# WHAT DOES THE DROPPING POINT TEST REALLY MEAN FOR GREASES: AN IN-DEPTH LOOK

Nearly 50 percent of mechanical component failures result from wear and friction underperformance of the applied lubricants or simply just the mechanical component reaching the end of its lifespan [1].

Therefore, the constant, unwaning performance of lubrication is necessary for a reduction in these mechanical component failures. Mechanical components include nuts, bolts, bearings, and belts, components which are used extensively in internal combustion engines (ICEs). The main lubricating agent used in ICEs is grease, a semi-solid, which in this case includes both motor oil and lithium grease. These greases are combined with additives and thickeners to improve lubrication properties and enhance other desirable properties. Lithium-based greases are used to lubricate ball bearings and bearing systems, while engine oils (motor oils with additives) are used to lubricate the crankshaft, camshaft, and rocker arms of an ICE [2]. In these internal combustion systems, components and surfaces are constantly moving and sliding in contact with each other. The surface area where these components or surfaces interact is called the contact surface and is observed in multiple areas of the engine, specifically the crankcase, piston and liners and the main and rod bearings [3]. Failure in the main crankshaft bearing can lead to engine failure [3]. Efficient lubrication is thereby integral to the effectiveness and longevity of the engine. The applied lubricants must be able to function optimally in extreme conditions, as an ICE coolant operates at temperatures of approximately 195-220 degrees F (90.6-104.4 degrees C) [4]. These high operating temperatures require applicable lubricating greases to have a high dropping point.

The dropping point is an indicator of the heat resistance of grease and can be defined as the lowest temperature at which the phase of grease changes from semi-solid to liquid [5]. When this dropping point is reached, the efficiency of grease vastly decreases as the semi-solid grease loses its structure and desired viscosity [6]. Some greases can regain their original consistency when cooled, but not all greases share this property [6]. Therefore, the desired consistency and the inherent tribological properties of the original grease are lost and the altered grease may no longer be sufficient for maintaining a proper lubrication film. In short, exceeding the dropping point leads to a drastic depreciation in the quality of lubrication and the lubricant is rendered essentially useless. This newly formed product has a lower viscosity, which causes the grease to have a decreased adherence to the components of the lubricating system.

*Table – 1*

*Physical and Chemical Properties of Tested Greases. Adaptation from [12]*

| Sample | Dropping Point Test, ºC (ASTM D556) | 60 Strokes Worked Penetration Lower Limit, 10^-1 mm (ASTM D217) | 60 Strokes Worked Penetration Upper Limit, 10^-1 mm (ASTM D217) | Oil Separation Test, % (ASTM D6184) |
|---|---|---|---|---|
| LCG 01/18 | 257.3 | 265 | 295 | 0 |
| LCG 01/5 | 272.7 | 265 | 295 | 1 |
| Industry K | 295 | 280 | 310 | 2 |
| Industry S | 214 | 295 | 310 | 3 |

*Figure 1*

*Koehler High Temperature Dropping Point Apparatus*

*Reprinted from Koehler Instrument Company Inc. [10]*

Grease is desirable for lubrication due to its high viscosity, which effectively decreases friction and generates heat at the contact surface. Viscosity is a liquid's (or in this case, a semi-solid's) resistance to a change in shape or movement [7]. Decreasing sliding or movement proportionally decreases the force of friction. Therefore, semi-solid grease reduces the friction force more efficiently than lower viscosity grease, which results from exceeding its dropping point. This decreased viscosity is due to an increase in the movement of adjacent oil layers caused by a lack of structure, which is lost above a grease's dropping point [5]. These reduced tribological behaviors lead to reduced lubricating ability and increased wear on the contact surfaces; in other words, the deformation and removal of material caused by rolling or sliding. a process that involves the interactions between surfaces and the removal and deformation of material caused by rolling or sliding [8]. This wear causes a decrease in the longevity of the contact surfaces. The dropping point is therefore fundamental to both the performance of the grease and the longevity of the surfaces to which it is applied.

