White Paper

The Basics of Lubricating Grease and In-Use Testing

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Introduction

Lubricants are applied to moving equipment for several reasons, including friction reduction, heat removal, corrosion prevention and contaminant removal. The most commonly understood reason lubricants are employed is to reduce friction, thereby minimizing wear between moving parts.

The three major categories of lubricants are fluids, semisolids and solids. In most cases, fluid lubricants are the most efficient, as they have the best flow properties. This strength can also be a fluid lubricant's weakness. In some types of moving equipment, it is impossible to contain the lubricant. In other words, it can easily leak out.

A lubricant cannot perform when it will not stay in the area where it is needed. In these cases, a solid lubricant that can be applied to a surface and not flow would be advantageous. Unfortunately, this creates a new performance challenge. In some cases, solid lubricants can be easily scraped away from moving surfaces. In such cases, a lubricant that possesses properties somewhere between a solid and liquid lubricant is needed. This semi-solid lubricant is called grease.

While many end users have just chosen to put lubricant into the equipment and let it run, many have learned that analysis of lubricants while in-service can provide useful information about the condition of the lubricant and the equipment in which it is installed. This condition monitoring of used fluid samples has been performed for many years and has become very mainstream. Due to the nature of solid lubricants, sampling and analysis often provides little to no sample and is often found to be of little use. Semisolid grease sample testing is, however, becoming more and more common, due to new sampling and testing procedures being developed within the industry. At this point, more useful information can be gained from in-service sampling of greases than of solid lubricants, but not quite as much as can be gained from fluids.

It is the goal of this paper to provide background and insight on grease and grease testing to help end users who are seeking to improve their lubrication reliability program through condition monitoring.

Grease

Grease is used in many different industries, including automotive, chemical, aircraft, manufacturing plants, power generation, and water purification plants. It is used in a variety of applications, including couplings, chains, bearings, gears and sliding surfaces. Grease has to withstand a lot of abuse and many different environments.

A. Grease samples in a tray

As defined by ASTM International, grease is a semifluid-to-solid dispersion of a thickener in a liquid lubricant. The dispersion of the thickener forms a two-phase system and immobilizes the liquid lubricant by surface tension and other physical forces. Other ingredients imparting special properties are often included (1).

Grease is usually characterized by its thickener composition, base fluid type and additive content. The following will provide a brief overview of the common base fluids, thickeners and additives used in grease formulations.

Base Fluids

Grease is often described as synthetic or nonsynthetic. This description generally refers to the type of base fluid used as a component of the grease. Lubricating grease base fluids are usually categorized into two broad categories – mineral oil or synthetic. Each of these categories can be broken down into subcategories.

Subcategories of mineral oils are paraffinic and naphthenic. Both of these types of mineral oil are derived through refining crude oil. The major difference is the level of hydrocarbon saturation. Paraffinic oils are more saturated than naphthenic base oils. This means that they are composed of more straight chain hydrocarbons and less aromatic and naphthenic ring structures in their molecular make-up. Many have found the solvency properties of the naphthenic oils to be superior to those of paraffinic oils, resulting in more stable grease with less base fluid bleed. However, paraffinic oils have generally been found to provide superior oxidation resistance and seal compatibility than naphthenic oils and are often used in the formulation of greases for which these properties are important

Numerous categories of synthetic base fluids exist, but some of the most common types used in grease formulas are polyalphaolefins (PAO), polyisobutylenes (PIB), esters, polydimethylsiloxanes, and perfluoroethers. PAO and esters are the most commonly used of these synthetic base fluids. These fluids are generally used to provide increased performance in applications that are exposed to the extremes of temperature or highly oxidative environments. The downside to the synthetic base fluids as compared to the mineral oils is their higher cost. For many applications, the extra cost may not be necessary as the mineral oil performance would suffice. The higher cost must be balanced against the performance.

Thickeners

Three broad categories of thickener systems used in grease formulations are insoluble solids, polymers and soaps. The first category uses insoluble powders that are introduced into the base fluid under high shear conditions until they are thoroughly dispersed and thickened into grease without any real chemical reaction. These formulations could be likened to the childhood favorite, the “mud pie.” Common solids include organo-clay, fumed silica, carbon black, and different types of pigments (2).

