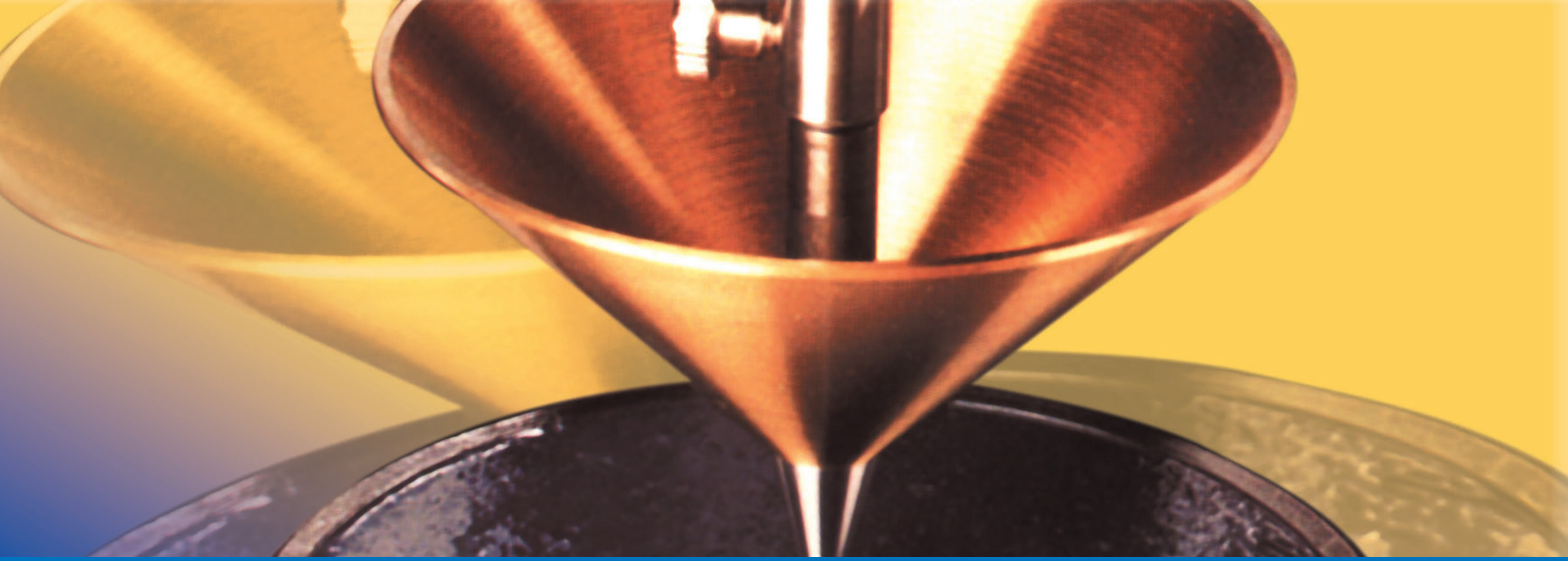

IMPORTANCE OF PENETRATION TESTING FOR STUDYING THE LONGEVITY AND EFFECTIVENESS FOR A WIDE VARIETY OF LUBRICATING GREASES

Tribology is the science of studying two interacting surfaces in relative motion and encompasses wear, lubrication, friction, and other related design aspects [1].

The consistency of a lubricating grease is one of its most distinguishing qualities. Lubricating greases often are formulated with additives and thickeners to influence its tribological behaviors. Some design aspects that are measured to determine tribological characteristics are performed using penetration, dropping point and anti-wear test methods, These properties give valuable insight on the characterization of a grease and its applications in industry.

