Jeanna Van Rensselar / Senior Feature Writer

Grease particle evaluation

Closing in on a consensus definition of contamination and measurement standards.

KEY CONCEPTS

- Particle measurement in new grease is important for quality control.
- Part of the issue is arriving at consensus definitions for grease contamination, grease cleanliness and cleanliness grades.
- A particle-evaluation test has been proposed that combines ASTM D1401 and Hegman gauge test methods.

A JOINT WORKING GROUP BACKED BY THE EUROPEAN LUBRICATING GREASE INSTITUTE (ELGI) and the National Lubricating Grease Institute (NLGI) continues to make good progress toward defining a system for evaluating the size and number of particles in a grease sample. The group, which is working to evaluate robust methods for measuring these properties, gets together at both the NLGI and ELGI Annual Meetings in addition to other scheduled discussions throughout the year. This group has been assessing the feasibility of combining the results from two test methods as a particle evaluation system. This Webinar reviews background information, as well as the status of current testing, including an update on an industry round robin that is currently underway.

This article is based on an STLE University-sponsored Webinar presented June 5, 2014, by Joe Kaperick, who holds NLGI's Certified Lubricating Grease Specialist (CLGS) certification and is a customer technical service advisor for greases at Afton Chemical Corp. See Meet the Presenter on page 33 for details on the Webinar and how you can download a version.

MEET THE PRESENTER

Joe Kaperick

This article is based on a Webinar originally presented by STLE University on June 5, 2014. "Grease Particle Evaluation: Meaningful Measurement of Matter?" is available at www.stle.org: $39 to STLE members, $59 for all others.

Joe Kaperick, CLGS, is a customer technical service advisor for greases at Afton Chemical Corp. Joe began working for Afton Chemical in its St. Louis manufacturing facility as an analytical chemist in 1991 and moved to its Richmond, Va. headquarters in 1994. Joe received a master's degree in analytical chemistry from St. Louis University as well as undergraduate degrees in chemistry, fine arts and classical humanities. He has been in the industrial technical service area with a primary focus on grease since 1999.

Joe is a board member of NLGI and has been recognized as a Certified Lubricating Grease Specialist™ by NLGI. He is also a member of STLE, the chair of the joint NLGI/ELGI Grease Particle Evaluation Working Group and the chair of Section G.01 Chemical and Laboratory Tests for ASTM. You can reach Joe at **joe.kaperick@aftonchemical.com**.

WHY PARTICLE MEASUREMENT IS IMPORTANT

Abrasive particles can shorten bearing life by increasing wear. And agglomerated thickener particles can cause excessive noise and vibration—this is especially important in smaller applications such as CD and DVD drives.

The goal is to measure particles in a grease sample—for quality control and to ensure that the grease is clean enough for the intended applications. Sources of contamination during the manufacturing process include thickener agglomerates, solid additives and even dirt. But particle measurement, through condition monitoring, is just as important for used grease, which can become contaminated with wear metals, dust and dirt.

DEFINITIONS

The Grease Cleanliness Working Group was formed in 2004 as a joint working group of the ELGI and NLGI. It is now known as the Grease Particle Evaluation Working Group.

The group developed the following definition of grease contamination: *contamination by particulate matter that has the potential to do damage in a particular application*.

The types of particulate contamination in this definition include scale and wear metals from the manufacturing process, burned soap and additives, dirt and sand. Given the definition, the early consensus was that the three most useful criteria were the number, size and hardness of the contaminant particles.

An article in the April 2006 issue of TLT, Clean Grease is Not an Oxymoron by Paul Bessette, proposes the following definitions:

- **Commercial grade.** This is grease as delivered from the manufacturer. It may or may not be filtered. The level of particulate contamination is not determined and is highly variable.
- **Clean grease.** The grease has passed through a nominal 125-micron filter, and residual particulate contamination is sufficiently low to permit satisfactory grease dispensing. The determination of residual levels of particulate contamination may not be warranted.
- **Super clean grease.** The grease is filtered at high pressure to render the grease free from all particulate contamination larger than 35 microns and no more than 1,000 particles per cm^3 in the 10-34-micron range. Super clean greases require special packaging to maintain cleanliness.
- **Ultra clean grease.** The level of contamination in the 10-34-micron range is no more than 500 particles per cm^3. Lower limits may be set based on customer demands.