The next thickener category is polymers, such as polyurea and polytetrafluoroethylene. Polymer-thickened greases are composed of a base fluid and a polymeric material with gel-forming capability properly dispersed to produce the desired thickness of grease. The thickener is a low-molecular weight organic polymer that is usually formed in situ in the base fluid and yields no byproducts that must be removed (such as the water or alcohols produced in the formation of soap greases) (2).

The final, but most common, category of grease thickener is soap. The term “soap” is derived from the saponification reaction that results in the thickener. Saponification is depicted in the following general reaction:

$$MOH + ROOH \rightarrow MOOR + HOH$$

In this reaction, a metal hydroxide (base) is reacted with carboxylic acid to form a metal carboxylate salt (soap) and water. When this reaction is carried out in a lubricating fluid, it results in the formation of a crystalline soap dispersed in the fluid (2). From this soap base, performance additives and the appropriate amount of additional oil are added to form a semi-solid or gel-like material.

Most soap-type greases are sheared, or “milled,” to form a consistent thickness throughout the material. The soap category of greases, along with the base fluid selection, will be discussed further in the section “Applications by Thickener Type.”

B. Grease being milled

Additives

In most cases, the grease base fluid and thickener do not possess all of the properties necessary to provide maximum performance in the applications in which they will be used. To enhance their properties, additives are incorporated. Many specific additives can be employed, but for the purpose of this text, general classes of additives will be discussed. They are:

- Antioxidants – As the name would suggest, antioxidants are incorporated to protect the grease from oxidative breakdown. Antioxidants allow the grease to last longer in use by preventing degradation of the fluid over time.
- Anti-wear and extreme pressure (EP) additives– Anti-wear additives are added to decrease the rate of wear by formation of sacrificial film to prevent direct metal-to-metal contact under light to moderate loads. Like anti-wear additives, EP additives also form a protective coating on a metal surface, but the coating chemistry can prevent damage to contacting surfaces under pressure conditions where anti-wear additives are inadequate.
- Corrosion inhibitors – These additives are added to enhance the grease’s ability to protect metal surfaces against corrosive attacks by rust or surface active additives that are incorporated into the grease and can be aggressive toward certain metal types.
- Tackifiers – Another common additive is a tackifier, which makes grease stickier, thus providing increased adhesion to metal surfaces under conditions of high impact and water contact.

The components that make up a grease formulation play an important role in how it is characterized. The previous discussion should provide a guide to help a user better understand grease types and how to choose the proper grease for a given application.

Applications by Thickener Type

Greases are usually marketed and recognized by thickener type. The OEM or manual often will specify that a grease of “X” thickener type is needed for an application. The three basic categories of greases are insoluble solid-thickened, polymer-thickened and soap-thickened.

Insoluble Solid-Thickened Greases

Usually greases thickened by insoluble powders, such as bentonite clay, are non-melt greases. In other words, they have extremely high or non-observable dropping points. These greases are typically used in high-temperature applications, such as kiln cars, ladle cars and furnace door bearings. (3) These greases only have fair to good mechanical stability, so they are not recommended for applications where high shear is inevitable. Because this thickener system is composed of solids, the grease can eventually leave behind heavy amounts of residue in applications where the base fluid is driven off by evaporation.

Typical applications for insoluble solid-thickened greases include: open gears, high-temperature applications, EP with high-temperature applications, rolling element bearings, high- and low-speed applications, heavy loads, shock loads, ball and roller bearings, high oscillatory motion, high vibration, and low-torque.

Polymer-Thickened Greases

Many polymer-thickened greases are oxidatively stable because they contain a thickener that is composed of organic polymeric materials similar to rubber. As described above, soap-thickened greases contain metals, and metals can act as oxidation catalysts that can lead to fluid degradation. Because polymeric thickener systems do not contain any metals, they have the ability to provide good continuous service in applications that are subjected to high temperatures or are exposed to oxidizing environments (3).

