WHITE PAPER

Lubrisense™ 201010

UNDESIRABLE PARTICLES

"Deleterious Particles in Lubricating Greases"

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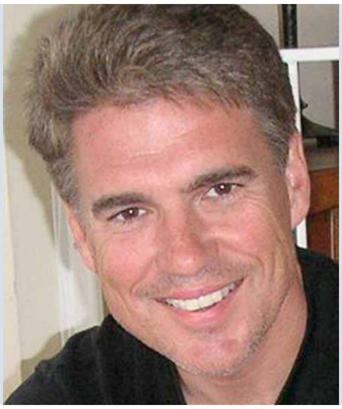
Clean grease = Long life

AXEL CHRISTIERNSSON

DELETERIOUS PARTICLES IN LUBRICATING GREASE



CHUCK COE holds a BS Chemical Engineering, Pennsylvania State University, along with NLGI CLGS and STLE CLS professional certifications. He worked for Mobil and ExxonMobil for 32 years, including 6 years as ExxonMobil's Grease Technology Manager. He retired from ExxonMobil and launched Grease Technology Solutions LLC, a grease training and consulting business in October, 2009. He is the Grease Education Committee Chair of STLE, and is both the acting Vice President and the Technical Committee Co-Chair for NLGI. He has authored several technical papers and articles on grease, and received Best Marketing Paper and Best Paper awards from both NLGI (2008) and ELGI (2009).

FIGURE 1 – METALLIC MACHINING DEBRIS

There are a vast number of different particle types which can be found in lubricating greases. In the previous issue of the Lubrisense™ White Papers, *desirable* particles were discussed at length by Persson and Fathi-Najafi. It turns out, in fact, that there are numerous types of these desirable particles, and many important purposes for their intentional incorporation into greases. Now we shall examine the other side of the coin: particles in greases which are viewed as harmful, or at best, not serving a useful purpose.

Types of particles

First, let us divide all particles broadly into two classes: abrasive and non-abrasive. In the *abrasive* category, we find such undesirable materials as metallic fines from machining (Figure 1) – iron, steel, copper, brass, bronze. Other metallic particles may include wear debris – iron, steel, copper, brass, bronze, lead, and oxides of the same (Figure 2). Corrosion of iron and its alloys creates iron oxides, which vary in hardness and therefore abrasiveness. And lastly, there is the ubiquitous dirt, usually composed mostly of sand, or silicon (Figure 3).

FIGURE 2 – METALLIC PARTICLE UNDER MAGNIFICATION

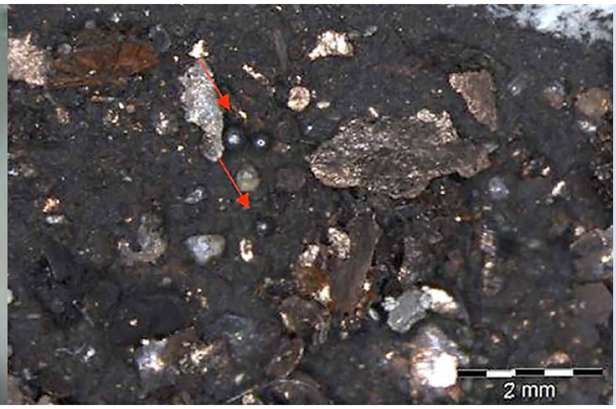


FIGURE 3 - METALLIC AND OTHER DEBRIS

In the *non-abrasive* class, some of the more commonly found undesirable particles consist of agglomerates of either soap or non-soap thickeners. Some cases of unintentional agglomerates or insoluble additives have been observed. Finally, we have the desirable additive solids – molybdenum disulphide, graphite, etc., which were thoroughly presented and discussed in the previous issue of the Lubrisense™ White Papers.

Sources of deleterious particles

There are easily as many sources of unwanted particles as there are types. Starting at the beginning, particles may be introduced into grease during manufacture via the *raw materials*. Contamination entering the grease this way may be due to contamination by the supplier's process or handling. An example of this might be incomplete reaction during preparation of a multifunctional additive, resulting in a material which is either insoluble or agglomerated, which could result in insolubles or agglomerates in the grease. Another example might be contamination from the supplier's packaging, such as rust, dirt, paper fiber or plastic debris in additive drums or bags.