MEASUREMENT METHODS[1]

- **The DIN 51825 specification.** This is a gravimetric rating that measures the amount of particles greater than 25 microns per kilogram of grease measured by DIN 51813, with a maximum of 20 mg/kg.
- **DIN 51813.** 500 grams of grease are passed through a 25-micron mesh. The residue is collected from the screen, and the grease portion is dissolved. The remaining portion is passed through a filter, and particles of 25 microns and larger are weighed and reported. A passing result is less than 20 mg of particles greater than 25 microns per kilogram of grease. The total allowable

Particle measurement, through condition monitoring, is just as important for used grease, which can become contaminated with wear metals, dust and dirt.

particulate is similar to oil with an ISO cleanliness code of 23/19. A variation of this test involves dissolving 20 g of grease and filtering the solution through a 1.2 micron filter. The weight of solid particles per kilogram is reported.

- **Def Stan 05-50 (Part 39).** This is used to measure graphite particles present in grease. A sample of the grease is boiled under reflux with a solvent. The mixture is then filtered through a sintered glass crucible and the solids retained are weighed. Sintered glass crucibles are available (to BS 1752) in a range of pore sizes—typically from 160µm to 5µm.
- **MIL-G-81322.** This is used to rate aircraft grease. It allows less than 1,000 particles per cm^3 for 25-74 micron particles, and no particles larger than 75 microns. The total particulate is similar to oil with an ISO cleanliness code of 22/19.
- **MIL-G-81937.** This is used to rate ultra clean instrumentation grease. It allows less than 1,000 particles per cm^3 for 10-34 micron particles, and no particles larger than 35 microns. The total particulate is similar to oil with an ISO cleanliness code of 19/16.
- **U.S. Federal Test Method (FTM) 791 Method 3005.4.** Residual grease contamination is determined by sizing and counting the contamination in 1 cm^3 of grease placed in a rectangular template supported between glass slides.
- **Crompton (Jim Horner).** 250 ml of grease is dissolved in 1,000 ml of clean solvent and filtered through a 200 mesh filter. Deposits in the filter are determined as mg/100 ml with a value greater than 3.0 mg/100 ml being unacceptable.
- **BeQuiet+.** This is an automated test stand that injects grease in a deep groove ball bearing (type 608 at 1800 RPM). The bearing is run in for 15 seconds, and then tested with an acceleration transducer measuring three energy ranges. High bands (1,800-10,000 Hz) are the best measure of disturbances, which may be caused by particles in the grease.

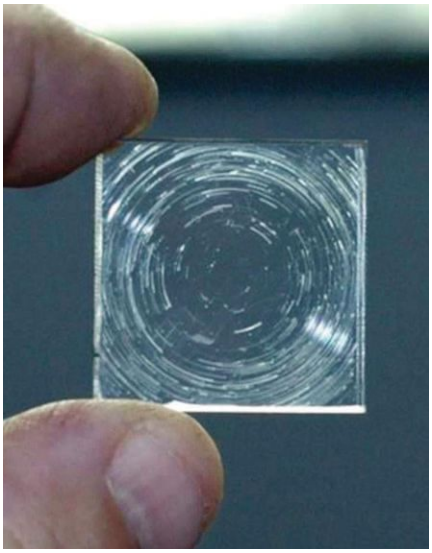

Figure 1 | **ASTM D1404: Deleterious particles.**

- **ASTM D1404: Standard Test Method for Estimation of Deleterious Particles in Lubricating Grease.** The number of arc-shaped scratches are counted that appear on highly polished acrylic plates when 0.25 g of grease is forced between the two plates and stressed under 200 PSI [1.38 MPa] pressure in a circular motion for 30 degrees relative to each other (*see Figure 1*).
- **Hegman gauge.** This is a proposed count and size measurement—with no standard method defined for grease (*see Figure 2*). ASTM D1210 cites the use of the Hegman gauge by the paint industry. Proposed parameters include:
  - A: Clean (0 particles <100u)
  - B: Moderate dirt (1-5 particles <100u)
  - C: Dirty (>5 particles <100u).
- **ASTM D1210.** "Standard Test Method for Fineness of Dispersion of Pigment-Vehicle Systems by Hegman-Type Gage" and *ASTM D1316 Standard Test Method for Fineness of Grind of Printing Inks by the NPIRI Grindometer*. Both methods are available in ASTM, Volume 6.[2]

PROPOSED DEFINITION OF GREASE CLEANLINESS

The following is a proposed two-part definition of grease cleanliness. The types of particulate contamination in this definition could include scale and wear metals from the manufacturing process, burned soap and additives, dirt and sand.

1. A measure of the absence of particulate matter that has the potential to do damage in a particular application
2. A measure of the suitability of a grease to be used in such applications in which contamination by particulate matter would cause damage to the pieces in contact with the lubricant; the extent of contamination can be measured by examination of the size, number and/or hardness of the particulate matter.

Figure 2 | **The Hegman gauge test.**

A COMBINED CLEANLINESS RATING SYSTEM

A rating system was proposed that would combine the ratings from both ASTM D1404 (number of hard particles) and the Hegman gauge (number and size) (*see Figure 3 on page 36*).