The most common polymer-thickened grease is polyurea. Polyurea greases have excellent shear stability. This means they have the ability to stay in place in applications operating at fairly high speeds and temperatures without having to be re-applied. For this reason, a very common application for this type of grease is small to medium roller bearings used in electric motors (4). Typically, these are closed systems where it is nearly impossible to inject additional grease after the motor has started to run. One of the negative aspects of polymer-thickened greases is that they are expensive to produce, which can raise significantly the price of the finished grease. An additional drawback from a production standpoint is that many polyurea thickeners use raw materials that are classified as hazardous materials (2). While the finished greases are not hazardous, precautions still must be taken to protect the production personnel during the manufacturing process.

In addition to roller bearings on electric motors, other typical applications for polymer-thickened greases include: moderate- to high-temperature applications, anti-wear/EP, electric motors, seal-for-life bearings, rolling element bearings, and constant velocity joints, such as in an automobile.

Soap-Thickened Greases

Soap-thickened greases are the most common type and have been proven to be the most versatile. Common soap types include lithium, lithium complex, aluminum, aluminum complex, calcium and calcium complex. Others include sodium and barium. Worldwide, lithium soap is by far the most common grease thickener system used. Soap-thickened greases can be formulated for almost any application. Depending on which metallic base is used, soap-thickened greases have been found to be very easy to work with, safe and versatile. Certain types of soap-thickened greases naturally possess properties such as rust protection, water resistance, shear stability, and good thickening efficiency with lower amounts of thickener than the other categories. With the help of additives, most soap-thickened greases can have acceptable to excellent EP performance as well as and oxidative stability (2).

Typical applications for soap-thickened greases include: electric motors, gearboxes, chassis, wire rope, chain, cable, food grade applications, high-temperature applications, EP, non-EP, and all purpose.

In-Service Condition Monitoring

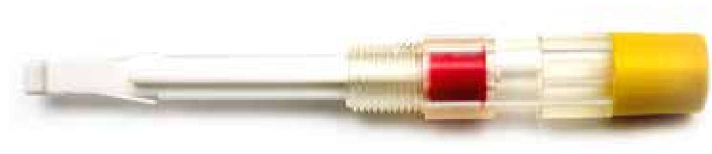
Analysis of in-service grease can provide useful information about the condition of the lubricant and the equipment in which it is installed. While condition monitoring of used fluid samples has been performed for many years and is mainstream, testing of semisolid grease samples is just now becoming more common, due to the development of new sampling and testing procedures within the industry.

Sampling

Grease has been found to provide more of a challenge as compared to contemporary lubricating fluids when collected for condition monitoring. This is mainly because it is not a liquid, so it does not flow as much in the application. This means that a sample collected from the improper location within a bearing may not truly represent the condition of the bearing or grease in the contact zone. In addition, many grease-containing bearings only contain a small amount of grease. Finally, due to spatial limitations, it is often difficult to collect a sample of grease from the equipment, especially when it is running (which is the recommended time to collect in-service lubricant samples).

Collecting enough sample of the used grease out of an application for testing can be challenging. The size and shape of the part, the re-lubing interval, and the quantity of grease used in the application can influence the amount that can be collected for testing. Each application can have more than one of these difficulties to overcome during sample collection.

The size of the recovered sample comes into play when the part that uses the grease is very small and the total quantity of in-service grease is less than an ounce. In this case, the part typically has to fail or be changed out in order to get a sample. Even then the sample size is normally very small, which limits the range of testing that can be performed.



C. Grease Thief Sampling Tool

The ASTM method for obtaining in-service samples of grease is ASTM D7718 Standard Practice for Obtaining In-Service Samples of Lubricating Grease. To collect grease samples from an application, available techniques include the use of a scraper, straw, spoon or any other utensil or tool that might aid in the removal of the sample from the application. On rare occasions, a sample can be collected when the grease is purged. Testing kits can be purchased that include a sampling device, such as the Grease Thief sampling tool made by MRG Laboratories.

Another challenge is purging a grease application when trying to collect an in-service or used sample. When adding grease to an application, in theory the used grease will be pushed out and can be collected. In the real world, sometimes there is no purged grease when grease is added to an application. There also is the chance that the amount put in, isn't the same amount that is purged. This has to do with how grease is used to lubricate and the design of the application or part itself.

The decision whether the sample should be tested in the field or at a laboratory is based primarily upon the need for immediate results, but secondarily upon what information is needed. It may seem like common sense, but a more thorough analysis can be performed in a laboratory than can be done in the field. Yet, if expediency is required due to equipment production requirements, a field screening test may be sufficient.