In addition, contamination from raw materials may occur as a result of improper storage or handling by the grease manufacturer. Poor raw material inventory management can lead to materials degrading due to aging or incorrect storage conditions. One example is overaged / poorly stored lithium hydroxide bags, allowing the formation of lithium carbonate particles. Another example is the undesirable formation of dimers in liquid diisocyanates used in the manufacture of some types of polyurea greases, caused by improper storage temperatures, or excessive age.

Next, after potential raw material sources of contamination, we have the *manufacturing process*, itself. During manufacturing, there are many possibilities for contamination to occur – let's face it, most grease plants tend not to be the cleanest of environments! First, one must be aware of the potential for improper incorporation of raw materials – wrong temperature, insufficient mixing or milling, inadequate filtration, etc. Any of these errors can result in agglomerates or undissolved ingredients (Figure 4). During the process of adding raw materials to a batch of

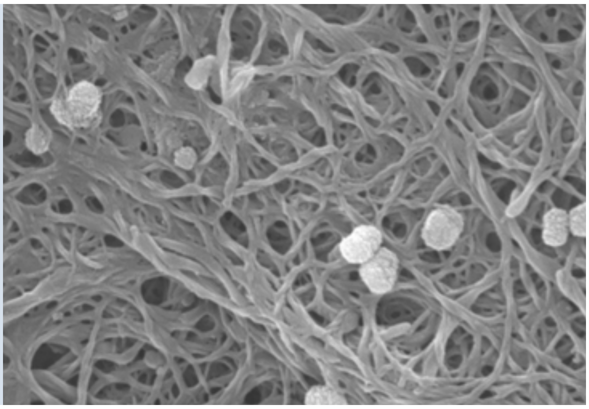


FIGURE 4 – UNREACTED LIME PARTICLES IN SOAP THICKENER MATRIX

grease, it is also quite easy for dust, dirt and other environmental contamination to enter the grease. Another less obvious source of contaminating particles is from poor equipment cleanliness, such as residual product or hardened thickener from prior batches, or environmental debris resulting from poor housekeeping practices. And finally, during manufacturing, if there are worn pumps, poor seals, etc., this may cause metallic or elastomeric contamination to enter the grease as it is recirculated.

Now, assuming the raw materials and manufacturing process are well controlled, there is still the potential to introduce contamination to the grease during the *packaging process*. Sources of dirt and other contaminants include dirty (improperly flushed) packaging equipment, inadequate filtration and contaminated containers.

Even if a grease is contaminant free post-manufacture and packaging, once it arrives at a customer's location, there are numerous opportunities for it to become contaminated. At the end user facility, *storage & handling* conditions and inventory control are critical. Poor housekeeping after containers are opened can lead to ingress of environmental contaminants such as dirt, dust or other airborne particles. And if a grease is stored beyond its shelf life, there is the potential for additives to recrystalize. Some rust inhibitors and antioxidants are particularly prone to this problem.

And finally, if the grease is still free of contamination up to the point where it is introduced to the *application*, poor relubrication practices (not cleaning grease fittings prior to re-greasing, etc.) or poor seals (allowing ingress of environmental contaminants such as water, dirt, dust, etc.) are common causes of contamination by deleterious particles.

Problems caused by particles

So what are the problems caused by deleterious

CLEAN GREASE = LONG LIFE

The importance of grease cleanliness on bearing life was highlighted by the award of the Tribology Trust's 2008 Gold Medal to Professor Stathis Ioannides, SKF Group Director for Product Engineering. He has been honoured for his work in developments of both theory and practice on rolling contact fatigue. During the 1980s, Ioannides and Harris developed the SKF Life Theory which introduced the presence of a fatigue limit as a major factor affecting bearing life. Further work recognised the detrimental effects of lubrication and contamination on bearing life.

The International Standards Organisation (ISO) has adopted this "new" SKF Life Theory into "ISO 281:2007 Rolling Bearings – Dynamic load ratings and rating life". Every major bearing manufacturer worldwide is now committed to the ISO 281:2007 standard. The revised equation includes a life adjustment factor (a_{skf}) which, in addition to the fatigue load limit, also takes into account the lubricant film thickness and the degree of solid contaminants. In turn, one part of the a_{skf} mathematics is a contamination factor (η_c) which is somewhat complicated particles in grease? Broadly speaking, we can view the problems in three categories (although they are not mutually exclusive): Damage to bearings, noise in bearings, and clogging of distribution equipment.

