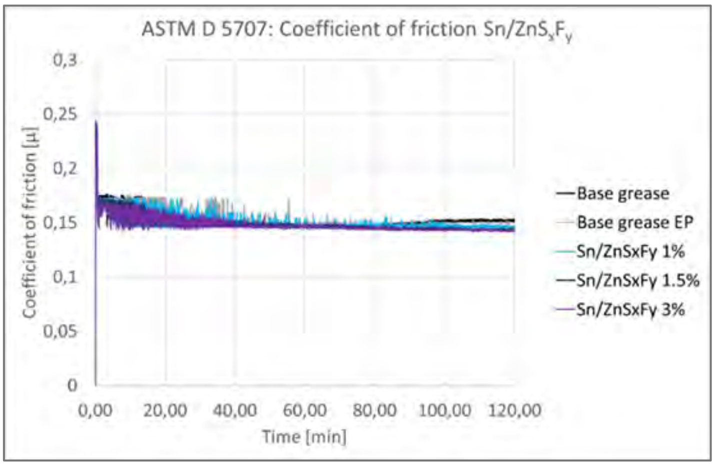


Figure 12: Coefficient of friction of Sn/ZnS_xF_y.

The coefficient of friction seems to run smoother with a higher solid lubricant content
but there are no significant differences at different treat rates and the coefficients of friction of the different greases are at the same level as the base grease

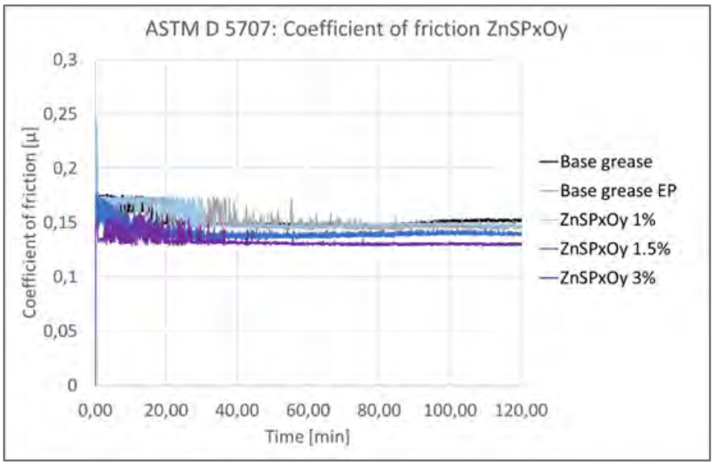


Figure 13: Coefficient of friction of $ZnSP_xO_y$.

The same trends can be observed and the higher the solid lubricant content is, the lower the coefficient of friction is for this additive based on zinc sulphide and phosphate

Concerning the coefficient of friction, solid lubricants contribute to reduce the value. Generally, the higher the concentration, the lower the value of the coefficient of friction, except for the product Sn/ZnSxFy. The wear results associated to the measurements according the ASTM D 5707 are shown Figure 14.

The wear results do not follow the trend. It seems that the higher the concentration of solid lubricant, the higher the wear is particularly for tungsten containing products. The most promising additive is the synergistic mixture that does not show any significant difference at the different concentrations. The substance ZnSPxOy combines a reduction of coefficient of friction and a stable wear.

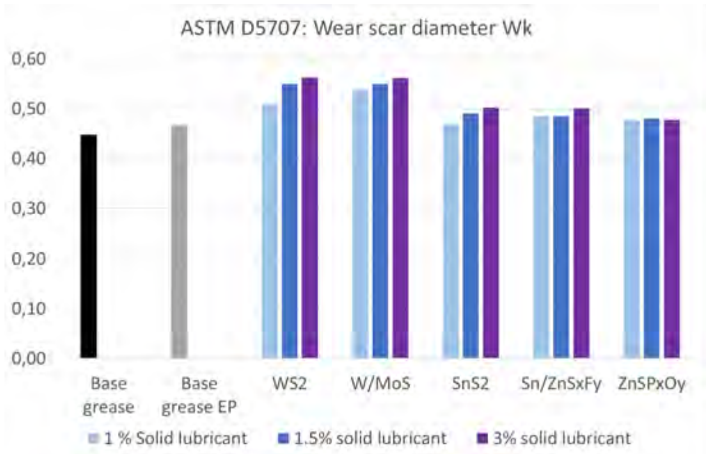


Figure 14: Wear results under ASTM D 5707.

Discussion

Tungsten containing additives seem to react differently to the calcium sulfonate base grease. As a comparative value, the wear at a 3% treat rate in a lithium base grease is compared to the wear obtained at a 3% treat rate in a lithium grease (Figure 15). Besides the enormous difference of wear scars between the two base greases, it can be observed that tungsten containing additives performed way better in lithium base. On the other hand, Tin and zinc sulphide based additives show lower wear scars in the calcium sulfonate base, which suggests different interactions and mechanisms within the different base greases.

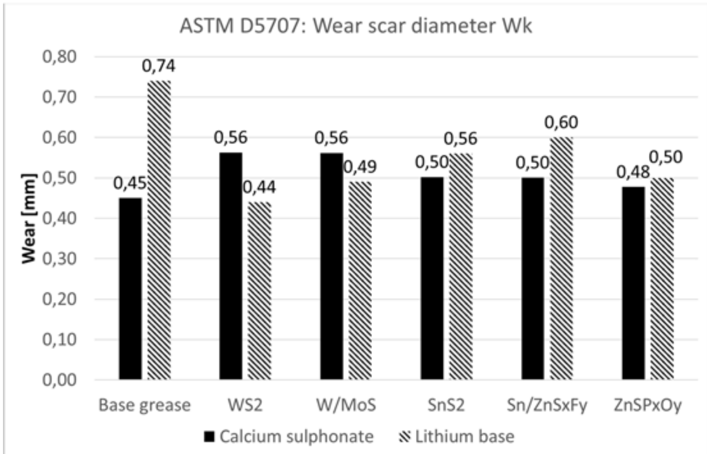
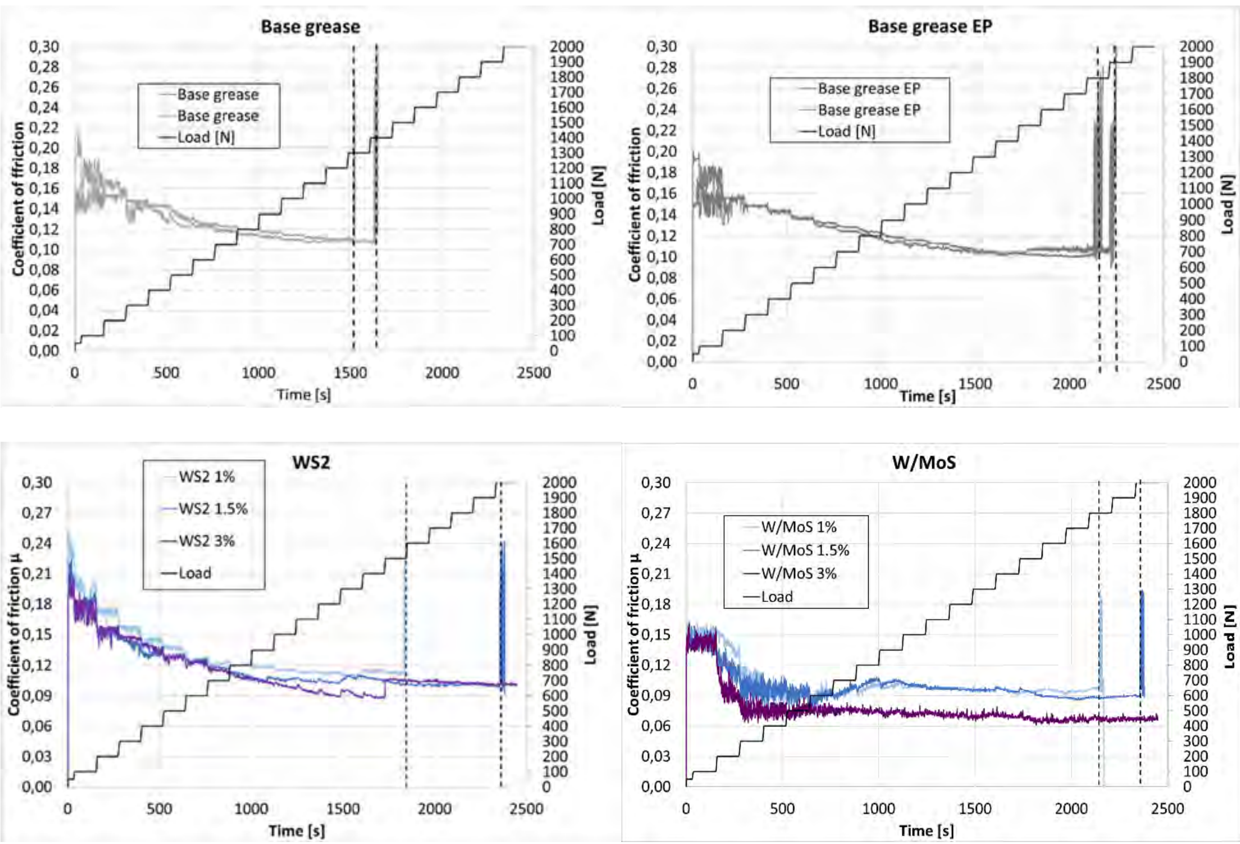


Figure 15: Comparison of the wear between Lithium and Calcium sulfonate grease at a 3% treat rate.

According to the ASTM D5707 testing procedure, the most promising compound is ZnSPxOy as it is able to reduce friction by keeping the wear at a stable value and very close to the base grease. A lower coefficient of friction means less friction synonym of reduction of the energy consumption of the tribosystem.

Results of SRV EP test ASTM D 5706

The ASTM D 5706 is performed on the greases. At least two repeating measurements have been carried out. Representative measurements are reported in the figures below.



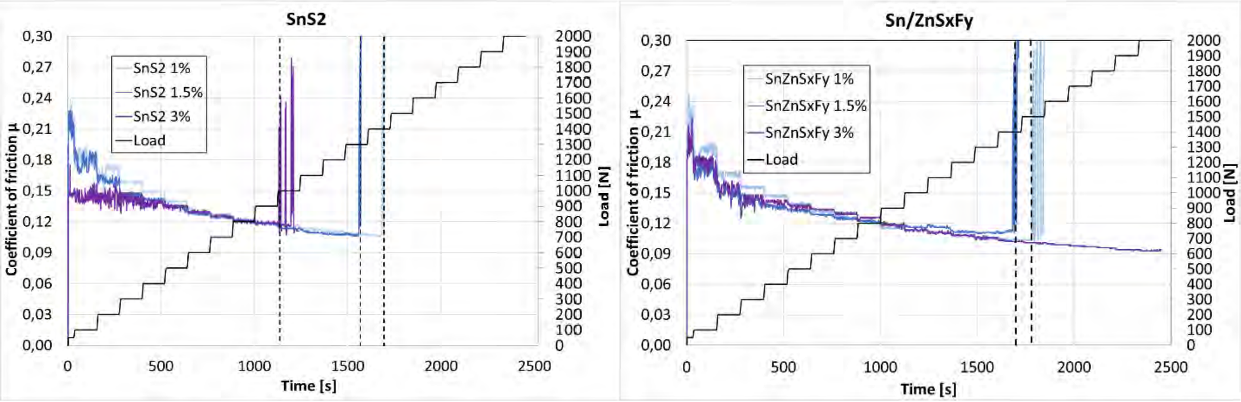


Figure 16: Results of ASTM D5706 of Base grease; Base grease EP; WS2; W/MoS; SnS2 and Sn/ZnSxFy.

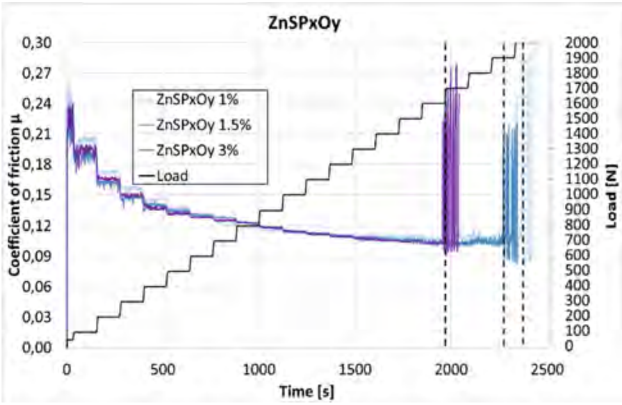


Figure 7: Results of ASTM D5706 of ZnSPxOy.

The pass loads are summarised in Figure. A higher pass load means a more stable tribofilm along the measurement. The data suggests that the tungsten-based additives at higher concentrations allow higher pass loads and extreme pressure properties. In comparison to the base grease, W/MoS shows a positive effect already at a 1% treat rate. On the other hand, SnS_2 shows lower performance when the concentration is increased and does not perform as well as other solid lubricants in this test. ZnSPxOy shows the same trend, and the performance is decreasing as the concentration is increased. Nevertheless, the performance at 1% remains interesting.

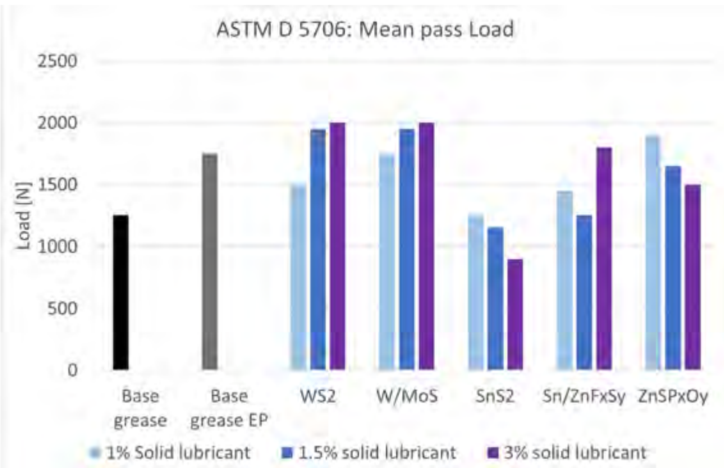


Figure18: Mean pass loads of the greases according to ASTM D5706.