Shipping & Documentation

If sending the sample to a laboratory, it is important to select a proper container so that the lubricant does not leak out of the container. All sorts of containers have been used to ship grease samples, including plastic bags, plastic bottles, rags, cans and disposable plastic containers. It is better to use a new container, not one that has been cleaned. Reused containers – even when they appear clean – create the possibility of contamination.

Anytime a sample is collected, in order to aid in the most thorough analysis, there are various background requirements. As much background as possible should be collected from the sample and either recorded on the sample container or sent on a data sheet accompanying the sample. If a sample of the new grease is available, it is preferable to include a new sample of grease from the same batch number, as test data compared to the used sample can be very beneficial. The side-by-side comparison highlights the changes that occurred in the used sample. This type of sample is often referred to as a baseline sample.

D. This disposable plastic container is an example of the proper way to ship a grease sample. It is sealed with tape and labeled with the product name and an identifying number for reference.

Testing

Testing by Observation

Whether testing in the field or in the lab, these initial observations should be made and recorded first.

- **Color/Appearance** – Taking note of color changes of a grease while in-service is important. For example, if new grease is red or blue, but when sampled the grease appears green it would be significant. A visible change of this sort might suggest that the wrong grease was installed or that it has changed over time. To avoid installing the wrong grease, some users employ clear grease guns that allow the user to see the grease before installing it into the lubrication point. Common visible contaminants such as dirt, sand or wear particles can be small, while others can be large, such as pieces of rags or organic material like bark or grass. Water is another very common contaminant. When it is present, it might look like beads of sweat dripping from the grease. Water can also cause the used grease to look cloudy, runny or much lighter colored than when the grease was new.

E. Initial testing of grease should include physical observation of color, odor, consistency and environment.

- **Odor** – Another thing to note is the odor of the sample. Does it smell burnt? Does it smell like fuel? Does it have a chemical odor or food odor? Depending upon where the sample was collected, caution should be practiced when smelling the sample, as certain chemical contaminants may be noxious or dangerous to inhale.
- **Consistency** – A change in consistency is frequently observed when comparing sampled in-service grease with a new sample. For example, it may be gritty, tacky, smooth or runny. Grit can indicate particle contamination or wear particle generation. A grease changing from tacky to smooth or smooth to tacky can indicate contamination or chemical transition resultant of the end of useful life.
- **Environment** – Seeing exactly where the grease sample was collected and taking note of the environment around the application can be helpful in any evaluation. This can be accomplished by physically sending in the part containing the lubricant or a photo of the part. Photos of the surrounding environment are also very helpful as they may provide clues about contamination sources or operating conditions.

Field Testing

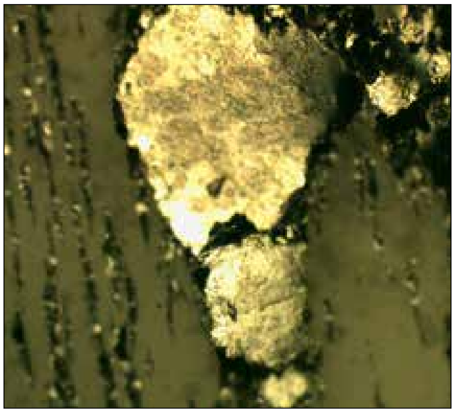
The SKF Grease Test Kit is a portable kit – about the size of a briefcase – that requires only a small amount of sample. The kit is designed to evaluate consistency between batches of purchased grease and to verify whether a particular grease is acceptable for a specific application per oil bleed characteristics. It can also be useful for root cause analysis.

Lab Testing

F

- **Physical**

A penetration test (F) can be used to check consistency in a grease sample. There are three different sizes of penetration tests: full size, ½ scale and ¼ scale. The ½ and ¼ scale penetration tests (ASTM D1403) are listed by their ASTM repeatability as less precise when compared to the full scale test (ASTM D217). As such, they should only be used when the sample size is limited.