As it pertains to grease and its industry, penetration is defined as the depth, in millimeters, to which a standard weighted cone sinks into the tested grease under prescribed conditions [2]. The penetration value gives information about the hardness of a grease, where a higher penetration value corresponds to a softer, less stiff grease. In an internal combustion engine (ICE), a too soft grease is undesirable, as the grease is more susceptible to migration [3]. This migration may remove the grease from the area of the system that requires lubrication, leading to heavy surface wear and high shear. Consequently, quantifying and understanding a grease's penetration value is vital for identifying its suitable applications and avoiding potential mechanical failure from unideal usage. A test method to determine the penetration of a grease has been developed and standardized by the American Society for Testing and Materials (ASTM) and is given the designation D-217 [4]. Similarly, roll stability is an indication of a grease's mechanical stability as it would act in service applications. As per the ASTM Method D-1831, roll stability is defined as the change in consistency of a sample after a specified amount of work in a test apparatus utilizing a weighted roller inside a rotating cylinder [5]. Roll Stability is quantified by the change in penetration of a worked grease before and after rolling. Ideally, the change in penetration before and after rolling should be zero, meaning that the consistency of the grease was unaffected by rolling or working. This would infer that the grease is able to hold its consistency despite being worked for extended periods of time under harsh conditions experienced in service applications. Therefore, little to no changes in penetration of a lubricating grease are highly sought after in the grease industry, as it shows stability, reliability, and sustainability of a grease over long periods of worked practical application.

ASTM test method D-217 details the importance of penetration and contains the procedure to quantify this tribological characteristic. This test method covers the measurement of the consistency of a grease via the penetration of a cone with specified dimensions, mass, and finish [4]. This test methodASTM D-217 evaluates the consistency of lubricating grease for half scale, quarter scale, and, full scale. The different testing scales are shown below in Figure 1, which captures Koehler Instrument Company, Inc's penetrometer accessories for ½ half and ¼ quarter scales. To run this test, fill the desired sized cup for the penetration test, ensuring no air bubbles, and level off the grease with the rim of the cup. Then, center the grease cup directly below the penetrometer, as shown in Figure 2, which. Figure 2 displays the Digital Penetrometer product from Koehler Instrument Company, Inc. [6]. This penetrometer, conforms to all ASTM, IP, ISO 9001 and related specifications for penetrometers [6]. The test is then run, and after 5 seconds the penetration value is determined. An average of 3 penetration values is taken as the true penetration value, which [4]. This designates the consistency of a grease [4].

Figure 1 Penetrometer accessories ½ scale (left) and ¼ (right) grease worker. Reprinted from [7]

Figure 2 Koehler Digital Penetrometer. Reprinted from [6]

ASTM test method D-1831 highlights how roll stability is an indicator of the sustainability of consistency and performance of a lubricating grease over extended periods of time. This test method covers the determination of the changes in the consistency of lubricating greases when worked in a roll stability test apparatus. The roll stability is used widely in specifications and is significant in that it shows a directional change in consistency that could occur in service [5]. Meaning, this test yields a general trend in penetration of the grease being tested. For example, if penetration increases after rolling (the penetration value is greater than before rolling), a positive change in consistency will be observed. It can then be concluded that the grease will harden when under stress in service applications. Prior to testing, the penetration of the grease is determined as per D-217. Next, 50 grams of unworked grease is uniformly distributed along the sides of the test cylinder shown in Figure 3, which shows the test cylinder, the roll stability apparatus, and the weighted roller. The roll apparatus, manufactured by Koehler Instrument Company Inc., conforms to ASTM D-1831 and related specifications, such as D-8022, which tests the roll stability of greases in the presence of water [8]. The weighted roller is then carefully placed inside the test cylinder and the cylinder is closed tightly. The test cylinder is placed in the roll stability apparatus shown in Figure 3 and subjected to rolling at 165 rpm for 2 hours ± 5 minutes [5]. This apparatus, manufactured by Koehler Instrument Company, Inc., conforms to ASTM D-1831 and related specifications, such as D-8022, which tests the roll stability of greases in the presence of water [8]. The cylinder is then subject to rolling, after which. The grease is removed from the cylinder and the worked penetration is quantified as per D-217 [5]. Note that the grease is worked in the worker after the 2 hours of rolling as per D-217. The change in penetration is then determined.