Solid lubricants can contribute to the improvement of the EP properties under oscillating motion. However, the choice of chemistry and concentration play a very important role.

Summary of the Performances of Standard Test Methods

The performances of the different additives are summarised in the spider charts below. Performances are ranked from 0 to 10, where 0 means moderate performance and 10 very good performance.

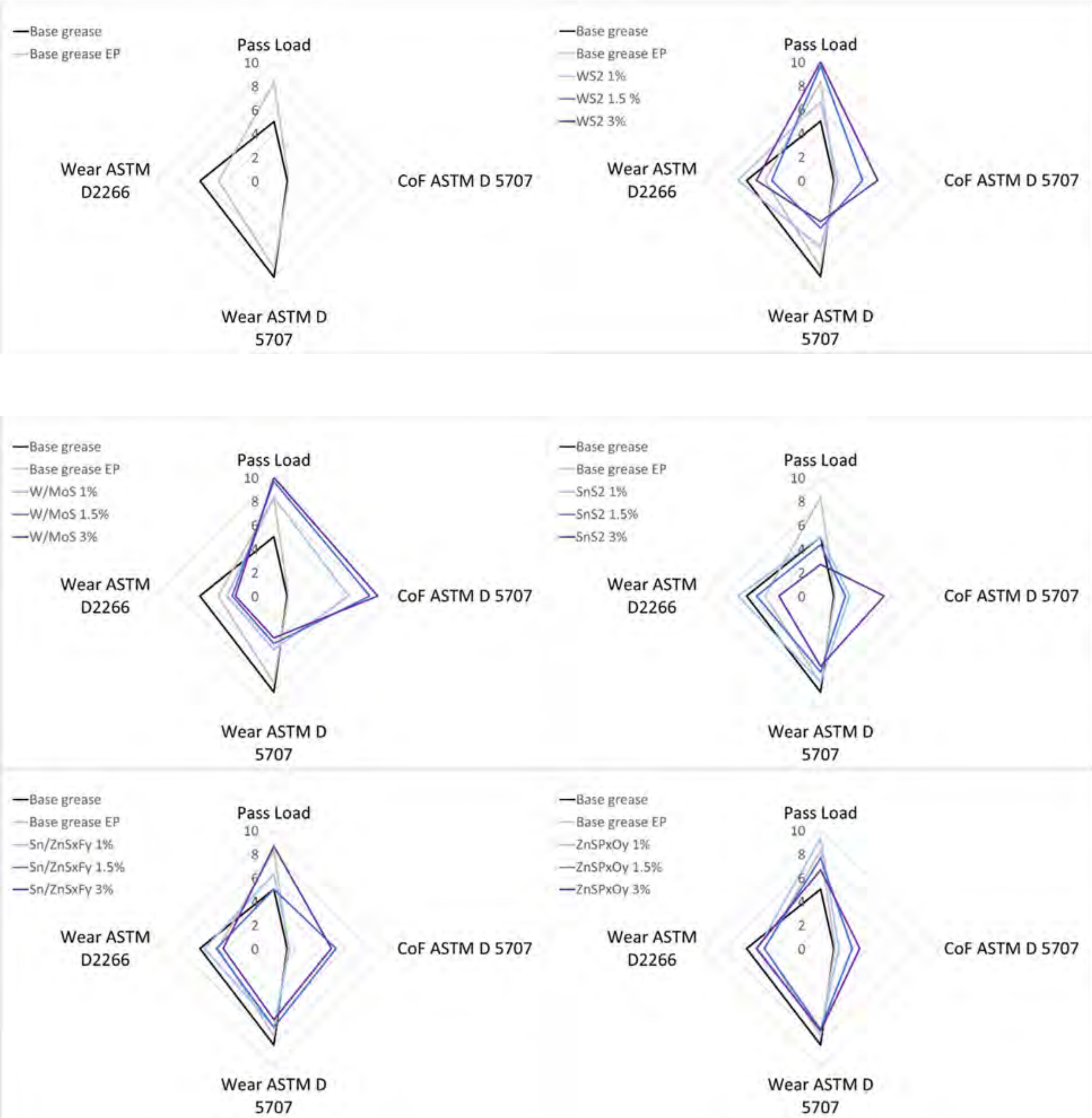


Figure 19: Performance overview of the different additive systems according to the standard test methods.

The most impressive performance improvement could be obtained on pass load and coefficient of friction using the product W/MoS. Unfortunately, the improvement on the one side, shows a deterioration in the wear performance on the other side. The Product ZnSPxOy is a good compromise as the wear performances remain very similar but the coefficient of friction and the pass load can be improved. This effect can already be observed at concentrations as low as 1%. SnS_2 shows a slightly different behaviour and only the coefficient of friction can be improved. In a previous work, this additive in combination with MoDTP showed the best overall performances in lithium grease. The results here are surprising and show that common additives used in Lithium base grease are not adapted anymore for calcium sulfonate greases.

Conclusions

Different additive systems have been tested at three different concentrations in a calcium sulfonate base grease. The base grease itself reaches excellent performances without any additives particularly on four ball weld point which is above the limit of the test rig. The additive systems contribute to performance improvement particularly in terms of coefficient of friction. However, additive systems react totally differently. A higher concentration of additive does not always mean better performance. Standard additives working for lithium greases are not adapted. The coefficient of friction can be significantly improved. However, it often means a loss in the wear performance in the bench tests according the common standards. The additive system based on W/MoS contributes to significant improvement of the coefficient of friction and pass load. If a solution was found to counterbalance the loss in wear performance, then the additive system would be a consistent solution for performance improvement of calcium sulfonate greases. Further investigations toward this target are ongoing.

The test methods used in this study highlight that modifying standard wear test methods can help in the differentiation between performance of given additive packages. As noted above the thickener system of calcium sulfonate grease reacts differently than say lithium grease. Also, calcium sulfonate grease is designed for use in applications that are more demanding in terms of high load performance compared to lithium greases. In the same way, traditional test methods are not the most suitable for testing these types of greases. A clear example of this is welding point, which exceeds the limits considered up to now by the standard ASTM method. Also, the LWI far exceeds what we typically expect with lithium greases. Finally, in terms of the wear, it can be seen that the scar diameter measured on a base calcium sulfonate grease tested at a load of 100 kgf is similar to that of a base lithium grease at 40 kgf. All of the above parameters need to be considered by grease manufacturers as well as end users in the future.

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Company Profile: HL Blachford

Following on from ELGI's successful 2021 Spring Virtual Events, HL Blachford was one of the companies that generously sponsored these events. To help you get to know HL Blachford, we have arranged for Suzy Jarman to interview Mr. Howard Kennedy (Vice President of Sales).

SJ: Hello Howard, thank you for agreeing to be interviewed. I'd like to start off by finding out a little about yourself. What is your job title, how long have you worked for HL Blachford, and what made you decide to work for them?

Hi Suzy. My name is Howard Kennedy. I am the Vice President of Sales. I joined Blachford about 8 years ago. I chose to join Blachford for a number of reasons but the main ones were its portfolio of a number of unique chemistries (such as Lithium 12 Hydroxystearate), its strong R&D and Sales staff and its commitments and efforts in the area of sustainability.

SJ: How does the ELGI provide a platform for your company?

Blachford became aware of ELGI through our participation in NLGI. We have found that participation in the ELGI is the best method to reach European grease manufacturers

SJ: What's it like to work for Blachford and what are their unique selling points?

Blachford is a great place to work. The key selling points for Blachford (with respect to grease) is the unique pre-formed thickener technology that helps reduce the time and energy required to make grease.

SJ: Howard, please tell me about Blachford, why was the company founded and when? And who founded it? Thinking about the development of Blachford what were the pivotal points and when did they occur (can a timeline be provided)?

Blachford was started in 1921 by Henry Lloyd Blachford in Montreal and is celebrating its 100th year of continual operation. In the mid-50's, Blachford began to produce more and more fatty acid based products (stearates) and built a new plant in Mississauga (near Toronto) in the early 70's. In the mid-90's Blachford developed a pre-formed lithium 12 hydroxystearate for a major Canadian grease manufacturer and has since developed that technology so that it is supplied globally. The most recent development is the HX-1 listing of that main lithium technology.

SJ: In which countries is Blachford active and how many people work across all these locations? How long did it take to get there? How has Blachford benefitted from a global presence?

*Bl*achford has approximately 350 employees across 3 divisions. Most of those people work at the 2 plants in Canada and the 4 plants in the USA. Blachford also has a warehouse and manufacturing site in the UK. Blachford operates approximately 14 warehouses and sales offices. Most of those facilities are in North America but there are sites in UK and Korea. Via distribution partners and directly, Blachford's products are supplied globally into most geographical markets including Europe, China, Taiwan, South America, the Caribbean, India, Australia, Middle East and Africa

SJ: Looking back over the years, which products and/or services were created originally and how do they compare with today?

Some of the products Blachford supplies are the exact same formulations that were developed decades ago because they meet the customers' requirements in terms of both cost and performance (if it ain't broke…). However, products are regularly improved to take advantage of new technologies and/or manufacturing processes in order to reduce cost, improve performance and improve sustainability. The best example of this is the stearate manufacturing method. Originally, stearates were made using a 'precipitation method' which produced a significant amount of environmentally unfriendly effluent. Now, most stearates are made using a more efficient and less expensive 'fusion' method in which no effluent is produced. The performance of the product produced by either method is identical in most cases. Blachford is also continually developing new products to meet new customer requirements and/or to replace old technology. The latest HX-1 offering from Blachford is an excellent example of that.

ELGI AGM London 2018

SJ: Does Blachford do business online, and does your company serve consumers, businesses or both, and which marketing channels do you use to promote your business?

The chemical industry has been very slow to move to an 'online' purchasing process because it largely provides little or no additional value to the customer due to the industrial nature of most of our business. Unlike the retail consumer business, most industrial businesses involve much larger transactions that are much more complicated with contracts, certificates of analysis and purchase orders. The vast majority of the business Blachford does with its customer base is on a 'direct' basis. The main steps to becoming more 'online' is to provide the customers with easier access to information via website access.

SJ: Reflecting on the recent global issues, what has been the most challenging aspect for the past year?

There have been three significantly challenging aspects for Blachford over the past 'pandemic' year. The first issue has been the challenge to keep our workers safe and our plants fully operational. To date Blachford has been largely successful with only a very few of our employees coming down with Covid and almost no plant downtime. The second challenge has been multiple supply chain issues that have restricted supply and increased costs including the lack of shipping containers, dramatically increased freight rates and force majeure declarations by a number of base chemical suppliers. The final challenge has been the significantly increased demand across a wide range of sectors.

Aldo Pighin at the ELGI AGM Athens 2019

SJ: There is a saying "adversity contains within it the seeds of opportunity and growth". As we head out of our recent global problems, what opportunities (digital or otherwise) do you foresee being developed?

I think that one of the biggest opportunities (for business) that has come from the pandemic is our ability to do business remotely very successfully. There will certainly always be a need to meet customers and suppliers and stakeholders 'in the field' in order to get a better understanding of problems, issues and opportunities but I think that we will be able to accomplish things much more efficiently than we did in the past by using the various video conferencing tools that have been developed. I know that many of my colleagues will be very reluctant to return to 2 hour face to face meetings when we have largely been accomplishing the same results with a 30 minute (or less) video conference.

SJ: Howard please tell me, what is the vision and mission of Blachford?

Blachford's vision is to make our customers' business better by providing innovative and sustainable chemical solutions.

SJ: And what are Blachford's core values, goals and objectives?

Blachford has dedicated itself, its technology and its business practices to sustainability – the betterment of society, the environment and the economy. Blachford follows the Responsible Care ethics and principles to innovate for safer and more environmentally friendly products and processes, and to work to eliminate harm throughout the entire life cycle of our products.

SJ: Are you able to share any future projects in the pipeline?

Blachford's latest project is the new HX-1 product using pre-formed technology for the grease industry

SJ: Please tell me about Blachford's markets and industry segments?

The chemical divisions of Blachford service a wide range of markets and segments with fatty acid chemistries that mostly act as lubricants in some form or fashion but sometimes provide unique properties. Some of those markets include grease pre-formed thickeners, lubricants for thermoplastics, stabilisers for PVC, oxo-biodegradable plastics, process aids for rubber, lubricants for powdered metal processing (pre and post sintering), anti-tack, mould release and lubricants for rubber, wire drawing compounds, metal working fluids, animal feed additives, concrete additives, abrasive paper coatings, food and pharma additives and oil field gellants,

SJ: How and where can Eurogrease's readers find out more about Blachford? Is it present on any social media platforms?

Blachford uses LinkedIn as its primary social media platform. All are welcome to follow the company for regular updates and/or to connect with me on LinkedIn.

SJ: And if we want to find out more about Blachford, how do we do that?

The easiest way to find out more information about Blachford is via our website at Blachfordcs.com

Howard Kennedy (Vice President of Sales) and Aldo Pighin (Product Manager) at the ELGI AGM London 2018

Company Profile: Cocan Graphite

Following on from ELGI's successful 2021 Spring Virtual Events, Cocan Graphite was one of the companies that generously sponsored these events. To help you get to know Cocan Graphite, we have arranged this interview with Mr. Adam Tseng. Interviewer Suzy Jarman

SJ: Hello Adam, it is good to meet you, and thank you for agreeing to be interviewed. I'd like to start off by finding out a little about yourself. What is your job title, how long have you worked for Cocan Graphite, and what made you decide to work for them?