G

Looking at the ferrous content of the sample (G) is most commonly done with analytical ferrography and ferrous density testing. This is accomplished using solvents to separate the grease components from the contaminants and then using a magnetic field to achieve further separation. The ferrous density test gives a ferrous metal concentration. Analytical ferrography involves microscopic examination of contaminants on a glass slide, allowing identification and categorizing of ferrous and nonferrous contaminants.

H

ASTM Subcommittee G is currently developing a rheometry test (H) for greases. A real advantage is that this new test uses an even smaller amount of sample than that of the ¼ scale penetration. Preliminary results look very promising for this technique, and it is possible that in the future this test may provide better data than the currently accepted standard tests.

The dropping point method (I), ASTM D2265, is useful for testing used greases for a couple different reasons. First, if the sample drops earlier than the published dropping point, then there typically is a contamination issue, such as lube mixing. Second, if the grease fails to drop, the test shows that the grease may have reached the end of its useful life.

I

Using a lightbox to examine the grease sample's appearance can be very useful. Foreign debris and inconsistencies can be seen when the sample is thinly spread out on the lightbox. This technique makes it easier to identify the type of debris. Pairing the lightbox up with a magnet can allow you to determine if the debris is ferrous.

- **Chemical**

J

Fourier Transform Infrared (FTIR) analysis (J) is a great test to be run on every grease sample. It becomes even more useful if there is a sample of the new grease that can be run and have its spectrum overlaid on the used sample's spectrum. This overlay allows the technician to immediately see organic contamination and lube mixing, as well as be able to determine the base fluid characteristics, thickener type, additive degradation (oxidation), and sometimes the ratio of thickener to base fluid.

K

In recent years, companies such as Spectro Inc. have introduced portable spectrometers, which have been certified by ASTM D7889 Standard Test Method for Field Determination of In-Service Fluid properties using IR Spectroscopy. When immediate onsite oil condition information is needed, these handheld devices make it possible for FTIR to be performed as part of field testing.

Elemental analysis (K) on a grease sample can be accomplished with many different methods. These methods include X-ray spectroscopy, inductively coupled plasma (ICP), spark emission spectroscopy, atomic absorption (AA), and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDS). In choosing the best analysis method for a given sample, considerations include particle size, sample size and sample degradation. Chosen correctly, the analysis method can provide very helpful results. Environmental contaminants, additives, thickeners and wear metals can be detected in the sample. In addition, it will be possible to identify if any lube mixing has occurred, and if so, what type.

Determining the water concentration of grease can be very important. Some of the environments that greases are used in are wet, but even when that is not the case, water can be a “surprise” contaminant that is detected in a sample. Some greases contain a minimal amount of water due to the cooking process, and some are even considered water-free. The simplest way to determine the water content in a grease sample is with Volumetric Karl Fisher. Other methods are more subjective, such as the crackle test, or require a larger sample size, such as ASTM D95 Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation.

Pressure Differential Scanning Calorimetry (PDSC) is a useful tool for measuring the oxidation stability of lubricating grease when run according to ASTM D5483 Standard Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry. However, a sample of unused grease will need to be available for comparison purposes. If an unused sample is unavailable, this test loses most of its value. The used and new samples are run under severe conditions that include elevated temperatures and pressurized oxygen.

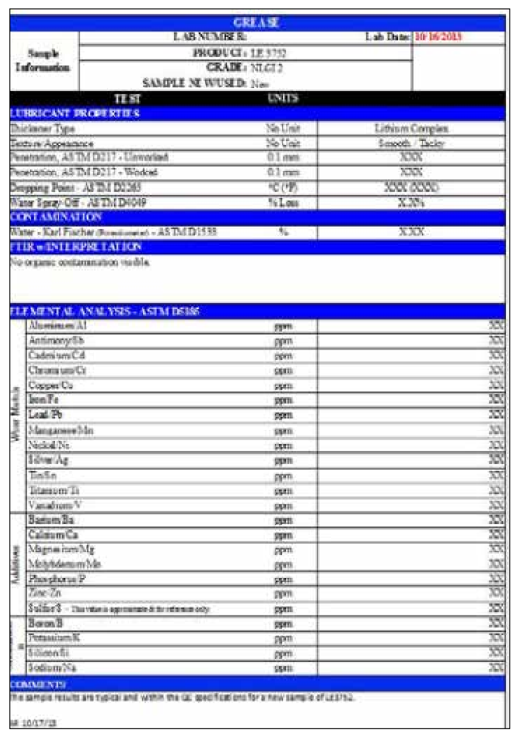
L

A recently developed grease test is the RULER test (L) ASTM D7527 Remaining Antioxidant by Linear Sweep Voltammetry, which measures the remaining phenolic and aminic antioxidants in a grease sample. This test also needs a new sample to run and use as a baseline for the used sample. If a new grease sample is unavailable, the value of this test is also greatly diminished.