Damage: If the hardness and size of the particles are sufficient, over rolling of the particles in a rolling element bearing can result in permanent damage to the bearing. This causes denting of the raceway, which in turn results in high stresses at the edges, leading to spalling and reduction in the fatigue life of the bearing (Figure 5). Additionally, if sufficiently hard, the particulate contamination may cause abrasive wear, also leading to premature failure of the bearing.

Noise: Even if the particles are not hard enough to result in permanent damage to the bearing, the over rolling of these particles will cause an increased level of noise in the bearing. Noise will result from over rolling of even relatively soft particles, such as undispersed thickener. Why do we care about noise anyway? The property of "quietness" or "low noise" is becoming more important in the world of grease. There are several reasons for this, the most well known being the growth in consumer electronics and appliances. Consumers don't want to listen to bearing noise when they are using a fan or a piece of audio equipment. A second reason is not

FIGURE 5 – DEBRIS DENTING

consumer related, but industrial in nature. Many types of equipment such as grinders used in metal processing produce high levels of noise, which can be a health and safety hazard. Grease which is "noisy" can contribute to this already challenging problem. And finally, a third area where noise can be a problem is during bearing manufacturer quality control processes, where grease noise can mask noise or vibrations caused by defects in the bearing itself.

Clogging of filters and distribution equipment by excessive particulates is also a significant problem, and depends on the size, hardness, shape and quantity of the solid particles as well as operational factors such as bearing size, lubricant film thickness, loads etc. Since it has been shown that bearing life can be reduced by *as much as 90%* compared to calculated nominal life expectations, the importance of cleanliness for lubrication becomes clearly obvious. In simple terms, the cleaner the grease, the better!

For fluid oils, cleanliness can be improved by conventional filtration of the finished product. This is especially important in regard to hydraulic fluids in aviation and other sensitive applications and environments. For lubricating grease however, it becomes somewhat more complicated. A common illustration of the structure of lubricating grease is as a sponge full of water (see Lubrisense™ White Paper 2004/01). This may not be entirely correct in a strictly scientific perspective but it does serve to highlight some of the problems arising in the manufacture and use of lubricating grease. How do you pump a sponge full of water, for instance? And in the context of this particular publication, how do you filter a sponge full

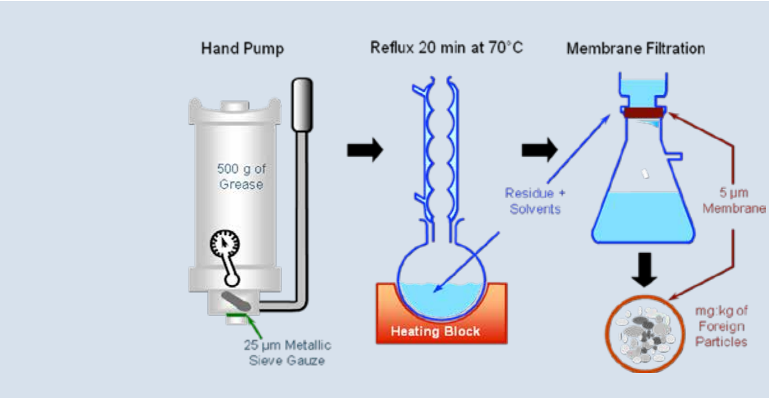


FIGURE 6 – DIN 51813

particularly where the grease is distributed by a centralized lubrication system. These systems usually have very small clearances, so may incorporate filters to prevent particulate material entering and plugging the grease lines and distribution blocks. If the filters become clogged, or if the distribution system itself becomes clogged, this can lead to lubricated equipment failures due to lubricant starvation in the bearings.