Hello SJ, I would like to congratulate the ELGI on a successful online conference and we are honoured that we could participate at the technical presentations and exhibition. I am the founder and General Manager of Cocan Graphite and have been working at Cocan Graphite since it was established in 1998

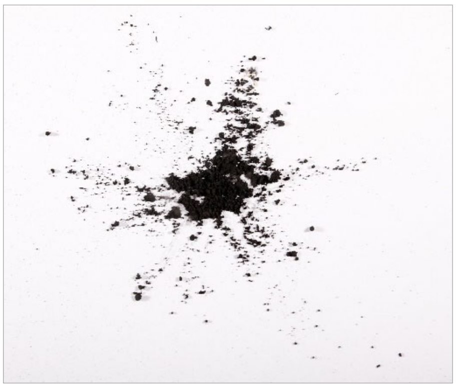
SJ: Adam, what is it like to work for Cocan Graphite and what are its unique selling points?

Cocan started up as a small to medium sized company by producing OEM powder carbon and graphite for a number of western carbon and graphite companies established in North America, Europe, Asia, Oceania, etc. Cocan has set up a comprehensive capacity by developing and producing many types of powder carbon and graphite for various industries. Cocan also owns a cutting-edge carbon R&D centre to ensure the quality and consistency of our products. Cocan has successfully shifted its corporate strategy from OEM products for carbon and graphite dealers to direct sales of tailor-made products for industrial end users. Cocan adds a lot of value to our full line of product portfolios with our mature supply chain management, internal zero defect quality management system, agile just-in-time door-to-door delivery etc.

Cocan Exhibition

SJ: Adam, please tell me about Cocan Graphite; why was the company founded and when? And who founded it? Thinking about the development of Cocan Graphite, what were the pivotal points and when did they occur (can a timeline be provided)?

Cocan was established at the end of last century, to be precise, in September of 1998 when the Chinese macro economy rapidly evolved from centrally-planned-economy to market-oriented-economy. State-owned export companies slowly went bankrupt while newly emerged private factories were not able to provide good quality management, swift mutual communication, fluent export procedures, etc. due to various different reasons. At that time, I decided to establish a decent company, to be honest about the products, and to be transparent in communications and transactions. Coincidently, at that time, the internet was introduced in China and since I could converse in English, I was able to reach most of the esteemed carbon and graphite companies outside China. I had insight, and with determination, bravely



Fine Powder

started Cocan, and within time, proved that I had the right foresight which eventually secured the success of Cocan. We still have a long way to go, but by continuously upholding our initial concept of selling authentic products and maintaining a good relationship with our customers, we have the ability to continue with our success. We appreciate the difficult times we are in, and more so appreciate our customers' support from all over the world.

Speaking of the pivotal points during our development over the past 23 years, I think the most important event occurred in 2014. Prior to 2014, our strategy was to become China's biggest powder carbon and-graphite OEM manufacturer and supplier to a couple of well-known carbon and graphite companies outside China. We were successful during the first 14 years (1998 – 2012). But between 2012 and 2014, we found that the traders and dealers had brought prices down to a situation where Cocan nearly had to close its production. So we made a strategical change; modifying our strategy to tailor-make carbon and graphite products for industrial end users by gradually utilising more and more automation in the carbon industry. Now, we are in the process of implementing such a strategy, but as mentioned before, we still have a long way to go.

SJ: In which countries is Cocan Graphite active and how many people work across all these locations? How long did it take to get there? How has Cocan Graphite benefitted from a global presence?

Cocan /R&D Centre

Cocan Graphite is based in China since the very beginning and we have been handling our global markets from our corporate office in Wuhan. Our sales team serves our global customers with multilingual capacity, Chinese, English, French, Spanish, Japanese and Korean. We have been trying to minimise the costs of our global marketing by doing direct sales instead of going through third party dealers to benefit the industrial end users. We will look into opening more local operations globally if the need is there to serve our global customers.

SJ: Looking back over the years, which products and/or services were created originally and how do they compare with today?

Cocan has established a top-notch R&D centre and has implemented more automation compared to most other powder carbon and graphite companies in the market. On the material side, there was not sufficient space for us to be particularly creative since carbon and graphite are products with a long history in traditional industries perhaps with the exception of the newly emerging energy segments. Cocan stays mostly inside the traditional industries and we are looking into new opportunities in the green energy industries.

SJ: Does Cocan Graphite do business online, and does your company serve consumers, businesses or both, and which marketing channels do you use to promote your business?

Yes, we serve our customers on-and-off-line, but mostly off-line because graphite is an industrial raw material, not a daily consumption. We promote our products via exhibitions, conferences, off-line calls, on-line magazines, etc., but nowadays, more and more customers are reaching us by our good reputation. All customers love inexpensive but good and true products. Staying humble, staying hungry, and staying true will be always our philosophy in business.

SJ: Reflecting on the recent global issues, what has been the most challenging aspect for the past year?

The most challenging aspect would be the impact of Covid-19. Covid-19 has strongly stirred up the world and a lot of business models will be re-structured due to the complexity of geopolitical alteration and in-balance of international trade relationships between different continents or countries.

SJ: There is a saying "adversity contains within it the seeds of opportunity and growth". As we head out of our recent global problems, what opportunities (digital or otherwise) do you foresee being developed?

Opportunities and growth always start up primarily from the inside and not from the outside, so what we are thinking is always how to continuously improve ourselves no matter whether it is before or after Covid-19. As Daoism says, 'You are invincible if you have no adversaries in your mind but yourself.' By structuring the best company, by making the best products, with best pricings, then we will reach our goal no matter what happens inside or outside China. At the same time, we are watching the current global problems and of course we are not able to solve these problems and we prefer to leave these to the politicians. We hope that they will be more rational rather than insane.

SJ: Adam, please tell me, what is the vision and mission of Cocan Graphite?

Our vision and mission are clearly stated on our website. Nothing big, but we just want to be one of the best powder carbon and graphite suppliers in the world and be a good supplier so that our global customers will pat us on the back.

SJ: And what are Cocan Graphite's core values, goals and objectives?

To be concise, our core value is to be true and our main objective is to be there.

SJ: Adam, are you able to share any future projects in the pipeline?

We do have a couple of projects in the pipeline and will reach out and advise our customers when needed.

SJ: Please tell me about Cocan Graphite's markets and industry segments?

We have been selling into a great number of industrial applications since we converted our corporate strategy. We are targeting most of the traditional industrial applications and we are looking for more opportunities in the new energy sector.

SJ: How and where can Eurogrease's readers find out more about Cocan Graphite? Is it present on any social media platforms?

We can be contacted at www.cocangraphite.com, but the best way is to communicate directly with our sales staff at LinkeIn.com or via email info@cocangraphite.com.

Our Sales staff will gladly take up contact.

Do Polymers Affect the Mechanical Stability of Grease?

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Erik Willett is the technical director at Functional Products Inc. in Macedonia, Ohio. His key research interest is the role of polymers and their structure in advancing lubricant performance. He has received the 2018 NLGI Development Author Award and the P.P.C. Gonsalves Memorial Award. Erik earned his PhD in polymer science from the University of Akron

Keywords: Mechanical stability, roll stability, shear, polymer additives, grease polymer, consistency, yield

Abstract

Polymer additives are routinely called on to improve the performance of grease in areas such as resistance to water spray-off and wash-out, static oil bleed, adhesion, and tackiness. The role of grease polymers in the 'mechanical stability' of grease is less known.
Mechanical stability refers to low changes in consistency after mechanical shearing by methods like worked cone (ASTM D217) and roll stability (ASTM D1831). Data collected over the years indicates that certain polymers reduce consistency loss in these tests. Higher molecular weight polymers tend to provide higher mechanical stability in grease.

Surprisingly, that trend in grease performance opposes the well-known convention in lube oil that lower molecular weight polymers are required for high mechanical stability in gear oil and hydraulic fluid.
This study seeks to 1) investigate how polymer structure influences the mechanical stability of grease, and 2) explain why high MW polymers contribute shear stability in grease but instability in lube oil.

1. Introduction

Preliminary Studies

The evidence presented in conferences and customer development over the decade shows that polymers affect the mechanical stability of grease. Until now, an overarching study has not been established.

Prior work in 2011 has shown that grease polymers are active in worked cone penetration.[1] A simple lithium grease (NLGI #1.5) demonstrated a +8.1% increase in cone penetration with 10,000 strokes ASTM D217. Addition of various olefin copolymers for grease reduced the change to +0.6% to +3.0% and therefore improved mechanical stability. Grease polymers were used in both solid (100% polymer) and liquid (5-10wt% polymer in 100N oil) form.

However in other cases grease polymers have provided negligible or negative changes to mechanical stability. Inorganic greases like silica and clay were shown to lose both consistency and some measure of water resistance (D4049 spray-off and D1204 wash-out) with the addition of various polymers.[2] However, two of four polymers tested in clay grease did improve D4049 spray-off which is considered to be a test of mechanical stability as well as static water resistance.

The question remains how do polymers interact with grease and why are certain greases so selective toward different polymer chemistries.

Grease Thickener Structures and Networks

Grease chemistry is highly varied but all thickener chemistries achieve the same goal, an immobilised network structure. The individual chemistry then defines the types of forces holding the network together, how those forces create a structure, and what unique properties emerge from that structure (texture, thermal/mechanical/water stabilities, etc.)
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Figures 1, **2**, and **3** demonstrate the hierarchy of structure from single molecules to grease thickener network.

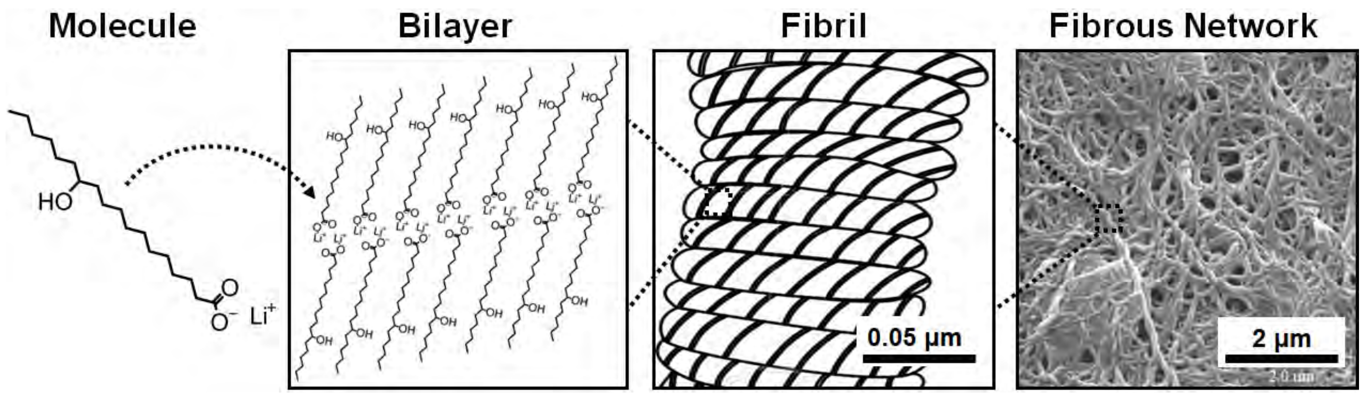


Figure 1. Fibrous grease structure (simple lithium, lithium complex). Bilayer based on [3][4][5]; microscopy from Kay[6].

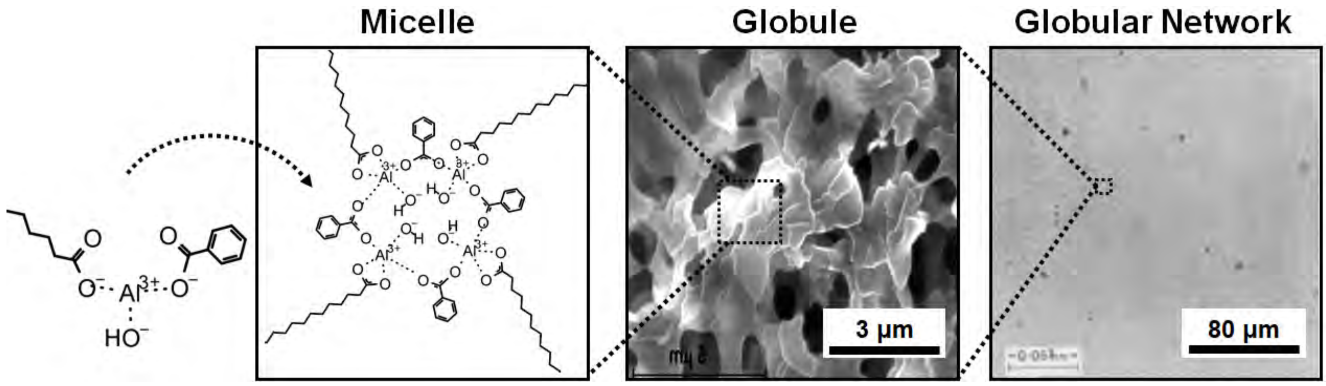


Figure 2. Globular greases structure (calcium sulfonate, aluminium complex). Micelle based on [7][8][9]; microscopy from aluminium stearates from [10][11].