The test method utilized for the determination of the dropping point is standardized by the American Society for Testing and Materials (ASTM) and given by the designation D2265. This test method yield results useful for identifying grease as to a type and for establishing and maintaining benchmarks of quality control [9]. In this test, a grease sample is heated and observed until a drop of material falls from the cup to the bottom of the test tube. The Koehler Instrument Company Inc, apparatus for the Dropping Point Test is shown below in Figure 1 [10]. This instrument conforms to ASTM D2265 and D4950 specifications and features a six-sample testing capability [10]. It additionally features a microprocessor programmable for high accuracy temperature control, reaching temperatures of up to 400 degrees Celsius [10]. Using the viewing window provided in the instrument, the moment when the material falls into the bottom of the test tube can be clearly seen and the reading on the sample thermometer can be recorded to the nearest degree. This temperature reading is designated as the experimental dropping point. Simultaneously, the temperature displayed on the temperature display screen of the instrument is recorded to the nearest degree and a third of the difference in the two temperature readings is added to the observed value and taken as the dropping point [9]

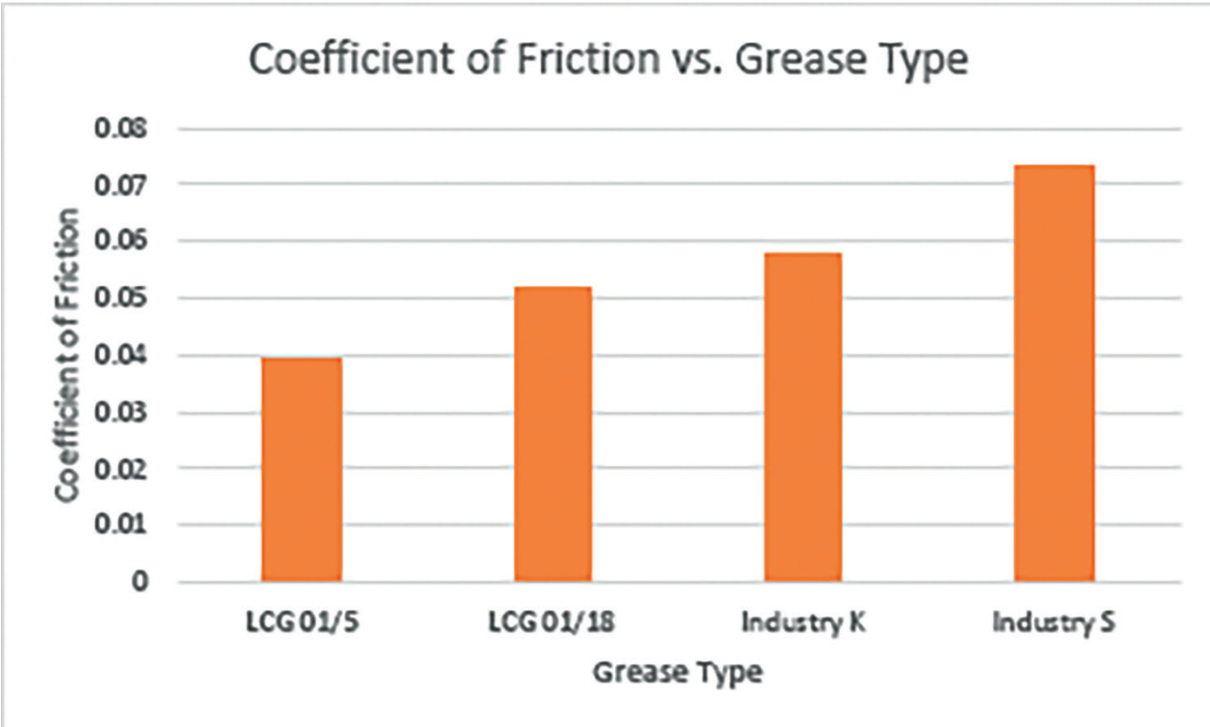


Figure 2

Coefficient of Friction vs. Grease Type. Adaptation from [12]

This test is quite simple from a laboratory experiment standpoint, but the recorded dropping point provides valuable insight into the temperature properties of grease and the proper applications it can be applied for. Greases with relatively high dropping point temperatures (in the range of 170-180 degrees Celsius) are highly sought after in commercial vehicle applications, such as in internal combustion engines in cars and tractor engine lubrication [11]. Meanwhile, greases with relatively low dropping point temperatures (in the range of 90-100 degrees Celsius) can be used for valve, conveyor, and spring lubrication, as well as many other purposes due to the multipurpose nature of these greases [11]. Therefore, different dropping points correspond to different uses, and higher dropping points are sought after for use in engine lubrication due to an engine's typically high operating temperature (where coolant reaches approximately 90.6-104.4 degrees C) [4].