Testing for deleterious particles

There are several different tests which can be used to determine the presence and/or type of particles in grease. First, we'll discuss the tests which can be used to determine the *amount* (size and concentration) of particulate. A test in use by the paint and coatings industries, which is designated ASTM D1210 uses a device called the Hegman gauge. In this method, the particles in a grease are counted and sized from zero to 100 microns using a machined depth gauge. This method is not in common use in the grease industry, but is being seriously considered as part of a draft grease cleanliness definition under development by a European Lubricating Grease Institute (ELGI) – National Lubricating Grease Institute (NLGI) Joint Working Group. Another method in use to determine size and concentration of particles in grease is designated DIN 51813, Solid Matter Content of Lubricating Greases. In this method, a sample of grease is passed through a 25 micron filter, and then the residue is solvent washed, dried and weighed to give the milligrams of particulate (greater than 25 microns) per kilogram of grease (Figure 6). And yet another method which finds occasional use is FTM 3005.4, Dirt Content of Lubricating Grease. In this method, a small sample of grease is examined under a microscope to determine the size and number of particles present (Figure 7).

Equally as important as determining the size and concentration of particles is evaluating their

of water? Slowly and carefully would perhaps be a good piece of advice. It is obviously imperative not to extract the solid fibres of the thickener system (or any other "desirable" particles for that matter) while, at the same time, essential to remove any deleterious particles potentially causing damage and noise. So too fine a filter may only serve to remove the thickener. Too coarse a filter will leave the contaminant particles in the grease. Too high a pressure will force the base oil out of the structure causing separation and possible clogging. For this reason, very few lubricating greases are filtered below 250 µm and many of the hard and sharp particles causing noise and reduced bearing life are left in the

GREASE FILTER

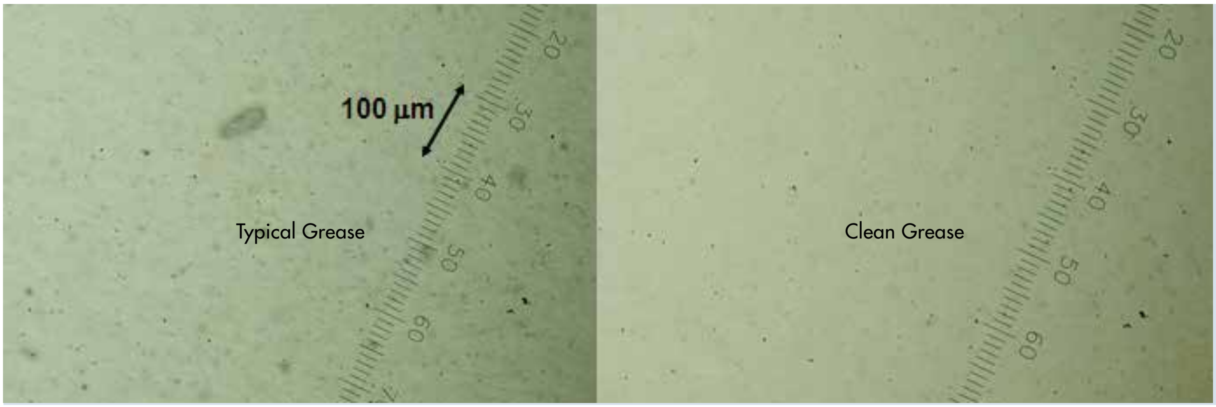


FIGURE 7 – MICROSCOPIC COMPARISON OF TYPICAL AND "CLEAN" GREASES

abrasiveness. Only one standardized test is in existence today to do this, designated as ASTM D1404, Estimation of Deleterious Particles in Lubricating Grease. In this method, the number of scratches that appear on the surface of highly polished acrylic plates under specified test conditions are counted. This method is also being considered as part of the draft grease cleanliness definition under development by the aforementioned ELGI – NLGI Joint Working Group.

It should be noted here that the Joint Working Group is moving towards adopting a definition of grease cleanliness which is "a measure of the absence of particulate matter that has the potential to cause damage in certain applications." As such, both the amount (or concentration) and size distribution, along with a measure of the abrasiveness of the contamination are likely to be proposed as part of a draft standard.

In order to measure the *noise* level of a grease, again there are no industry standard test methods in existence. Various bearing manufacturers have developed their own in-house methods, some of which are available to the general public. There

finished product. The sources of deleterious particles have been described in the white section and greasemakers can contribute to cleanliness by focusing on the raw materials, the manufacturing process and the packaging process. Once the product has left the grease making facility, the responsibility moves to the lubricant distributor and the end user.