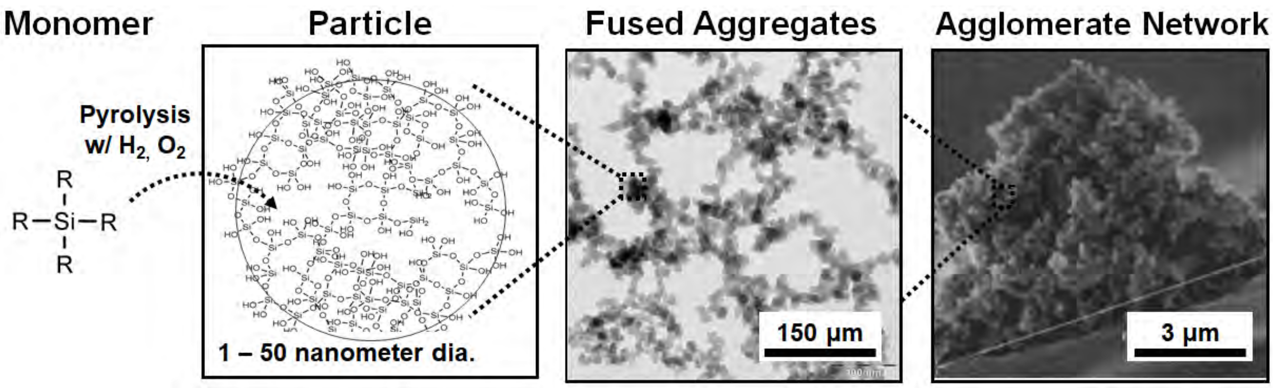


Figure 3. Particulate greases structure (silica, clay). Structures and microscopy from [12][13].

Grease Polymer Networks and "IPN" Theory

Polymers that are effective in improving the properties of grease at large ("grease polymers") typically form rigid networks much like soaps in petroleum oil. This structuring can occur inside a grease soap network and form an "interpenetrating network" or IPN. **Figure 4** demonstrates the IPN formation process between grease soap (orange) and polymer (blue). VI improvers for lube oil are typically highly amorphous and often make poor grease polymers.

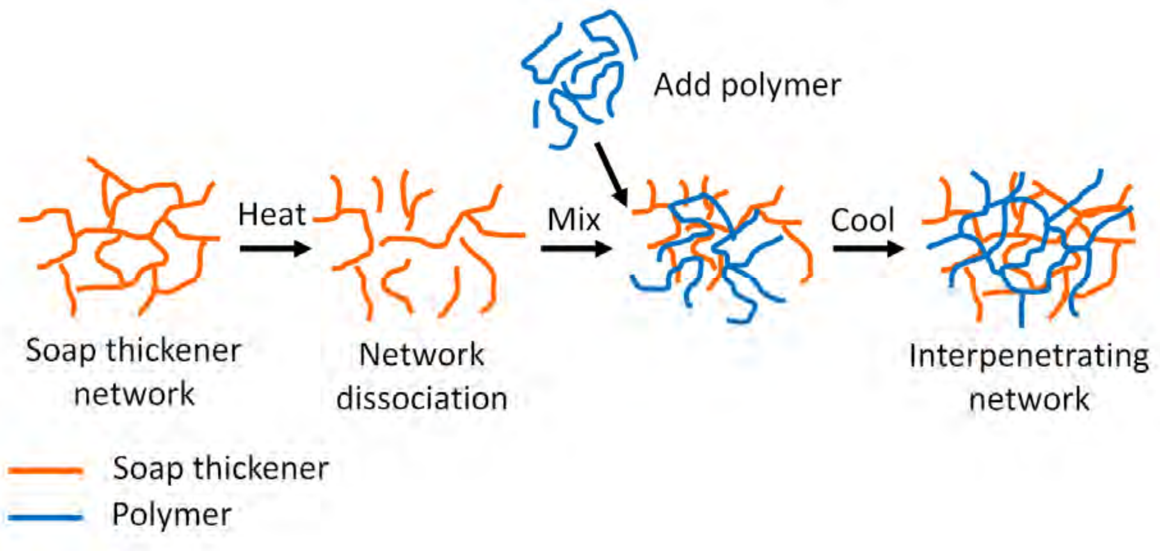


Figure 4: Formation of the interpenetrating network of polymer in grease

To be effective, the grease polymer must achieve a stable and rigid polymer network through one of three common mechanisms shown in **Figure 5** [1]:

1. **Temperature Sensitive** – long uninterrupted runs of one monomer (ethylene, styrene, etc.) along the backbone of the polymer form waxy crystallites between chains

2. **Hydrogen Bonding** – polar nitrogen and oxygen sites on the polymer separate from the non-polar oil and associate together in an acid-base attraction

3. **Reactive** – acid anhydride sites on the polymer react with metal ions to form insoluble coordination complexes (like diacids) or react with –OH sites to form covalent bonds in the thickener (like boron esters)

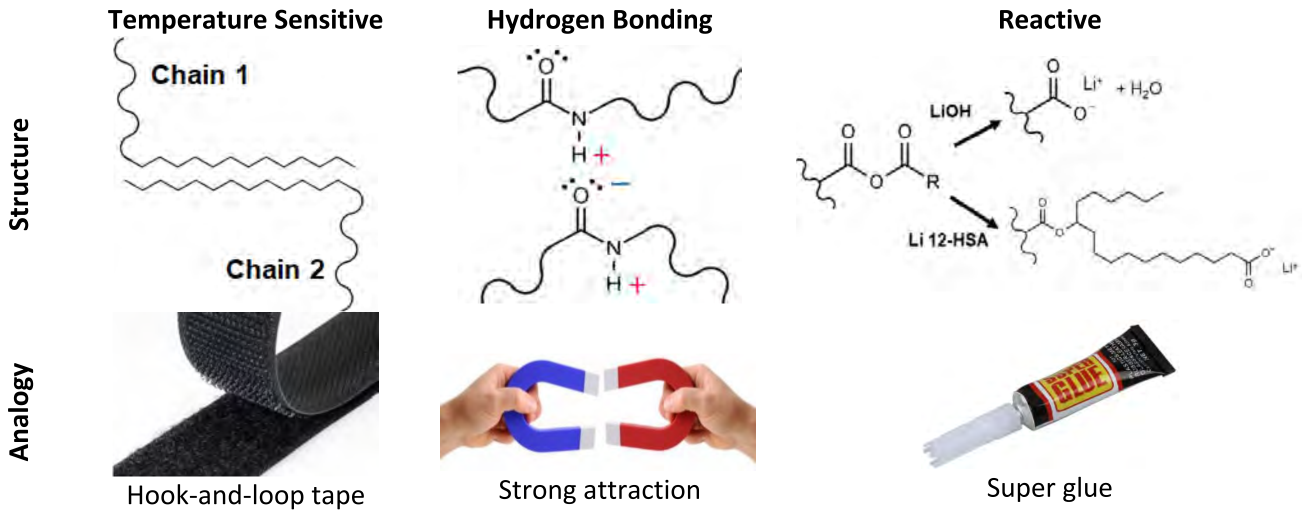


Figure 5: Comparison of the three main mechanisms for IPN formation by grease polymers and an analogy to help remember how each one works

While formation of the IPN network of grease and polymer is generally understood to be beneficial, there are many cases where networks can be inhibited by interactions with other networks or additives. It is well-known in the field that combining two or more grease types can cause a failure of the resulting blend.[14] In this case two fully formed networks combine to form something less than the sum of their parts. Pour point depressants are polymers with long chain waxy segments that actively prevent the formation of orderly wax networks, or gel, in low temperature lubricants.

Whether or not the IPN strategy works between any given grease thickener and grease polymer has yet to be explored in detail. This study will provide an array of different grease/polymer combinations and rationalise qualitative trends in the findings.

The goal of this work is to demonstrate, in the case of a handful of sample greases, that careful grease polymer selection matters and not to definitively show which grease polymers should be used with each grease thickener. A formulator's success in modifying grease with polymer will depend on the formulation as a whole – including base oil, complexing agents, additive packages, and production methods.

2. Experimental

2.1. Materials

<u>Base Greases</u>

Table 1 compares the six base greases from NLGI grade 0 to 3 which were obtained with different thickener chemistries from manufacturers "A" – "E" utilised in this study.

Thickener / Name	Cone Pene. (D217)	NLGI Grade (D1403)	Base Oil (D445, D2270)	Dropping Point (D2265)	Roll Stability (D1831)	Manu.
Simple Lithium	272	2	ISO 150 VI 98	391F / 199C	+1.2%	A
Lithium Complex	264	2.5	600N VI 113	518F / 270C	+36.1%	A
Calcium Sulfonate	242	3	ISO 100 VI 111	>752F / 400C	+3.1%	B
Aluminium Complex	253	2.5	ISO 150 VI 108	542F / 283C	+1.5%	C
Silica	309	1.5	600N VI 124	642F / 339C	+2.3%	D
Clay (Bentonite)	350	0.5	ISO 460 VI 101	>752F / 400C	+11.3%	E

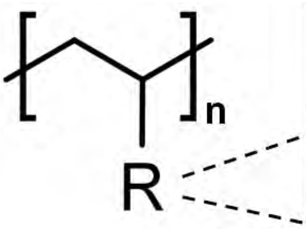
Table 1. Basic properties of the six base greases

Each grease was referenced in this study by the thickener chemistry. Base greases were formulated using typical performance additives for multi-purpose industrial grease minus any grease polymers. Base oil from each grease was obtained from oil bleed under mild conditions.[15] All six greases exhibited viscosities from ISO 100 to 460. Based on a VI range of 98 – 124, all greases were made with heavy paraffinic oils, likely Group II.

<u>Polymers</u>

A total of nine polymers were added individually to the six greases.

Two polymers each, a low MW (Mw 100K-200K) and high MW (Mw=300K-600K) version, were selected from each of the three grease polymer categories explained previously.[1] The exact composition of each grease polymer is proprietary but the typical functional groups are described in **Figure 6**. Performance of the polymer will vary based on the number and ratio of these monomers, the molecular weight, crystallinity, and other structural factors.



R =	Hydrogen H	Alkyl C_xH_y	Styrene	None C=C in backbone	Amide	Alcohol OH	Ester	Anhydride
Temp. Sens.	X	X	X					
H-Bonding	X			X	X	X	X	
Reactive	X	X	X				X	X

Figure 6: Typical functionality of the three grease polymer mechanisms

Additionally, two tackifiers of low MW (Mw 500K-1M, OCP) and high MW (Mw 4M-6M, PIB) were included due to their ability to form entangled networks in lube and grease. A dispersant polymethacrylate was also included due to its high hydrogen bonding capability and to cover recent interest in PMA in grease.

2.2. Methods

Polymer Solution Preparation

Polymers were pre-dissolved at 8-10wt% in 100N Group II oil to allow consistent and high-throughput blending of different polymers into the base greases. Grease polymers were solubilised for 24 hours at 100-120°C and filtered at 150 micron before adding hot to grease. 0.1wt% BHT was included.

Polymer Modified Grease Preparation

Base greases were top-treated with 5 parts polymer solution to 95 parts grease and mixed on low speed at 80-100°C for 2 hours. Base greases were milled during initial production and not milled again after adding liquid form polymer. Greases were left to rest at 20-25°C for 24 hours before testing.

Consistency Measurement and Cone Penetration

All cone penetrations reported in this study are 60 double-stroke worked penetrations converted from quarter-scale measurement and reported in mm/10 (ASTM D1403; Precision Scientific Co. #D-5). NLGI grade was assigned by ASTM D217 with half grades: “0.5”, “1.5”, etc. Extra care was taken to remove voids in tacky samples before testing.

ASTM D1831 Roll Stability

ASTM D1381 roll stability was used to quantify mechanical stability at an outside ISO 17025-2017 laboratory. Standard conditions (2hrs, 25°C) with ¼ cone penetrations were used. Roll stability was reported as the percent change in initial consistency vs. sheared consistency. A positive percentage (“+1%”) means penetration increased and the grease has softened, i.e. from 300 to 303. Negative (“-1%”) means penetration decreased and grease thickened.

3. Results and Discussion

3.1 Changes in Grease Consistency with Polymer

Before evaluating changes to grease’s consistency with mechanical shearing, it is important to understand the contribution an added grease polymer can make to the grease’s consistency pre-shearing. A basic grease uses heavy oil and thickener to achieve the desired NLGI grade. Thickeners can be used at up to 30wt% of some grease, may be costly, and have a supply chain in competition with other markets (e.g. electric vehicles).[16] Polymers are routinely used to increase the consistency of grease and reduce a portion of the need for thickener.

A comprehensive study was performed with low and high MW variants of six grease polymers, two tackifiers, and a dispersant PMA.

In all, 54 greases (6 base grease times 9 polymers) were prepared to screen for the influence of different greases and polymers in combination on the resulting NLGI grade. Samples where polymer improved the NLGI grade by ≥ 0.5 were later used in the roll stability study in the second half of this work.

Table 2 summarises the observed changes in NLGI grade with the addition of polymers versus simply adding the diluent oil without polymer (the control). Out of 54 samples, 21 (39%) experienced no change in NLGI grade after adding 5wt% liquid grease polymer versus the control of 5wt% pure 100N diluent oil. 28 (52%) observed a half (39%) or full (13%) increase in NLGI grade. 5 samples (9%) lost NLGI grade by a half (7%) or full (2%) grade. “Half” grades are denoted as consistencies that fall within the gap of cone penetrations between two grades (e.g. an NLGI #1.5 falls between the NLGI #1 and #2 ranges)

.