Research has shown that specific nanoparticle additives yield improved dropping point temperatures. This is seen in the research of N. M. Ramli et al., which studied the synergistic effect of molybdenum disulfide (MoS2) and butylated hydroxytoluene (BHT) in lithium complex grease [12]. The greases tested included two industrial greases, denoted Industry K and Industry S, which were purchased and two lithium complex greases, which were produced with different weight percentages of MoS2 and BHT. The first grease, denoted LCG 01/18, contained only the BHT additive, with a weight percentage of 0.13% [12]. The second lithium complex grease produced contained both BHT and MoS2 and was denoted LCG 01/5 [12]. Both additives were held at 0.13% weight, meaning the grease contained 0.13% wt BHT and 0.13 % wt MoS2 [12]. These greases were then tested for their tribological properties, including dropping point, worked penetration, and oil separation according to the ASTM standards D556, D217 and D6184, respectively [12]. The results of these tests are shown below in Table 1 and a vast improvement in dropping point in the nano-additive greases with respect to the Industry S grease can be seen [12]. However, the Industry K grease beat out all the other tested greases when only considering the dropping point.

Although the Industry K grease was observed to have the highest dropping point, the improvement in dropping point from Industry S grease to the two lithium complex greases is large and is greatest in the LCG 1/5 grease. It was additionally found that the LCG 1/18 experienced the lowest coefficient of friction when tested using the four-ball test, which examines the wear scar on a surface lubricated by the grease being tested. The result of the test is shown in Figure 2 below, which plots the coefficient of friction versus grease type [12].

Due to the observed reduction in the coefficient of friction, and heightened dropping point in the LCG 1/18 grease, it was concluded that the two additives (MoS2 and BHT) have a synergetic effect and vastly improve all tribological parameters tested, specifically in terms of dropping point [12]. This finding shows that these two additives, when included in the same grease thickener, can improve chemical and physical properties more effectively than these additives can achieve alone. Thus, the MoS2 additive independently improves the dropping point, as shown by LCG 1/18, as well as synergistically with BHT, as shown by LCG 1/5.

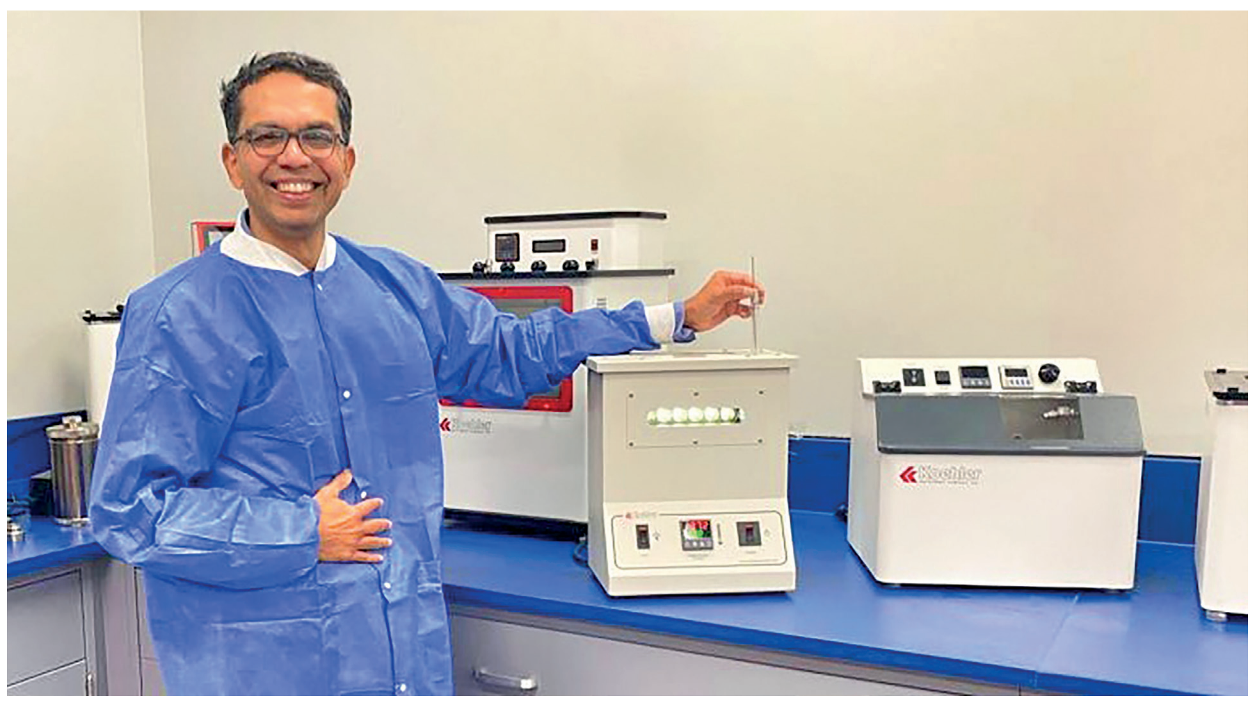

*Figure -3*

*Dr. Raj Shah, explaining the dropping point and other grease tests during the NLGI training course held at Koehler Instrument Company's HQ in NY*

