

# WHITE PAPER

# Lubrisense<sup>TM</sup>

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HEAVY DUTY GREASE  
GREASE MANUFACTURING

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# Heavy Duty Grease

According to one of the most renowned tribologists of our time, Professor Duncan Dowson, “Fluid film lubrication is associated with the physical rather than the chemical nature of lubricants, and modern bearings rely substantially on this exceptional mechanism for separating sliding solids. However, all bearings start and stop, and the surfaces of the sliding solids come into contact, with each interruption of movement ... In many dynamically loaded bearings ... and gears ... the friction and life of the machine are greatly influenced by films of molecular proportions formed on the solids by additives. Such substances, added to mineral oil, have contributed in a major way to the spectacular development of lubricants in the second half of the 20th century”.

Dowson, D.,  
Introduction chapter in  
“Chemistry and Technology of Lubricants”  
Second edition, 1997  
Edited by Mortier & Orszulik  
ISBN 0 7514 0246 X

For this very reason, and in particular for heavy duty applications, the great majority of lubricating grease formulators have relied on additive technology to compensate for the shortcomings of the base product itself. When the physics are not sufficient, the thin film chemistry of the additive system saves the bearing from catastrophic failure through different reaction mechanisms, even though there may only be a mono-molecular layer of material available. Obviously, it is highly essential that these chemistries be available at the right place, at the right time, at the point of contact between the moving surfaces. Available on the present day grease market is therefore an abundance of products which the manufacturers claim to have been specially designed to perform under such high load conditions. These are often called EP greases (EP = Extreme Pressure) and within that particular category of products, there are many different levels of EP performance. The great majority of commodity EP greases are simple lithium thickened greases based on the patents of Clarence E. Earle from the 1940s but with a small amount of some secret potion designed to give the required performance in one

## Grease Manufacturing

The great majority of present-day lubricating greases are produced in some kind of a saponification process where most of the manufacturing parameters are predetermined by the reaction mechanisms necessary for the particular soap in question. Fatty acids are melted in oils, metal hydroxides added, the temperature raised to provide energy for the reaction, the water removed and, in most cases, the temperature raised to a given peak in order to melt or at least effectively disperse the soap in the total mass. The material is then cooled according to a specific temperature cycle, additional oils and additives introduced, the consistency adjusted to the required NLGI class, and the final product is put through a rigorous finishing

process including homogenisation, deaeration, filtration and packaging. This requires a complicated and, by implication, expensive series of different kinds of equipment.

The saponification process is carried out in two different types of production units, in batch production, in open or pressurised kettles or so-called “Contactors™”, or alternatively in continuous production through small reaction chambers. The chemistry is however, in principle, the same in both cases. Most thickener systems are produced in-situ by saponifying the fatty acids with metal hydroxides in a portion of the base oil and in the

or more of the EP test procedures accepted in our industry (4-ball weld load, Timken OK load, etc). Most of these are “me too” products where different chemistries are used to achieve the same effects. “It’s an EP 2 !” However, with an increasing understanding of the mechanisms involved, some interesting progress has been made over the recent years.

After the Second World War, lubricant manufacturers started to become more interested in producing lithium based greases and early experiments using lithium hydroxide can be traced back in AXEL’s laboratory journals to 1948, where the first commercial product was a low temperature prototype using a combined lithium-barium soap in a low viscous mineral oil. In order to improve the load carrying capacity of these new lithium based greases, many different combinations of additives were tested. The most successful packages were actually designed primarily for gear oil formulations and often contained a combination of lead and sulphur. To meet the Volvo specifications of the early 1960’s, for instance, a combination of lead naphthenate and sulphurised sperm oil was used. This type of additive package became the industry standard for many years, since it offered both an improved EP effect and, at the same time, excellent corrosion inhibition and it is, in fact, still used in some parts

of the world today. However, with an increasing concern about issues regarding health and the environment, the development process continued in an effort to find better and safer products.

Lead is a toxic heavy metal and sourcing sperm oil was seen as a threat to the world’s population of whales. Slowly but surely, these products have been replaced by other effective materials.

In the mid-1970’s AXEL introduced a fully qualified non-lead lithium EP grease which was tested thoroughly and eventually approved by the Swedish Railways and this was the start of a new era in heavy duty grease formulation. The replacement materials were still however originally developed primarily as gear oil additives.

Additives for motor oils could not be used since they often contained detergents of some kind and these were highly polar in nature and detrimental to the formation of a proper grease structure.

The grease industry moved on from lead and sulphur to a combination of sulphur and phosphorous.