Reporting

Collecting the sample and having the testing done on a sample of grease is important, but completely worthless if you do not use the resulting data. When the report is received, some action needs to be taken, even if that action is to continue the normal sampling and testing.

Grease is sampled and tested typically because a problem is suspected. If that is the case, one or more of the following actions should be taken: Increase lube frequency, decrease lube amount, replace seals, remove contamination sources, or change grease. In addition, sampling and testing of the grease should continue.



GREASE			
Sample Information	LAB NUMBER:	Lab Date:	10/16/2013
	PRODUCT: LE 3752		
	GRADE: NLGI 2		
	SAMPLE NEW/USED: New		
TEST		UNITS	
LUBRICANT PROPERTIES			
Thickener Type		No Unit	Lithium Complex
Texture/Appearance		No Unit	Smooth / Tacky
Penetration, ASTM D217 - Unworked		0.1 mm	XXX
Penetration, ASTM D217 - Worked		0.1 mm	XXX
Dropping Point - ASTM D2265		°C (°F)	XXX (XXX)
Water Spray-Off - ASTM D4049		% Loss	X.X%
CONTAMINATION			
Water - Karl Fischer ([illegible]) - ASTM D1533		%	X.XX
FTIR w/INTERPRETATION			
No organic contamination visible.			
ELEMENTAL ANALYSIS - ASTM D5185			
Wear Metals	Aluminum/Al	ppm	XX
	Antimony/Sb	ppm	XX
	Cadmium/Cd	ppm	XX
	Chromium/Cr	ppm	XX
	Copper/Cu	ppm	XX
	Iron/Fe	ppm	XX
	Lead/Pb	ppm	XX
	Manganese/Mn	ppm	XX
	Nickel/Ni	ppm	XX
	Silver/Ag	ppm	XX
	Tin/Sn	ppm	XX
	Titanium/Ti	ppm	XX
	Vanadium/V	ppm	XX
Additives	Barium/Ba	ppm	XX
	Calcium/Ca	ppm	XX
	Magnesium/Mg	ppm	XX
	Molybdenum/Mo	ppm	XX
	Phosphorus/P	ppm	XX
	Zinc/Zn	ppm	XX
	Sulfur/S - [illegible]	ppm	XX
	Boron/B	ppm	XX
	Potassium/K	ppm	XX
	Silicon/Si	ppm	XX
	Sodium/Na	ppm	XX
COMMENTS			
The sample results are typical and within the QC specifications for a new sample of LE3752.			

10/17/13

M. Sample grease analysis report

Conclusion

To summarize, used grease samples are typically very small in size, which makes it difficult to get a representative sample for testing. Traditional grease testing methods require large samples, so they are not well suited for testing used grease. New and improved tests are being introduced and implemented to properly test used grease, and the result is better, more meaningful data. With these recent innovations – and more on the horizon – condition monitoring of in-use grease is set to become an important tool for maintaining or troubleshooting a lubricated part or system.

Today, many plants are embracing equipment reliability-centered maintenance programs to establish safe minimum levels of maintenance, make necessary changes to operating procedures, and establish capital maintenance plans. The successful implementation of an RCM program leads to increases in uptime, efficiency and cost effectiveness. One of the cornerstones of an effective RCM program is a well-managed lubrication program, which includes in-service analysis of lubricants.

Analysis of in-service lubricants has been employed as a predictive tool to prevent unplanned maintenance as well as a root cause analysis after a failure to prevent recurrence. While in-service analysis has been commonplace for quite a few years, grease analysis has generally been considered of lesser value. Under the proper conditions, it is hoped that this paper provides useful insight into how to make in-service grease analysis a practical and useful part of world-class RCM programs.

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