Grease Category:	Fibrous		Globular		Particulate	
	Simple Lithium	Lithium Complex	Calcium Sulfonate	Aluminium Complex	Silica	Bentonite Clay
Control	1.5	2	2.5	2.5	**1**	**0**
Temp. Sens. Polymer (Low MW)	**2**	**3**		2	**2**	
Temp. Sens. Polymer (High MW)	**2**	**2.5**				
H-Bonding Polymer (Low MW)		**2.5**		1.5		**0.5**
H-Bonding Polymer (High MW)	**2.5**	**2.5**	**3**	**3**	**2**	**1**
Reactive Polymer (Low MW)	**2**	**3**	**3**			**0.5**
Reactive Polymer (High MW)	**2**		2	**3**		**0.5**
Tackifier (Low MW)	**2**	**2.5**	**3**	2	**2**	
Tackifier (High MW)	**2**		**3**			**0.5**
Dispersant PMA (High MW)	**2**		2			

Analytics:
of +0.0 NLGI:
21 (39%)
of +0.5 NLGI:
21 (39%)
of +1.0 NLGI:
7 (13%)
of -0.5 NLGI:
4 (7%)
of -1.0 NLGI:
1 (2%)

Table 2: NLGI grade of the 54 modified greases (with 5wt% of 8-10% polymer in oil) vs. controls (with 5wt% 100N). Blank entries mean no change (+0.0) to NLGI grade. **Bold** entries indicate a gain (+0.5 or +1.0). Red, a loss (-0.5 to -1.0).

Fibrous grease like simple lithium and lithium complex show high compatibility with the various grease polymers. 14 out of 18 (78%) grease/polymer combinations produced an increase in NLGI grade which indicates the interpenetrating network formation was successful. Higher consistency gains appear to occur with lower molecular weight variants of each grease polymer type. Two large (+1.0) grade changes occurred between lithium complex and low MW temperature sensitive and reactive polymers.

Globular grease like calcium sulfonate and aluminium complex were highly variable in response to grease polymer. Nearly half (39%) of prepared samples showed no effect on NLGI grade. One third produced a +0.5 increase and the remainder (28%) gave consistency losses with one -1.0 grade. Both globular greases favoured higher MW additives: low and high MW tackifiers produced the best NLGI grade improvement in calcium sulfonate; aluminium complex gave NLGI grade improvement for high MW versions but lost grade with low MW versions of the same polymer type.

Negative changes to NLGI grade were not anticipated at the beginning of this study. Coincidentally globular structure greases, especially aluminium, that exhibited the NLGI grade loss have long been known to form "false body".[17][18]

Over time a grease during production and storage can increase in consistency over time as it ages. When the grease is sheared this extra consistency (false body) is quickly lost.

On negative results for calcium and aluminium greases, false body may have caused the control to be higher NLGI grade but the addition of polymer inhibited false body leading to a lower grade with polymer. The net result would be a negative change in NLGI grade with polymer.

Particulate grease, silica and bentonite clay, produced either no change in NLGI grade with polymer or a mix of low and high NLGI grade increase. Modified silica grease results were dramatic with either a full NLGI grade improvement or no effect. The three cases where silica grease was improved occurred with additives that were subjectively adhesive in handling quality and OCP-based.

Bentonite clay exhibited high compatibility and consistency gains primarily with polymers containing polar functionality, both hydrogen bonding and the polar-grafted reactive types. This result was reasonable due to the high surface functionality of –OH on clay which requires the use of hydrogen bonding dispersants to incorporate the clay into oil. Silicas, in contrast, tend to have lower surface functionality and less hydrogen bonding ability if produced from $SiCl_4$ at very high temperature as fumed silica rather than precipitated silica. This may explain the large difference in response to polymer between the two particulate greases.

3.2 Changes in roll stability with polymer

Grease and polymer combinations that produced a notable increase in consistency ($\geq$ +0.5 NLGI grade) were considered to possess a strong interaction between the grease and polymer indicative of a stable interpenetrating network (IPN). Stable IPN formation has long been hypothesised to be the driving mechanism for most benefits to polymer in grease.

ASTM D1831 roll stability was used to evaluate the relationship between greases with strong IPN formation (showing high consistency increase) versus their shear stability. D1831 with ¼-cone measurements has an 11 point reproducibility; this gives a relative error of ±3.8% based on an average 290 cone penetration.

Twenty-seven polymer-modified grease samples were tested and compared against the roll stability changes of the control greases. **Table 3** summarises the changes to consistency after ASTM D1831 roll stability with positive values meaning a loss of consistency (thinning) and negative values meaning an increasing in consistency (thickening). Samples within the 3.8% relative error of ASTM D1831 were deemed negligibly different.

Grease Category:	Fibrous		Globular		Particulate	
	Simple Lithium	Lithium Complex	Calcium Sulfonate	Aluminium Complex	Silica	Bentonite Clay
Control	+1.2%	+36.1%	+3.1%	+1.5%	+2.3%	+11.3%
Temp. Sens. Polymer (Low MW)		**+21.5%**			*+2.6%*	
Temp. Sens. Polymer (High MW)	*+1.5%*	**+19.8%**				
H-Bonding Polymer (Low MW)		*+37.9%*				*+11.5%*
H-Bonding Polymer (High MW)	*+1.5%*	**+25.6%**	+11.7%	+18.9%	+7.9%	*+9.4%*
Reactive Polymer (Low MW)	*-2.6%*	**+22.5%**	+9.8%	**-5.2%**		
Reactive Polymer (High MW)	*+0.0%*			+14.5%		*+12.5%*
Tackifier (Low MW)		**+17.8%**	*+4.6%*		*+2.6%*	
Tackifier (High MW)	*-2.1%*		*+1.6%*			*+13.8%*
Dispersant PMA (High MW)	+6.6%					*+8.6%*

Analytics:

Same
15 (56%)
More Stable
6 (22%)
Less Stable
6 (22%)

Table 3: ASTM D1831 roll stability changes in polymer modified greases. Blank entries were not tested. *Italic entries* indicate no significant change versus control (±3.8). **Bold** entries indicate improved roll stability (lower % change). Red entries indicate worse roll stability (higher % change).

Of the 27 samples, 15 samples (56%) demonstrated negligible changes, within error, to roll stability versus the control. The implication, however, is quite significant. Simple lithium and clay greases were improved by up to a full NLGI grade using polymer without an effect on shear stability. By reducing thickener to account for the polymers' effects, 0.45wt% polymer could replace up to 3.5wt% lithium 12-hydroxystearate preformed thickener or bentonite clay.[19][20]

6 samples (22%) showed an improvement in shear stability, the majority being lithium complex. **Figure 7** shows the highly structured nature of fibrous grease which can be destroyed during mechanical shearing. Lithium complex grease demonstrated high compatibility with most of the polymers tested showing both high NLGI grade improvement and reduced shear losses. These fibres are help together through weak, short-range ionic and hydrogen bonds and waxy interactions. Polymers diffused throughout this network provide strong covalent bonds over long-range to provide strength.

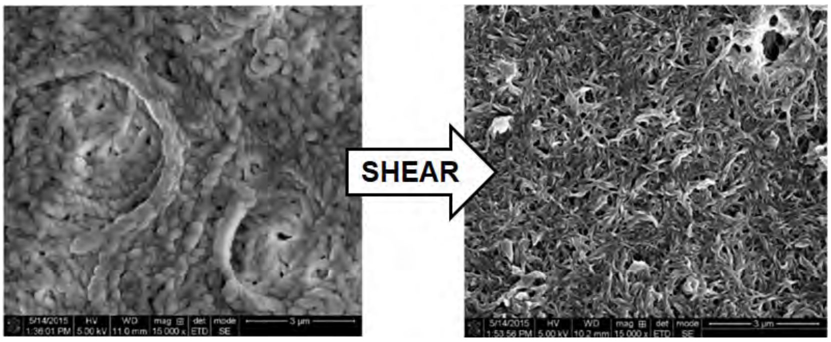


Figure 7: Damage to a fibrous grease structure; image from [21]

6 samples (22%) showed a loss of shear stability with the use of polymer. Four of six polymers that caused worse shear stability were high molecular weight hydrogen bonding polymers including the dispersant PMA. The majority of cases occurred with globular greases which tended to be highly 'selective' toward polymer – only a few polymers improved NLGI grade and qualified for roll stability testing. These globular greases tend to be held together by waxy interactions of long chain alkyl groups along the exterior of the micelles. It may be that large polymers with hydrogen bonding capability may be interrupting the structure of the micelles in order to hydrogen bond with the hydrophilic cores of these structures. Reactive polymers also gave poor shear in the globular greases. These reactive additives operate by interacting with and grafting to the metal centres contained in the metal. This could inhibit the formation of stable micelles which make up the globular grease structure.

Oddly, three samples exhibited negative roll stabilities, i.e. showed higher consistency after shear than before shear. Two of the samples occurred with the low molecular weight reactive grease polymer, a negligible result in simple lithium and a significant result (>3.8 difference) in aluminium complex. There may be many explanations or competing effects that lead a grease to become tougher after shearing. Since the common element was the reactive polymer it is hypothesised that the reactive polymer generated a weak but fast-forming kinetic product which rearranged to a more stable thermodynamic product with the aid of aggressive milling in the ASTM D1831 experiment.[22] For example, the reactive anhydride group on the polymer can either coordinate a metal ion to form ionic metal carboxylate salts (like a diacid in complex grease) or chemically bond with hydroxyls on the thickener molecule to form a new ester linkage (like a boron ester used to enhance dropping point in grease). The proposed idea is that the energy of mechanical shearing could either promote hydrolysis of that ester bond to a metal carboxylate or promote the formation of the ester from the carboxylate depending on which is more chemically stable long term.

3.3 Overall comments on findings

The stark contrast between the effects of polymer on NLGI grade and yield of lithium versus non-lithium grease demonstrates the need to include a variety of grease types in additive research. However, lithium continues to remain a majority (>70%) of the market and subsequently the focus of much R&D.[23] This work specifically includes six grease types to expand the body of work available on polymer/grease interactions. Lithium greases behaved as expected because most expectations for grease polymer performance are made using lithium grease. The results in globular and particulate greases were far more nuanced.

Prior to this study there was much data to support that polymers are generally good for improving shear stability. However, the results above show that only 22% of 27 cases tested resulted in significant improvement and the remaining 78% showed either a loss of stability or no change. Yet only 22% of the cases showed a defined loss of shear stability (increasing roll stability changes) from adding polymer. This signifies that the majority of times polymer is used for water resistance, tack, adhesion, oil bleed control, or added yield will not come at the expense of mechanical stability. These losses occurred primarily for a specific group of grease polymers (hydrogen bonding type) in a specific category of grease (globular structure like Ca and Al) which may now be better anticipated at the beginning of a formulation project due to these findings.

With regards to yield, many cases (52%) demonstrated that grease polymers can increase NLGI grade which ultimately means improved yields and economics after replacing more thickener with oil. A small number of cases showed NLGI grade loss which may be from reducing occurrence of false body. Cases of no NLGI grade change may actually be an opportunity to add that polymer for a different advantage without having to reformulate the thickener and oil content.

4. Conclusions

Grease is a highly complex material where simple changes to composition or process can have complicated results on performance. Tools like the interpenetrating network concept provide high level strategies for narrowing right polymer for a grease but much empirical work in optimisation remains. Thus, grease remains both an art and a science.

This study elaborates on the interpenetrating network model of grease/polymer interactions and importantly considers a wide variety of grease types. It is shown here that not all grease polymers form IPN in all greases and the best option will vary for each grease. Even when an IPN did form (suggested by increases in NLGI grade) not all the proposed benefits of an IPN were obtained (lack of shear stability improvement).

The results of this study are intended as a starting point for formulators and not a definitive guide. Most greases, even of the same kind, will vary widely by factors like the specific thickener used, the base oil, NLGI grade, and production.

Acknowledgements

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Maintaining safe and quiet railways with "top of rail materials"

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Marc has over 10 years of experience working in Tribology. He has a master's degree in Chemistry and a PhD in Engineering (Tribology). He has had a successful career at Imperial College, PCS Instruments and Afton Chemical. Marc has extensive knowledge of all tribology test methods including both standardised and non-standardised lubricant tests. He has designed, modified and improved countless methods, to ultimately aid in the development of new lubricant products. Marc now works for his own company Ingram Tribology.

Key Words: Friction, top of rail, flange, creep curves, railway

Abstract

Lubricants are used on the rail/wheel interface to control friction, reduce wear and fatigue along with reducing noise. As a train proceeds around a curve its wheels (which are fixed) travel different distances and thus some sliding between the wheel and the rail is inevitable. This sliding at the wheel /rail interface can cause an unpleasant high pitched noise. The noise is generated by an unsteady dynamic where the sliding wheel alternates rapidly between two sliding speeds. This unsteady dynamic can be suppressed and controlled using lubricants which are added directly onto the rail or wheel. It has been found that to reduce noise, the friction between the wheel and rail should increase as the percentage of slip (creep) increases. This friction characteristics can be evaluated using "creep curves" in controlled test machines. A benchtop test method has been developed which can generate creep curves under realistic conditions of speed and contact pressure. This method was found to differentiate 11 different railway products. An inter-laboratory study/ round robin was then conducted using this new method. We report on the results of the round robin with a statistical analysis of repeatability and reproducibility.

Introduction

Lubricants are used on the rail tracks for numerous reasons including: ensuring safety, reducing wear and noise and improving fuel economy. Lubricants can be found on two sections of the wheel/rail interface (Figure 1). Top of rail (TOR) materials are used on the wheel tread/ rail crown area, whilst flange products are used on the wheel flange face/rail gauge shoulder interface. Flange products have traditionally been used on railways to control wear at the wheel/rail interface. Whereas the use of "top of rail materials" (friction modifiers) has become prevalent recently.