Other interesting options were the use of borate technology, phosphinic acid derivatives or additives containing antimony or organic molybdenum compounds. Another very interesting alternative available today is the use of bismuth containing additives. Bismuth is, of course, a heavy metal but studies show that it does not have the same kind of toxic behaviour as its predecessor, lead.

presence of some water. The temperature is raised to the specific reaction temperature, kept there for a suitable period of time to ensure a complete saponification and then heated up to an even higher temperature to dehydrate and, in many cases, melt the soap. This can be at temperatures in excess of 200°C. Because lubricating greases have so poor heat transfer properties, the cooling process is often the bottleneck of any production process. In addition, on cooling, the soap crystallises into its characteristic fibre structure (analogy with a sponge full of water) and the cooling rate is often vital in achieving the required physical properties. So speeding up the process is often undesirable. In order to optimise this stage of the

process, cooling is often promoted under continual stirring. No matter how smoothly the cooling operation proceeds, the resulting mass is a relatively lumpy mixture and must be homogenised.

This can be done in a multitude of ways, by pressure valves, tooth colloid mills and high pressure homogenisers. The finishing process, in which the additives are introduced, the consistency adjusted to the required level and the whole mixture homogenised is supplemented with a deaeration unit, special filters and a whole array of packing systems. Different greases require different manufacturing methods and each grease plant has its own particular detailed technology.

Even if a saponification process is used in the

Greases for heavy duty applications need optimised EP properties but that is not sufficient in itself. There are many other parameters to be taken into consideration and this complicates things further.

Most of these so-called heavy duty applications can be found in wet and dirty environments. Some examples can be found in steel works where, in a rolling mill for instance, there is an abundance of water and/or emulsions and the surrounding environment is far from clean. Heavy duty vehicles drive around in quarries and forestry processors and excavators are expected to work in ditches and other wet and muddy locations. The ability of the grease to stay in place and act as a seal against both fluid and solid contaminants is vital for the optimal functionality of the vehicle and the adhesive and water repelling properties of the grease become paramount. This has been addressed by incorporating special thickener systems into such greases. Calcium greases are much more water resistant than lithium greases and a new generation of anhydrous calcium products has been developed for this very purpose. Combinations of lithium and calcium are also used where the good properties of both soaps have been used with reasonable success. To improve the adhesive properties of the grease (not to be confused with the cohesive properties where the

material becomes like chewing-gum) different types of polymers have been used and a recent innovation has been the use of so-called “functional” polymers where they are attached to the soap structure rather than being dispersed throughout the base oil. Some polymers are even designed to thicken in the presence of water, further enhancing the sealing properties of the product. Since the soap thickener in a lubricating grease is often much more highly polar than the base fluid, it will win any competition for the metal surfaces available and one way of optimising the effect of the additive systems is to attach the active components to the soap matrix instead of dissolving them in the base oil. Traditional calcium complex greases, for instance, contain calcium acetate and that performs well in itself as an EP agent. This effect opens up many possibilities for the formulation of new and more effective heavy duty greases and, rather than having to rely on the use of primarily gear oil additives to achieve the load carrying capacity, the present day grease formulator can attach different chemistries to the soap structure. The soap has not only a thickening effect but also a number of different functions such as EP properties, adhesion, corrosion inhibition, durability etc. Therefore, in correspondence to polymer terminology, AXEL have chosen to classify these modified thickeners as functional soaps.

great majority of grease making methods in the world today, there are many other types of products which require other types of production processes and equipment. Some metal soaps can be produced in advance and the use of these pre-formed soaps is common in the manufacturing of bio-greases and greases containing other base fluids sensitive either to the high temperatures or to the alkaline environment present in a saponification process. Other greases are based on non-soap thickeners. Polyurea, for example, is a low molecular weight “polymer” formed by first dispersing specific types of iso-cyanates and amines in separate portions of the base oil and then mixing the two substances together. The final reaction is exothermic and

because of the toxicity of the raw materials, the reaction vessels must be efficiently sealed off from the working environment. Another example of a non-soap thickener is clay. These clay thickened greases are fairly easy to prepare, by dispersing the platelets in the base oil at relatively low temperatures. A polar additive is then added as an activator and a high shear milling or homogenisation process completes the formation of the gel structure. On a smaller scale, a wide range of other non-soap thickeners are used for more specialised applications. These include silica-gel, carbon black and synthetic organic polymers such as PTFE. Before being put to use, each one of these different thickeners has been exposed to different manufacturing techniques.

Whether intentionally, or not, there are a number of different functional soaps already available on the market and these are often excellent for heavy duty applications. Examples here are the previously mentioned traditional calcium stearate-acetate complexes, and their present day successors, the calcium sulphonate complex greases. By combining such soaps with a high viscous base oil, modern inhibitors and perhaps even solid additives, excellent heavy duty greases can be offered.

Some of these functional soaps have however disadvantages in addition to the above mentioned positive effects. Calcium complexes have a tendency to “age harden” and many of the new generation of sulphonate complexes have inferior lubrication abilities at ambient temperatures.

To compensate for this, we at AXEL have chosen to combine the advantages of calcium complex with the excellent lubricating properties of lithium complex. This complex soap type is called “Alassca”, which has nothing to do with any geographic location but is, in fact, an acronym for the components of the soap.