- **ASTM D1404.** Recall that this is an account of the number of arc-shaped scratches that appear on highly polished acrylic plates, separated

UNLOCK FORMULATION FREEDOM WITH ONE REVOLUTIONARY MOLECULE.

Introducing Elevance Aria™ WTP 40.

A new lubricant base stock that uniquely bonds synthetic PAO and ester functionality for superior formulation flexibility and performance to extend equipment life.

With the high viscosity, shear stability and elastomer compatibility of Elevance Aria™ WTP 40, you can formulate the low-foaming, wear-resistant solutions your customers need in high-viscosity gear and compressor lubricants.

Learn more and request a sample at elevance.com/aria

Elevance
RENEWABLE SCIENCES®

Rating	# of particles
1	0
2	2
3	5
4	10
5	20
6	80
7	160
8	320
9	640
10	1,300
11	2,500
12	5,000
13	10,000
14	20,000
15	40,000

Figure 3 | **Suggested cleanliness rating scale.**

by grease and placed under stress. The rating:
  - 1 = less than 10 scratches
  - 2 = 10-40 scratches
  - 3 = more than 40 scratches.

- **Hegman gauge.** The procedure used in the paint industry to measure pigment distribution starts with a narrow trough precision-milled from 0-100 microns. A small amount of grease is forced into the length of the trough using a stainless steel gauge held at a 45 degree angle. If particles in grease are larger than the depth of the trough at any given point, they are caught by the gauge and dragged along leaving a track in the grease. The size of the particle is determined by where the track stops. The total number of tracks correlate to the total number of particles. Particles will leave tracks behind in the grease until they can roll under the straight edge of the gauge. Due to differing widths of the troughs, the results need to be normalized to the width of the trough or the volume of the grease.

Greases would then have a cleanliness rating between 1A (very few particles, small, not very hard/damaging) and 3C (many particles, larger, hard with potential for damage).

EARLY ROUND ROBIN

The total of five samples included three standard lithium greases and two that were spiked with calcium carbonate and MoS2. The solids were small in size (<10 microns) and the results were mixed. All involved agreed that more realistic contamination was necessary.

They then used ISO 12013-1, A4, Coarse Test Dust, which ranges from 0-180 microns. ISO Coarse Test Dust is identical to SAE Coarse Test Dust produced by Powder Technology, Inc., prior to formation of this standard. The basis for acceptable particle size limits specified per ISO 12103-1, A4 was derived from analysis of eight batches of SAE Coarse Test Dust manufactured between 1992 and 1994.

DISCUSSION

The group noted that there is likely a shelf life on the acrylic material composing the plates for D1404. Some of the acrylic plates were old and this could be a source of variability. Another variable that needed to be controlled was the temperature at which the Hegman test was conducted. They corrected for these and other variables, ran the test again using more real world samples and adjusted the ratings scales.

SUMMARY OF RECENT FEEDBACK/ DISCUSSION

Going forward, the group decided to use the name Grease Particle Evaluation Working Group to avoid the potential negative connotations of *grease cleanliness*. They also decided that they needed more feedback from OEMs and end-users and that the ultimate goal is to align OEMs and end-users with a measurement tool that can be used for specifications. Then they took a realistic look at the pros and cons of the experiment:

Pros

- Possible uses are in the nuclear industry or used grease evaluation where sample size is limited.
- Some end-users seem interested.
- Having a tool for evaluation would be desirable.

Cons

- Bearing manufacturers and OEMs don't know what they want.
- The method would lead to everyone wanting the cleanest possible grease.
- It would add cost to the industry through marketing and the demand for the test.

GOING FORWARD

The group will conduct another round robin using eight commercial samples with an eye toward reproducibility and identifying potential issues and areas for improvement. Kaperick is looking for volunteers. If interested, please contact him via email at **joe.kaperick@aftonchemical.com** with the following information:

1. Which methods you are able to run—Hegman gauge, scratch test or both
2. The size of your Hegman gauge—depth, length and width
3. Your shipping address and contact person (for sending samples). TLT

Jeanna Van Rensselar heads her own communication/public relations firm, Smart PR Communications, in Naperville, Ill. You can reach her at ***jeanna@smartprcommunications.com.***

REFERENCES

1. Based on the presentation at the 70th Annual Meeting of NLGI. October, 2003, "How Clean is Your Grease," by Nicolas Samman of Petro-Canada and Mike Johnson of Noria Corp.
2. Canadian Government Standard, 3-GP-0, Method 76.1a was withdrawn in 1986 with FTMS 3005.4 recommended as a replacement.