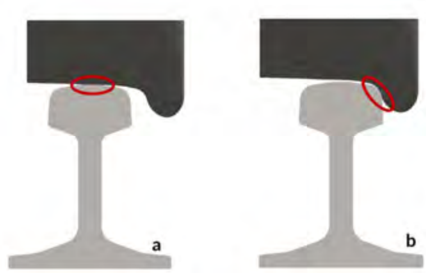

Figure 1 – Cross section of a wheel and rail, showing the contact between (a) the wheel tread and rail crown and (b) the wheel flange face and rail gauge shoulder

Flange products

Flange products are similar in composition and appearance to traditional greases. The flange products evaluated here are those applied to the rail via trackside applicators or trainborne equipment. These can be petroleum based with mineral or synthetic base oils. They can also be based on biodegradable oils for environmentally sensitive areas.

TOR Materials

The formulation of TOR materials is considered confidential by their manufacturers so exact compositions are not available in the public domain. Water based TOR materials are most common in Europe, these contain:

- Water
- Glycols
- Thickener/stabilisers (e.g. clays, PVA styrene copolymers, latex and silica)
- Solids (e.g. graphite, calcium carbonate and metal powders)

Some TOR materials are designed to work with a carrier system, which evaporates once the material has been deposited leaving a thin film of the active friction controlling substance. Oil based products are common in America, where axle loads are much higher. Oil based TOR Materials have a lower friction coefficient and generally not used in Europe.

Dual/hybrid systems

Dual/hybrid products aim to do the job of both the TOR materials and the flange product, having acceptable performance on both the rail crown and rail gauge shoulder.

Wheel/rail traction

Traction between railway lines and train wheels is paramount for the safe and effective operation of trains. High traction is required to allow the controlled acceleration and braking of the train.

The level of traction available for the safe operation of the train can be affected by environmental conditions (rain water, falling leaves) and also any material present at the interface.

In the railway industry it is customary to describe the friction forces in the wheel/rail interface as adhesion. This "adhesion" term is synonymous with the term friction coefficient or traction coefficient. Adhesion can be used to define the traction available to transfer tangential forces between a driving train wheel and the rail. If the driven wheel applies a tangential force larger than the limit (defined by the traction coefficient) the wheel will spin causing severe damage to the rail. Hence the need to maintain high traction at the wheel tread/rail crown interface, for both performance and safety of the train, as well as the longevity of the wheels and the rails.

TOR materials act to condition the rail to providing a consistent friction/ traction value, while reducing both micro slip and sliding. A controlled level of traction is preferred, within a range of 0.25 to 0.4 traction coefficient [1]. If the traction is too high, it can increase the wear and fatigue processes on the wheel and rails. If the traction coefficient is too low, excess slips and slides can occur leading to wear and uneconomical operation of the train and potentially give braking problems.

For the wheel flange/rail contact a friction coefficient of below 0.1 is desirable [1]. This is achieved with flange lubricating products, which are known to reduce noise and wear at this interface.

The friction coefficient of a "dry" rail has been measured and is found to be between 0.4-0.6 [2]. At this level short pitch corrugations are found and sometimes referred to as roaring rails [3]. Very high friction forces can be achieved with the use of sand at the wheel/rail interface, but with the accelerated demise of the wheels and rails. This method can be used for example in areas with heavy leaf fall, which is known to reduce friction at the wheel rail interface [4].

Railway noise

An unpleasant high pitch noise can be generated from the wheel/rail interface as the train proceeds around curves. As the train moves through a curve the wheel pairs (which are fixed) travel different distances, and thus some sliding between the wheels and rails is inevitable. It is believed that the high-pitched squeal noise (in the region of 200 – 2000Hz) occurs at the leading inner wheels due to a lateral slipping at its natural mode [5]. When the friction characteristics of the wheel/rail interface allow it, the wheels can enter this unsteady dynamic. The unsteady dynamic is due to the wheels alternating between two sliding speeds, generating vibration. The vibration causes oscillation at the wheel web [6].

The noise can be reduced by controlling the friction characteristics at the wheel rail-interface. To reduce noise the friction at the wheel-rail interface should increase with the percentage creep [5]. Creep is defined as the percentage of sliding at the rail wheel interface relative to the speed of the train. Defined here as:

$$Creep\ (\%) = \frac{V_S}{V_T} \times 100$$

where V_S is the sliding speed between the rail and wheel (the slip) and V_T is the speed of the train.

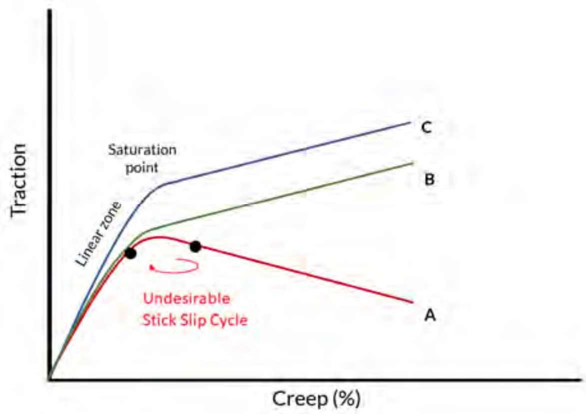

Figure 2: Representation of an idealised creep curve. Curve "A" would indicate noise generation in the field. "B" with a positive slope would suppress noise. Curve "C" with high overall traction and a positive slope is the ideal system.

A representation of a creep curve is shown in Figure 2. At lower levels of creep the traction increases linearly. This linear zone is due to more and more of the contact become sliding as opposed to rolling. At a certain percentage creep the contact becomes fully sliding, sometimes referred to as "saturated". The traction forces are then dependent on the metal surfaces and any third body material. Three possible scenarios are shown in Figure 2:

- Curve "A" shows a traction force falling with increasing creep. Where the creep curve has this negative gradient, the wheels can enter an unsteady dynamic, where the speeds can quickly alternate between the two points of same traction either side of the saturation point. This produces a highly undesirable stick slip cycle, which will lead to the development of vibration and ultimately noise.
- Curve "B" will suppress the noise, due to the removal of the negative damping effect of the system depicted in Curve "A".
- Curve "C" depicts the ideal system for a TOR material with high overall traction and a positive slope after the saturation point.

Despite the widespread use of TOR materials and flange products they are not yet covered by any international standards, although their use is accepted and practiced by many railway operators around the world.

The aim of this work is to develop a test method for TOR material and flange products to ensure performance in the field. This method would then be added DIN EN (possibly ISO) Standard "Railway applications – Wheel/rail friction management – Lubricants for trainborne and trackside applications" (EN 15427)
As all the railway lubricants and high traction products used in this study are approved for use in the field in Europe, the friction characteristics of these products can provide a benchmark on which to compare other formulations.

This paper details the development of the test method, followed by a inter laboratory study to evaluate its reproducibility.

Method development

A standard PCS Instruments Mini Traction Machine (MTM) was used to develop this new test method. The MTM uses a ¾ inch ball, loaded against a 46 mm diameter disk. Both ball and disk are manufactured from AISI 52100 steel and have a surface finish of approximately 10 nm Ra. The hardness of the ball and disk is 760 HV. The loads of between 5 and 75 N on the instrument allow the application of a contact pressure between 0.5 and 1.25 GPa. Both the ball and the disk are independently driven using accurate DC motors and encoders, allowing the application of controlled slippage at the contact. The slippage is defined with a slide/roll ratio. This definition is similar but not exactly comparable to the "creep", more commonly used on railways:

$$SRR\ (\%) = \frac{U_S}{U_E} \times 100$$

Where, $U_S$is the sliding speed and U_E is the entrainment speed given by:

$$U_E = \frac{[U_A - U_B]}{2}$$

Where U_A and U_B are the linear speeds of the ball and the disk.
Thus in sliding/rolling tribometers such as that used in this study the slide/roll ratio is slightly lower than the equivalent quoted "creep" values from the railway industry.

Both steel specimens were cleaned prior to testing to remove any corrosion inhibitor product from their surface. The cleaning procedure consisted of 20 minutes immersion in Heptane (analytical grade) with ultrasonic excitation. Followed by 20 minutes immersion in propan-2-ol (analytical grade) with ultrasonic excitation.

Figure 3 shows a photograph of the ball and disk test specimens attached to the MTM, before the application of a TOR material or flange product. A small quantity of TOR material or flange product is applied to the top of the disk using a clean mask. The quantity of material is higher than would be typically applied on the rails in the field, but as this is a non-conformal contact, much of the product is pushed from the contact zone. Only a very small quantity of TOR material or flange product remains in the running track during the test.

Figure 3: Photograph of the ball and disk attached to the MTM

An applicator mask was developed to help apply a controlled volume of material on the disk. The applicator contains 12 straight holes of 2.5 mm diameter and 1 mm depth. Thus each hole has a volume of 4.91 mm^3 and the mask will help deposit a nominal 58.9 mm^3 or 0.0589 ml around the running track of the disk in total. The semisolid substance is wiped over the mask, ensuring all the holes are filled. The substance is then made flush with the top of the mask with a flat spatula. This deposits 12 small dimples of material on the disk when removing the mask. This procedure is shown stepwise in Figure 4.

Figure 4: Photographs showing the step-by-step application of semisolid product onto the surface of a MTM disk.

Once the product has been applied to the disk, the disk and the ball are added to the MTM instrument as normal. The ball is loaded against the disk at 30 N. The temperature of the pot is not controlled. A run-in is used to spread the material evenly over the test specimens and condition the steel specimens. A speed of 100 mm/s and SRR of 50 % is used for the run-in for 30 minutes in total. At 15 minutes the speed is increased to 4000 mm/s and sliding stopped for 1 minute. This high-speed step is used to “fling” any loose material from the steel samples, to prevent any excess material being re-introduced into the contact during the high-speed traction curves, which occur later in the test. This step was found to be beneficial for repeatable creep curves in early experiments.

Creep curves are generated at two entrainment speeds, 1 m/s and 3.8 m/s. These correspond to train speeds of 3.6 km/h and 13 km/h respectively. Traction measurements are taken at slide/roll ratio values of 0.25, 0.5, 0.75, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 %. Table 1 shows the speeds of the samples for each point of the creep curves at 1 m/s. Traction forces are measured in both directions and their average reported.

Table 7: Speeds of the ball and disk during the measurement of friction

Entrainment Speed (m/s)	SRR (%)	Disk speed 1	Ball speed 1	Disk speed 2	Ball speed 2
1	0.25	0.99875	1.00125	1.00125	0.99875
1	0.5	0.9975	1.0025	1.0025	0.9975
1	0.75	0.99625	1.00375	1.00375	0.99625
1	1	0.995	1.005	1.005	0.995
1	2	0.99	1.01	1.01	0.99
1	3	0.985	1.015	1.015	0.985
1	4	0.98	1.02	1.02	0.98
1	5	0.975	1.025	1.025	0.975
1	6	0.97	1.03	1.03	0.97
1	7	0.965	1.035	1.035	0.965
1	8	0.96	1.04	1.04	0.96
1	9	0.955	1.045	1.045	0.955
1	10	0.95	1.05	1.05	0.95

The test proceeds alternating between a measurement creep curve step and a 2 minute pure rolling step. This allows the steel samples to cool between each measurement.

Table 2 shows the test profile used on the MTM. All steps were carried out at 30 N load (~1 GPa contact pressure) and ambient temperature.

Table 8: Test conditions used on the MTM instrument

Step No.	Description	Speed (mm/s)	SRR (%)	Duration (min)
1	Run-in	100	50	15
2	Run-in	4000	0	1
3	Run-in	100	50	15
4	Creep curve medium speed	1000	0.25 - 10%	~ 1
5	Pause	50	0	2
6	Creep curve high speed	3800	0.25 - 10%	~ 1
7	Pause	50	0	2
8	Creep curve medium speed	1000	0.25 - 10%	~ 1
9	Pause	50	0	2
10	Creep curve high speed	3800	0.25 - 10%	~ 1
11	Pause	50	0	2
12	Creep curve medium speed	1000	0.25 - 10%	~ 1
13	Pause	50	0	2
14	Creep curve high speed	3800	0.25 - 10%	~ 1
15	Pause	50	0	2
16	Creep curve medium speed	1000	0.25 - 10%	~ 1
17	Pause	50	0	2
18	Creep curve high speed	3800	0.25 - 10%	~ 1

At the end of the test the disks were cleaned using the same procedure as the fresh specimens. The wear tracks are then analysed with a metallurgical grade microscope – (Brunel SP400 metallurgical). The width of the wear track is measured digitally using ImageJ software. The software is calibrated for each magnification using a calibration slide with increments of 0.01 mm, to relate each pixel on the camera to a length value.

Eleven materials were supplied by the ELGI Railway working group. The designation provided is noted here for reference in Table 3. The manufacturer or the type of material was not known during the development of the test method.

Table 9: Top of Rail Material Codes - as supplied

Sample	Lubricants Designation	Application
A	18-03125	TOR Material
B	18-03126	Flange
C	18-03192	Flange
D	18-03127	Flange
E	18-03128	Flange
F	18-03129	Dual product
G	18-03130	TOR Material
H	18-03131	TOR Material
J	18-03132	TOR Material
K	13-03133	Flange
L	Supplied by ProRail	Dual

Data processing

For each test, eight creep curves are generated. Four at medium speed (1 m/s) and four at high speed (3.8 m/s). The first two creep curves (steps 4 and 6) and the last two creep curves (steps 16 and 18) were not used in the analysis. Although care was taken to run the samples in and ensure the TOR material is distributed evenly across the disk, the first creep curves sometimes showed variation. This could be due to the material still settling. At the final creep curves the samples may begin to wear and heat up, causing variation. The traction curves from step 8 to 14 were used in the analysis. Each material was evaluated at least twice using this method.