An Alassca complex grease containing no more than the soap and the base oil, with no additional additives, will meet the highest possible specifications in the 4-ball weld load test machine (> 7000N).

In addition, it is extremely adhesive and water resistant. This “base grease” can then be tailored

to meet the exact requirements of different heavy duty applications. One illustrative example is in lubricants for heavy open gears, often found in the mining and cement industries, where the use of large quantities of graphite has been the industry benchmark.



FIELD TESTING OF A HEAVY DUTY OPEN GEAR GREASE.  
GOLD MINE, MALI, NORTH AFRICA

As an example, AXEL has recently been awarded the European patent for a novel polypropylene based thickener system and this requires a completely different manufacturing technology (this will be highlighted in a future version of the Lubrisense White Papers™).

The manufacturing of lubricating greases on a large scale involves, more often than not, a complicated saponification process requiring an extremely high investment in both capital resources and manpower. The number of commercial grease plants is steadily decreasing in the western world mainly because of two important factors :

the ongoing rationalisation and mergers within the major lubricant manufacturers and the cold economic facts of investing in in-house manufacturing for products perceived to be both non-core and low price commodities. There are therefore very few hypermodern grease plants in existence, most of the current facilities originating from the 1950's or even earlier.

## Once upon a time



"THE LITTLE OLD GREASMAKER"  
COURTESY, NLGI, KANSAS CITY, USA

## State-of-the-art



NEW MODULAR GREASE PLANT UNDER CONSTRUCTION AT  
AXEL'S DUTCH FACILITY

The front page of the June 1992 edition of the NLGI Spokesman gives a typical illustration of how grease manufacturing is perceived within our own industry. This "little old greasemaker" concept is something out of the history books which has survived the perils of time. In the very same issue of the Spokesman, Burkhalter claimed that "compared to grease product development, grease process development has stagnated during the last 20 to 30 years, with little change in equipment or systems occurring since the commercialisation of the continuous grease process. Although new products were developed, actual production was via the old methods. The lack of improvement in process development can be attributed to the long-held

belief that grease production was a "black art" characterised by low profitability. Because of the lack of process development, (North American) grease facilities have not kept pace with advances common to the chemical and petroleum industries." And, in addition, the one positive development mentioned here, i.e. the commercialisation of the continuous grease process, has not proved to be as successful as was expected. Because of the changing nature of the grease business, this type of production unit is now slowly but surely disappearing since it is geared towards the manufacture of large volumes of one and the same product and it does not offer the flexibility needed by the major grease producers of today.

Today, with an Alassca soap, this has become completely unnecessary. Not only can the specifications be met, this new technology offers the possibility of monitoring wear on the gear teeth by using a stroboscope, since there is no longer a thick layer of graphite blocking the way. In addition the build-up of graphite between the gear teeth can be completely eliminated and the vibration levels reduced significantly. Another new and exciting functional soap can be produced by introducing bismuth into the thickener structure. This has previously been illustrated in a special edition of the Lubrisense™ White Papers issued in early 2006. Advantages of this particular functional soap are low friction, longer bearing life, a wide temperature range, better mechanical stability and better pumpability.

Even though a very big part of our lubricating grease industry is still exceptionally conservative and extremely conventional, the metamorphosis from know-how to know why has given us the possibility to take a quantum leap forward. There are admittedly still very many conventional “me-too” (EP 2) greases offered on the market and which are often recommended for heavy duty applications. In contrast, at the other end of the scale, where the properties of a lubricating grease are a vital part of operations and maintenance, there are now highly effective and modern products available as solutions to the ever increasing demands from both OEMs and end users.

Hard criticism indeed for the present generation of grease makers.

Modern lubricating greases contain a wide variety of different chemical substances, from complicated mixtures of natural hydrocarbons, through well defined soap structures, polymer solutions and even complex organic molecules in the additives to “simple” chemicals such as calcium carbonate and graphite. If the formulations are regarded as proprietary and confidential, then the manufacturing processes must be classified as top secret. The “art” of making grease has seemingly more to do with the making than the grease.

There are normally two reasons for keeping things secret. You either have something to hide, or you don't !

Most “modern grease plants” have the know-how to produce “modern greases”. However, very few have the know-why and this is the key to grease innovation. A paradigm shift from know-how to know-why might eliminate all the art and the black magic and we can get on with developing the greases of tomorrow in a much more scientific manner.

In the next issue of the White Papers, we will address the issue of lubricating grease in the food industry, including both the current standards (NSF listing etc) and some new proposals (SS-EN ISO 21469 : 2006). In addition to technical and safety parameters, certain religious features (Kosher, Halal etc) need attention. Contributions will be made by external guest writers and by experts from our

newly acquired production facility in France (Christol Grease) who are specialised in foodgrade lubricants. As usual we encourage reader contribution, feedback and proposals for topics in future editions of the White Papers..

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