UNDESIRABLE PARTICLES

“Deleterious Particles in Lubricating Greases”

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Clean grease = Long life
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-Najaf. 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).
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

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 insolubes 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.

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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 recrystallize. 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 particles?

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.

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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 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...
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 (NGLI) 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 particularity 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 (NGLI) 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).
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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 is only one standardized test 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.

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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 7 – MICROSCOPIC COMPARISON OF TYPICAL AND “CLEAN” GREASES

Typical Grease

Clean Grease
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...
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

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

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.
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.

During the packaging process, the most straightforward 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
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.

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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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