Results

The results of the creep curves at 1 and 3.8 m/s are shown in Figures 5 and 6 respectively for all the materials.
Most of the creep curves show a positive gradient, this might be expected as all these products are known to be approved for use in the field and are expected to have a good performance. The test method can also distinguish between the overall friction range of the different products groups, with TOR Materials demonstrating the highest overall friction, flange products the lowest and dual products middling. Sample A, J and G show creep curves with a greater gradient that the other samples. Sample H showed the highest overall traction throughout all creep values. The samples mostly show similar friction characteristics at 1 and 3.8 m/s. Although some, such as Sample A and B show higher overall friction characteristics at the higher speed.

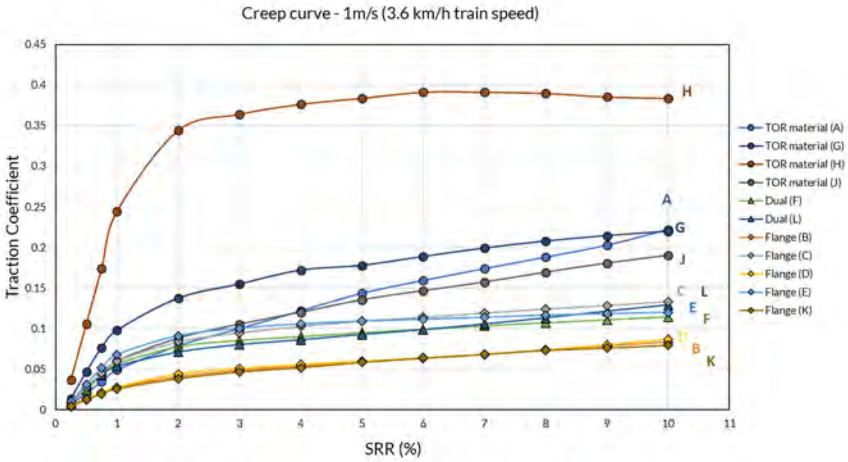

Figure 5: MTM Creep curves at 1 m/s for materials A through L

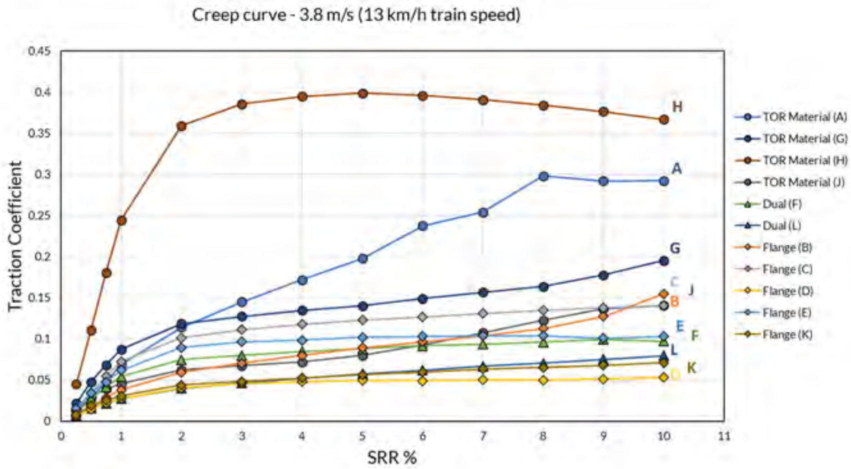

Figure 6: MTM Creep curves at 3.8 m/s for materials A through L

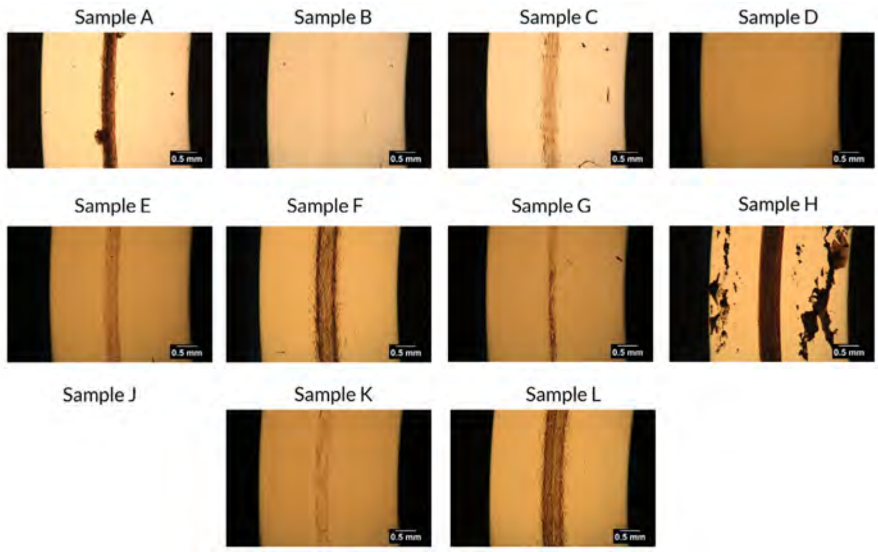

Figure 7: Micrographs of the disk surface after the MTM tests for all 11 samples

Images of the disk surface after the tests are given in Figure 7 for all 11 samples. Showing a large variation in the nature of the deposited material of wear to the metal surface. Sample B and D show a barely visible wear scar, whereas samples A, F, H and L have deposited solid particles on the disk surface. Sample G has deposited a thick, viscous residue. The wear track width was measured digitally using the photographs from the microscope. The results are given in Figure 8.

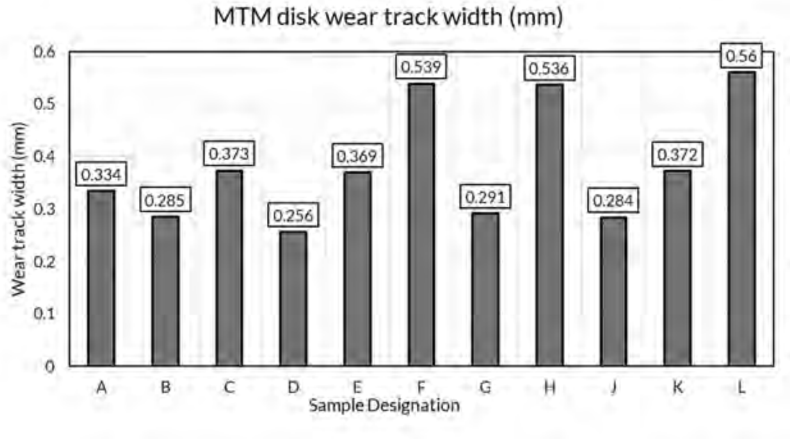

Figure 8: Wear scar width as measured on the MTM disc after the test

The variation in the wear track width at the end of the test show the formulation style used to achieve TOR materials with Sample J and G having significantly lower wear compared to sample H.

Test Method development summary

The MTM creep curves method is shown to measure the friction characteristics of high-pressure steel contacts, such as those found within rail/wheel interface. This method was found to differentiate the performance of TOR materials, flange products and dual products, to an extent expected by railway engineers. This method was adopted by the ELGI railway working group and a inter laboratory study was initiated – detailed below.

Inter laboratory study (Round Robin)

An inter laboratory study (ILS)/ Round robin was set up by the members of the ELGI Railway working group in 2019. 12 laboratories volunteered to participate in the ILS. The participants are to remain anonymous, but it can be said that the ILS was conducted by a group of international oil companies, lubricant additive manufacturers, instrument manufacturers and test houses.

Four test samples were selected for the ILS and are detailed in Table 4.

Table 10: Details of the four materials used during the ILS

Sample A	TOR material
Sample D	Flange product
Sample F	Dual product
Sample H	TOR material

The test samples were provided to each participant along with a reference oil and the grease applicator. The reference oil was used to check the performance of each MTM instrument before starting the testing of the railway products. If this qualifying test was satisfactory, the participants were asked to conduct 3 tests on each railway sample, following a defined method. The test method was changed slightly for the ILS, with the applied load being reduced from 30 N to 20 N. This was to reduce the chance of the instruments exceeding their maximum force and tripping out or causing damage.

Results – ILS

The creep curves generated on the four samples during the ILS are shown in Figures 9 and 10. Eleven of the twelve labs submitted data back to the group. This gives the entire spread of the results. Where the mean of the results is given for the entire population (every test from every lab). The error bars denote the spread of the data (showing the highest and lowest recorded result)

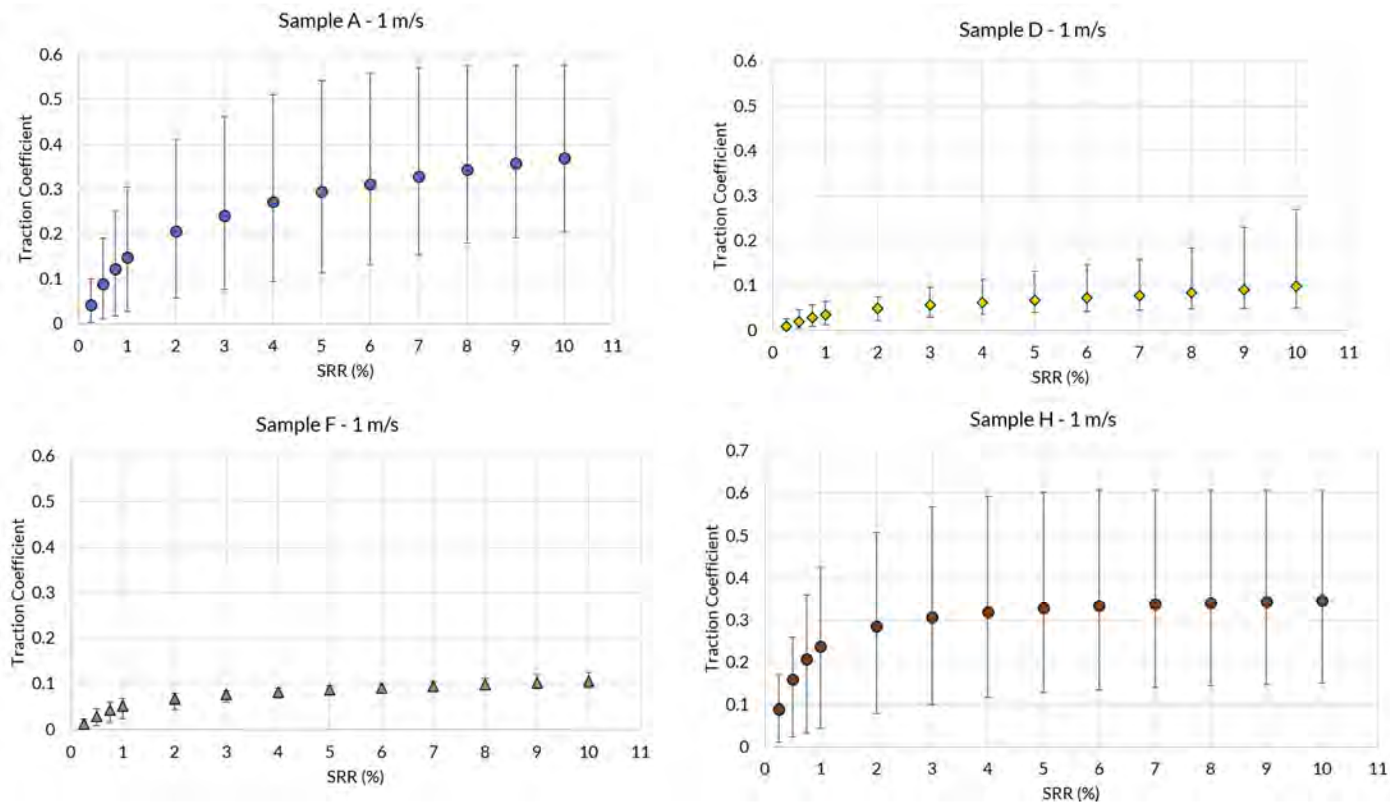

Figure 9: Raw data from the ILS – medium speed creep curves for each material

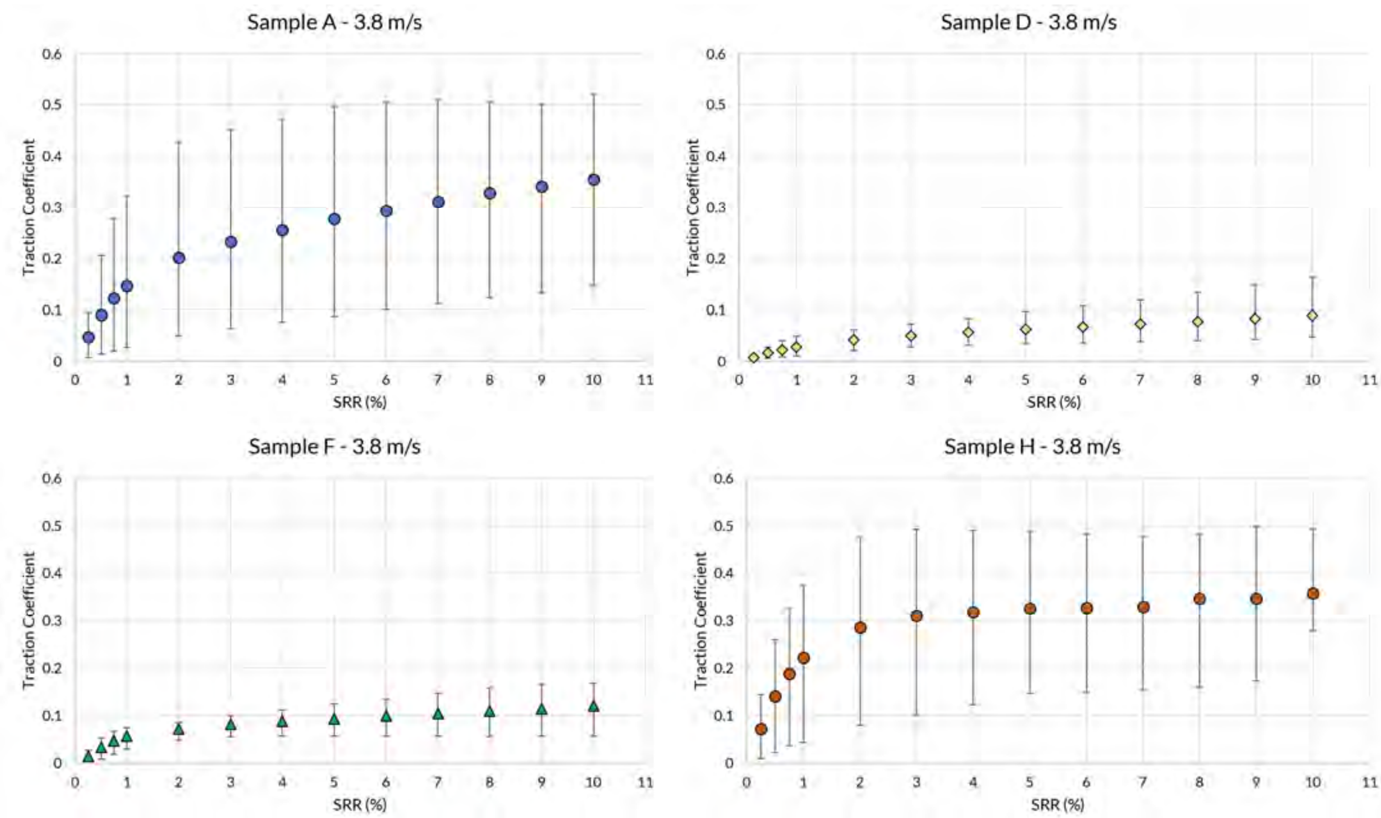

Figure 10: Raw data from the ILS – high speed creep curves for each material

It can be seen that Sample A and H have a greater variation across the 11 labs, than Sample D and F. This was attributed to the inhomogeneous nature of the TOR materials, leading to differences in the composition of the coating on the