Being based in Sweden, Axel Christiernsson has been well aware of the SKF equations and the importance of cleanliness for extended bearing life and has invested considerable time and resources to continuous improvement in this field. A major project was initiated to identify any contaminants, to ascertain the source of such contaminants and, in turn, to try to eliminate as many of them as possible. Industrial scale greases were carefully passed through a series of filters in an ever declining (finer) order. Particles of different sizes and shapes were found and the source of the particles identified. Since we built the Swedish plant in the early 1980s, all base oils are filtered just before entering the different production kettles through a 6 µm system originally designed for aviation fluids. This ensures the cleanliness of the bulk oils. Other important high volume raw materials are the constituents of the thickener systems and these are often delivered and

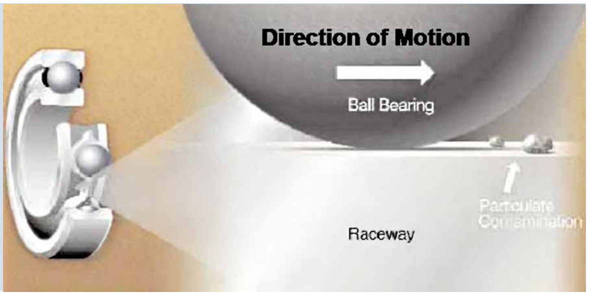


FIGURE 8 – ANDEROMETRY

are two different approaches to measuring noise, one using *anderometry*, and the other employing *acoustic* techniques. In anderometry, which appears to be the preferred technique, noise is evaluated by measuring the vibrational velocity of a bearing's stationary outer ring while the inner ring is rotated (Figures 8, 9, 10). Included in this category are: FAG MGG11, SKF BeQuiet+, and other OEM proprietary methods. In the acoustic techniques, noise is evaluated using sound amplification, and requires the use of a special sound chamber (Figure 11). Included in this category are JIS B 1548 and various OEM proprietary methods.

Specifications and Standards for cleanliness
There is a growing trend to define and include cleanliness requirements in a number of grease industry standards and OEM specifications. A few examples include:

- DIN 51813, Solid Matter Content of Lubricating Greases is part of the DIN 51825 standard for Type K Lubricating Greases. The limit is up to 20 mg/kg.
- The US Military specification MIL PRF 81322, General-Purpose, Aircraft includes FTM 3005.4, Dirt Content of Lubricating Grease. The limits are 1000 particles/ml max between 25–74 microns, 0 particles/ml max 75 microns or larger.
- ISO 15242-1:2004 defines and specifies measuring methods for vibration of rotating rolling bearings under established test conditions together with calibration of related measuring systems.
- Quite a few OEM internal standards and specifications have noise test requirements, in some cases based on proprietary test methods.
- As mentioned previously, the ELGI – NLGI Joint Working Group is developing a draft grease cleanliness standard, which is likely to be based on two tests, one for size and concentration of particulates and one for abrasiveness.

added to the grease kettles in solid form. Since it is difficult to remove particles once the grease structure has been formed, we try to remove any such contaminants before they ever get into the system. For typical lithium soap based greases, this can be done in two ways. The fatty acids can be melted and filtered before being used. The lithium hydroxide can be dissolved in water and then filtered. This latter process requires the evaporation of enormous quantities of water and the time and cost of the operation does not motivate the effort. Instead, lithium hydroxide can be added as a slurry, either in oil or in water, and homogenised using a high-speed dissolver to minimise the risk of any large and/or hard particles entering the system. A second possibility is to filter the soap at the peak temperature (> 200°C) after saponification and while it is still in the molten state. This requires very special equipment. We invested in such a filter system but it was later shown that very few particles were removed by this operation and once again, the time and cost did not motivate the effort. To our surprise, more than 90% of the particles actually came from the *additives*. Today, all our additives are filtered twice, first on arrival (either in bulk or drum by drum) and then again before being added to the finishing kettle. This simple operation of filtering the additives removed