*Table – 2*

*Formulated and Industrial Grease Characterization. Adapted from [1]*

| Sample | Droppin g Point (º C) | Volume resistivity (Ω*cm) | Contact Resistance (μΩ) |
|---|---|---|---|
| Lithium Complex Grease (LCG)- Base grease | 235 | 3.4E+11 | 29.3-29.5 |
| 4 Percent Weight tungsten disulfide (4% WS2) | 274 | 3.21E+11 | 29.3-29.6 |
| 4 Percent Weight ionic liquid -polyanilini/tungsten disulfide (IL-PANI) | 267 | 2.91E+11 | 29.3-29.7 |
| 4 Percent Weight ionic liquid -polyanilini/tungsten disulfide with 10 Percent Weight tungsten disulfide (4% PW10) | 269 | 1.9E+11 | 29.3-29.8 |
| 4 Percent Weight ionic liquid -polyanilini/tungsten disulfide with 30 Percent Weight tungsten disulfide (4% PW30) | 271 | 790000000 00 | 29.3-29.9 |

These additives are a preliminary step to improving both the tribological characteristics of lithium complex grease and its efficiency in lubricating mechanical systems in automobiles.

Similarly, research into tungsten disulfide additive in lithium complex grease also yielded improved dropping point. Tungsten disulfide (WS2) is a layered solid lubricant often used as a solid or fluid lubricant and is preferable over molybdenum disulfide and graphene due to its better conductivity [1]. Research by Yanqui et al. investigates the use of ionic liquid-polyaniline/tungsten disulfide (IL-PANI/WS2) composite in lithium complex grease. The IL-PANI/WS2 additive was tested against lithium grease containing only one of the additives (grease containing only IL-PANI and a separate grease containing only WS2) and tribological characteristics of the different greases were compared [1]. It was determined that the greases containing both additives (IL-PANI/WS2) exhibited better antiwear performance, electrical conductivity performance, and an improved dropping point [1]. This property of increased dropping point is shown in Table 2, where the lithium grease containing 4 % wt. WS2 was observed to have the highest dropping point [1]. The antiwear performance was evaluated using steel-steel and copper-copper fraction pairs lubricated by each grease [1]. A scanning electron microscope was then used to examine the surface and the wear scar on the steel and copper plates [1]. Table 2 illustrates the physical and chemical properties of the tested greases and clearly illustrates an amplified dropping point as the percent weight of the tungsten disulfide increased in the IL-PANI/WS2 grease [1]. Also included in this table are contact resistance and volume resistivity. Contact resistance describes the resistance occurring when two conductors contact each other [13]. Grease is only slightly conductive, and a depressed contact resistance shows heightened electrical conductivity [1]. This trend is expected and was sought after when choosing tungsten disulfide in this experiment and is shown in the decreased contact resistance as tungsten disulfide percent weight increased in the grease composite [1].

This observed increase in dropping point proportional to percent weight of WS2 is explained by both tungsten disulfide and PANI having large specific surface areas [1]. The increased surface area hinders the flow of liquid molecules and thereby delays the liquid from dropping out of the grease. This synergetic effect of WS2 and PANI represents a viable option for additives to improve dropping point while simultaneously improving additional tribological properties, such as viscosity, antiwear, and corrosion.

Simply, the dropping point is the maximum temperature at which grease retains its structure and quality. Knowing this temperature is important in deciding when a certain grease can be applied and utilized in a mechanical component system, such as an internal combustion engine. While not applicable for ICEs currently, by integrating additives such as tungsten disulfide and carbon nanofibers , greases with low dropping points containing improved antiwear or anticorrosion properties may have future applications. Additionally, additives placed in greases that are currently used in ICE lubrication have yielded improved dropping point, wear resistance, and viscosity. Providing improved lubrication to these systems, these improved greases may reduce mechanical component failures and increase the longevity of the ICE and its components. Additionally, this test method is used for quality control in the industry across batches of grease. Therefore, the measurement of the dropping point of lubricating greases and its corresponding ASTM test method is still relevant in today's industry due to its use in quality control and its importance in determining the operating conditions of lubricants, especially for extreme temperature applications.


*Figure -4*

*The attendees during the NLGI hands on training course held at Koehler Instrument's Training lab, Nov 2022*

## Works Cited

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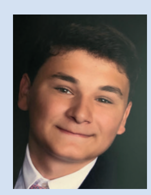
**William Streiber** and **Mrinaleni Das** are part of a thriving internship program at Koehler Instrument company and students of chemical engineering at State University of New York, Stony Brook, where Dr. Shah currently heads the External advisory board of directors.



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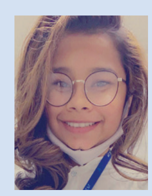

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