MTM disk, leading to differences in the measured friction. It has also been noted in field trials that the friction coefficient measured on rails is variable when conditioned with TOR materials [7].

The data points at a speed of 1 m/s and 1 % creep are plotted in Figure 11 for each sample, along with the mean with the standard deviation denoted by the error bars. Table 5 gives the mean, standard deviation and number of data samples (n) shown in the plot.

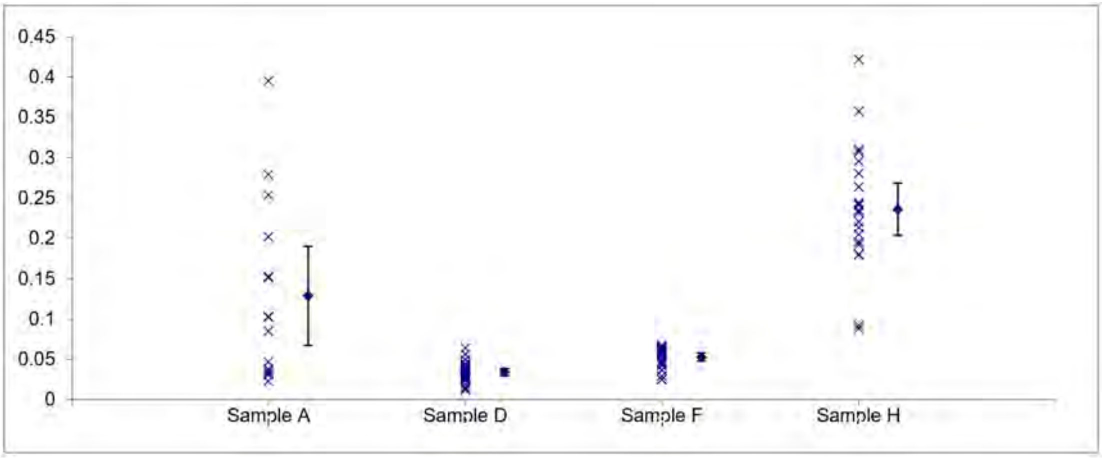

Figure 11: Plot showing the distribution of the measured traction coefficient at 1 m/s and 1 % SRR

Table 11: Population statistical values for the ILS

Multiple Blob Diagram	Sample A	Sample D	Sample F	Sample H
Mean	0.13	0.03	0.05	0.24
SD	0.111	0.012	0.013	0.075
n	15	31	31	23

Figure 11 shows that although the spread of the results is large for the TOR materials across the 11 different labs and operators, there is still a statistical difference measured between the different product types. Where samples D and F are clearly differentiated from Sample A and H using this method.

The data was analysed using the Analysis of variance method to ascertain the likely precision of the MTM test method. The measured traction coefficient at each SRR was treated independently in the analysis, to understand the variation across the creep curves. At 1 m/s the reproducibility (95% confidence interval) can be estimated as 0.02 for the flange/dual products and 0.2 for TOR materials. The repeatability (95% confidence interval) can be estimated as 0.02 for flange/dual products and 0.15 for TOR materials.

The variation of the measured traction within the laboratories is shown in Figure 12. This shows graphically the confidence interval for repeating this experiment on one instrument with one user. Clearly, variation in the measured traction can still be expected for TOR materials. The flange and dual products (Samples D and F) show a very narrow range of expected measured friction for repeat experiments.

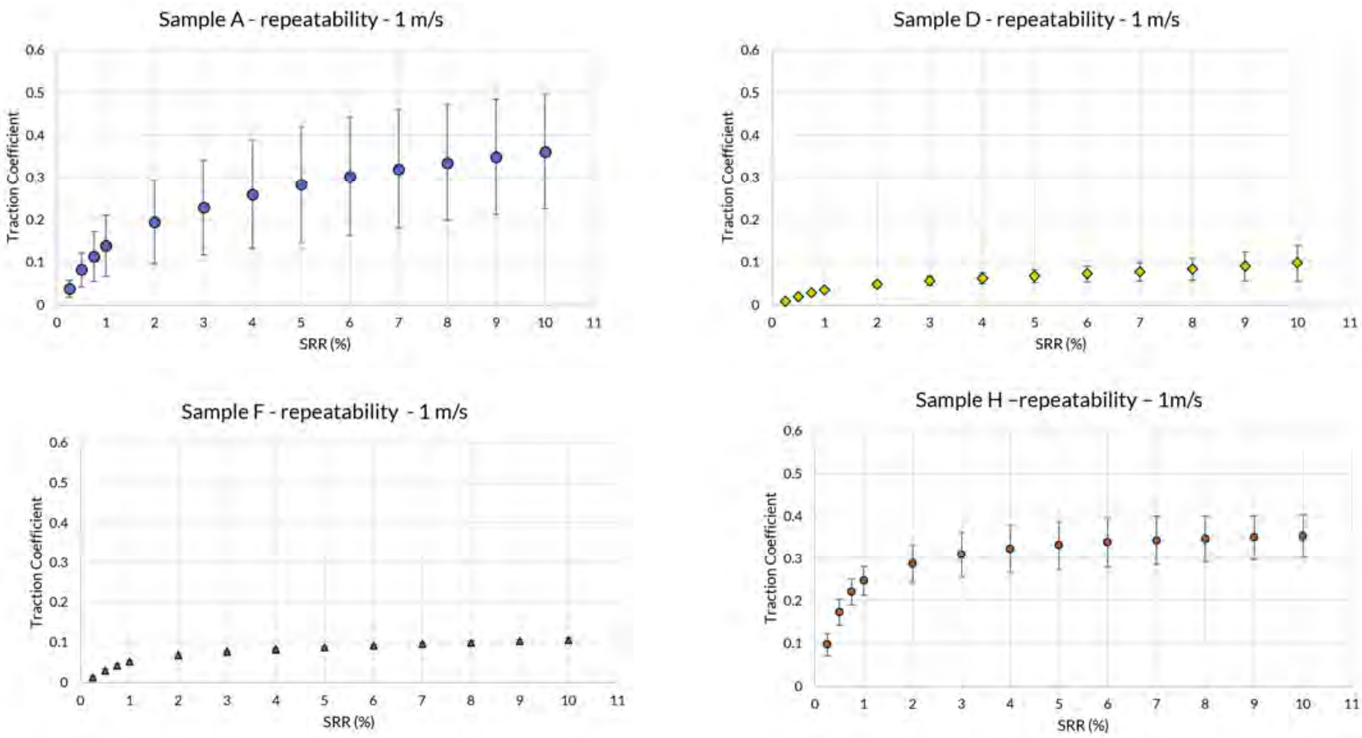

Figure 12: Average creep curves traction coefficient showing the expected confidence interval for a single user and laboratory

<u>Standardisation</u>

This method is currently included in the draft standard for TOR materials and flange products as an indicative test [8]. Meaning that this method can help in early stage evaluation of new candidate products for railways, before further full scale testing is required for qualification.

Currently flange products are specified to have a traction coefficient of below 0.13 over the entire range of the creep curve at 1 m/s (medium speed). TOR materials are required to have a traction coefficient of above 0.11 at 1 m/s and 10 % SRR using this method.

Summary

A new test method has been developed to evaluate the performance of TOR materials and flange products that are used in wheel rail interfaces. This method demonstrates the friction characteristics of the products through conventional creep curves, at 1 and 3.8 m/s linear speeds. These curves give an indication of noise generation and available traction at the wheel/rail interface.

An ILS has been completed using this MTM creep curve method with 4 railway products. The method is able to differentiate the performance of the products. The method is now being added as an indicative test to the European standards for TOR materials and flange products.

Declaration

The work described here was sponsored by the Rail Safety and Standards Board (RSSB) in collaboration with the European Lubricating Grease Institute (ELGI) working group on Railway grease.

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[8] Railway Applications – Wheel/Rail friction management. EN 15427: 2019, CEN/TC 256 (in draft)

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Preliminary Grease Training Course Programme

26th-27th October 2021 Amsterdam

Tuesday 26th October	Timing	Lecturer	Company
• Introduction	09:00 - 09:15	Leandro Muntada	Brugarolas
• Base Oils	09:15 - 10:30	Mehdi Fathi	Nynas
Coffee Break	10:30 – 11:00		
• Thickeners	11:00 - 12:30	Constantin Madius	Axel
Lunch	12:30 -13:30		
• Additives + Solids	13:30 - 15:00	Joe Kaperick	Afton
Coffee Break	15:00 – 15:30		
• Production	15:30 – 16:00	John Kay	Stratco
• Round table / discussion Day 1	16:00 – 16:30	Leandro Muntada Gaia Franzolin / All	Brugarolas / Nynas /All
Wednesday 27th October	**Timing**	**Lecturer**	**Company**
• Testing & Rheology	8:30 – 10:00	Olaf Hoeger Michael Anderson	Shell Falex
Coffee Break	10:00 - 10:30		
• Applications	10:30 - 11:30	Aleksandra Nevskaya	Dow
• Compliance and Packaging	11:30 - 12:30	Andreas Dodos	Eldon's
Lunch	12:30 – 13:30		
• Trends	13:30 - 14:30	Manfred Jungk	MJ-Tribology
• Food	14:30 -15:30	Andre Adam	Fragol
Coffee Break	15:30 – 16:00		
• Round Table / Discussion Day 2	16:00 – 16:20	Leandro Muntada Gaia Franzolin / All	Brugarolas / Nynas /All
• Hand Out Diplomas	16:20 – 16:30	Leandro Muntada Gaia Franzolin	Brugarolas /Nynas

For Organisational Questions	For Technical Questions	
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Calendar of Events

13th International Colloquium Fuels 15th-16th September 2021 Stuttgart-Ostfildern, Germany	maschinenbau@tae.de www.tae.de
53rd GOMA Symposium Fuels 2021 16th-17th September 2021 Zagreb Croatia	goma@goma.hr www.fuels.goma.hr www.goma.hr
NLGI 88th Annual Meeting 27th-30th September 2021 Loews Ventana Canyon Tucson AZ.	www.nlgi.org crystal@nlgi.org
ELGI Autumn Events: 25th - 29th October 2021 Amsterdam Netherlands and/or Virtual	
ELGI Bi-Annual Working Group Meetings 25th October	
Food Grade Lubricants WG Meeting	a.adam@fragol.de
Grease Particle Evaluation WG Meeting	Joe.Kaperick@AftonChemical.com
Test Methods & Rheology WG Meeting	olav.hoeger@shell.com
Bio-Based Greases WG Meeting	g.dodos@eldons.gr
Railway Lubricants WG Meeting	matt.smeeth@pcs-instruments.com
ELGI Grease Training Course 26th - 27th October Amsterdam Netherlands	lmuntada@brugarolas.com gaia.franzolin@nynas.com carol@elgi.demon.nl www.elgi.org
ELG-STLE Tribology Exchange Workshop 28th - 29th October Amsterdam Netherlands	manfred.jungk@mj-tribology.com carol@elgi.demon.nl www.elgi.org
UEIL Virtual Annual Congress 20th - 22nd October 2021	www.ueil.org secretariat@ueil.org
11th Global Lubricant Exhibition & Conference 2021 October 2021 Moscow Russia	www.rpi-conferences.com konstantinova.elena@rpi-inc.ru
UNITI Technology Congress 3rd-4th November 2021 Stuttgart Germany	www.umtf.de loeser@uniti.de
F+L Week Live 15th - 18th November 2021 Sukhothai Hotel in Bangkok, Thailand	conference@fuelsandlubes.com
OilDoc Conference & Exhibition 2021 17th - 19th November 2021 Rosenheim Germany	www.oildoc.com pb@oildoc.com
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International Colloquium Tribology 25th-27th January 2022 Stuttgart-Ostfildern Germany	www.tae.de emarketing@tae.de

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