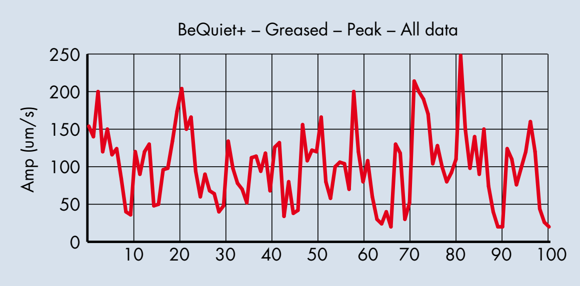


FIGURE 9 – NOISE RECORDING ON A "DIRTY" GREASE

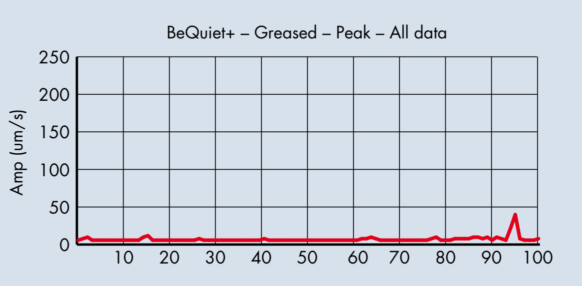


FIGURE 10 – NOISE RECORDING ON A "CLEAN" GREASE

– There had been some effort within ASTM to adopt a noise test standard, based on the SKF BeQuiet+ method, but at present, this effort appears to have been abandoned.

Measures to control particles

There are nearly as many techniques to control particles in grease as there are different sources of them. Again we begin at the beginning of the process. Improved *raw material quality control* is a good place to start. Purchasing raw materials from reputable suppliers who demonstrate a commitment to quality and purity is critical, along with specifying freedom from contaminants and shelf life in certificates of analysis. Another suggestion is for the grease manufacturer to perform incoming inspections on the raw materials to confirm packaging integrity and the absence of visible contamination. Filtration prior to use may be appropriate for base stocks and other high volume liquid raw materials. Stringent raw material inventory control is also necessary to prevent use of materials which are beyond their shelf life.

With raw material contamination controls in place, we next address the *manufacturing process* itself. Ensuring a clean environment in the grease plant is not always an easy task, but is critical to eliminate many sources of contamination during manufacture. A clean grease plant also helps create a culture of cleanliness and quality for the manufacturing personnel. Careful addition of solid raw materials using screens and other devices to prevent ingress of paper fiber and plastic from

almost all of the contamination which can be attributed to the raw materials.

In the manufacturing and packing process, the key to the whole issue is, of course, housekeeping. It is of the utmost importance to keep both the equipment and the buildings free from dirt, dust, debris and any other potential contaminants. Earmarking responsibility and empowerment as well as offering incentives for a job well done are possible ways to secure this "mindset". From our experience, particles from the process including unreacted materials, remains from previous batches, wear particles from the equipment etc are nearly always large in

LITHIUM HYDROXIDE DISPERSER

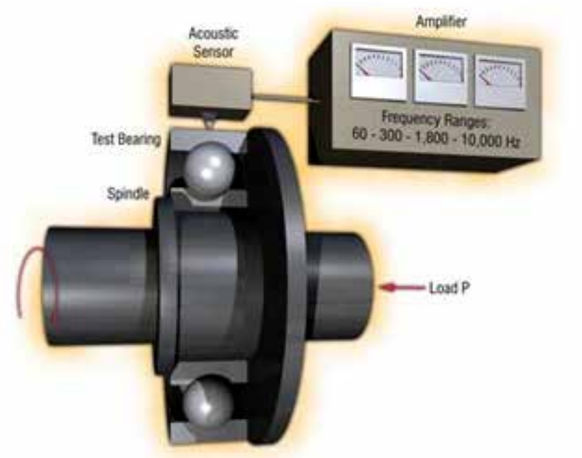


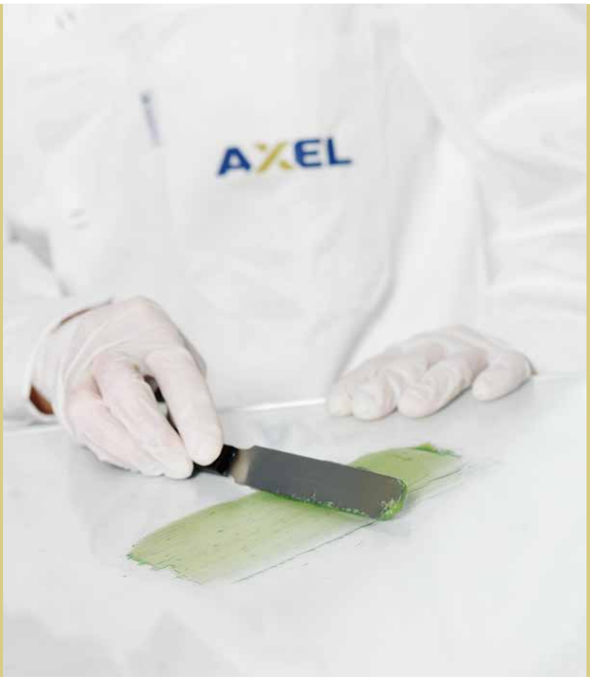
FIGURE 11 – ACOUSTIC GREASE NOISE MEASUREMENT

packaging materials is a very basic necessity. Thorough flushing of kettles, pumps, lines and other vessels between batches is an effective way to minimize contamination from residual product or hardened thickener. Following a well defined manufacturing procedure is also quite important to ensure addition of raw materials at the correct temperatures for proper dissolution. This procedure should also specify precise control of milling time, pressure, clearance, etc. in order to break up and disperse any large or crystalline particles or thickener agglomerates. And finally, if appropriate for the specific grease, filtration either during or at the end of manufacture can be used to remove any residual particulate over a specified size.

During the *packaging process*, the most straight-forward way to minimize contamination is through proper sequencing of filling operations and effective flushing of the filling lines and pumps between products. Additionally, new containers must be inspected prior to filling to ensure there is no evidence of paper fiber, plastic debris or rust. For some products and/or applications, the use of plastic liners in drums or kegs may be appropriate, which reduces contact with the container itself.

At the end user's location, we cannot overstate the importance of proper *storage & handling* practices. All containers of grease should always be stored indoors, or at least under a protective roof, to prevent water and concomitant dust and dirt ingress (Figure 12). Once a container is opened, any attachment to a distribution system, pump or filling of grease guns should be done in a manner to prevent introduction of dirt or debris into the open container. All open containers obviously must be stored indoors with the correct lids properly reattached. And in the process of introducing the

size and, as such, relatively easy to extract using conventional filters. A 250 µm slit filter followed by a 100 µm basket filter will normally suffice to remove this kind of contamination. Other options are to use "polaroid" filtration i.e. first orientate the particles and then catch them immediately afterwards or to use a much finer filter size and to reduce the pressure. This latter process will, of course, take a much longer time. Once the product is packed and leaves the production site, the responsibility of clean handling and housekeeping passes on to distributors and users. It is therefore extremely important that they also understand the importance of cleanliness to secure a smooth running operation.



grease to the *application*, a good lubrication program will include guidance to ensure identification of the proper grease, cleaning of zerk or other grease fittings, cleaning of the grease gun nozzle, etc. A frequently overlooked best practice is to ensure all purge plugs and pressure relief valves are in proper working condition to prevent over pressurizing during regreasing, which can damage seals, thus allowing easier ingress of contaminants to lubricated equipment.

Conclusions

There are numerous types of deleterious particles which can be found in grease. Some can be harmful to rolling element bearings or clog distribution systems. Some may not be directly harmful, but can cause increased noise levels in bearings, leading to other types of problems.

As with types of particles, there are also many different sources of contaminants in grease. Through good practices, particles can be controlled in every step from the manufacture of the raw materials used in the grease, all the way to the introduction of the finished grease to the end user's application. There are several different tests which can be used to quantify and / or characterize grease particulate contamination. Unfortunately, today there is no industry standard or consensus to define grease cleanliness. Continued work is needed within NLGI, ELGI, ASTM, DIN or other standards organizations to develop and promote such tests.

FIGURE 12 – PROPER LUBRICANT STORAGE

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NEXT ISSUE

The next issue of the Lubrisense™ White Papers will highlight ongoing changes in the global base oil industries and the impact of these changes may have on the formulation of lubricating greases. Replacement of Group I oils by "higher quality" Group II and Group III products could possibly cause problems for the grease formulator due to low solvency and perhaps even low viscosity. Fine tuning of thickener chemistries and new optimised additive packages will be necessary to convert grease production to these "new" base oils. Group I oils are produced mainly by a separation process whereas Group II and Group III oils are produced by a conversion process. By the end of 2015, it is expected that the highly saturated "converted" oils will constitute the biggest share of paraffinic base oil volumes.

As usual, we encourage reader contribution, feedback and proposals on topics for future editions of the White paper series.

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