Figure 3 Koehler Roll Stability Apparatus Test cylinder and Weighted Roller (right) Roll Stability Apparatus (left). Reprinted from [8]

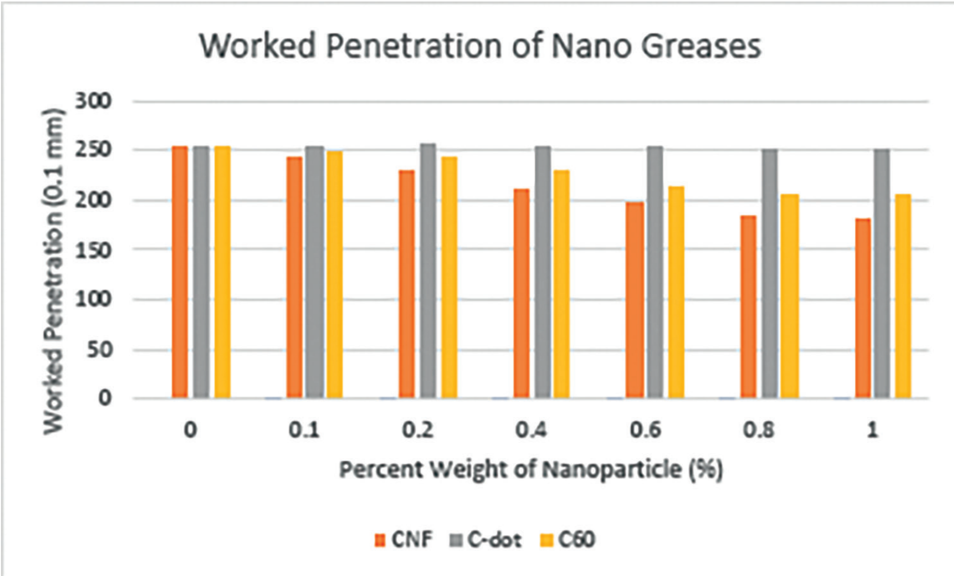

Figure 4. Worked Penetration of Prepared Nano Greases at Different Percent Weights. Adapted from [9]

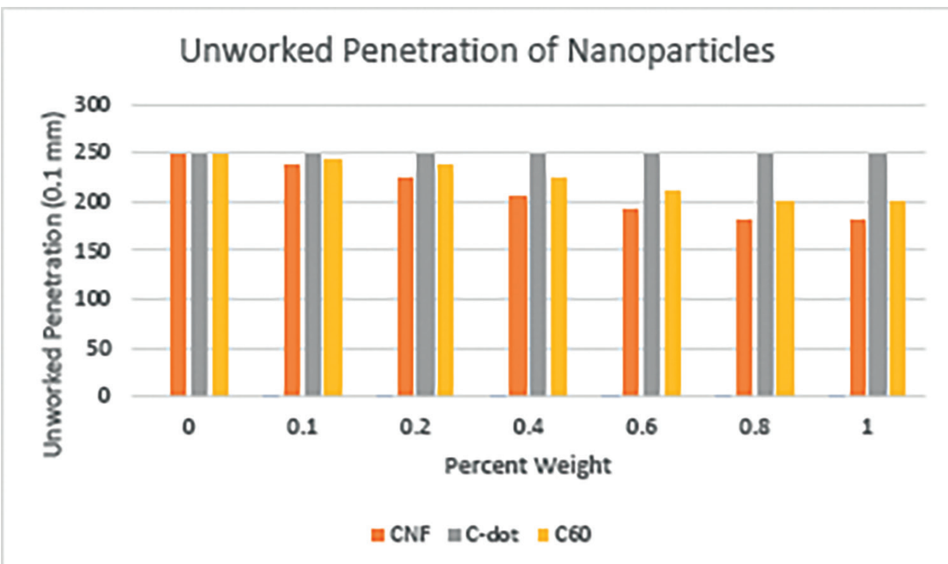

Figure 5. Unworked Penetration of Prepared Nano Greases at Different Percent Weights. Adapted from [9]

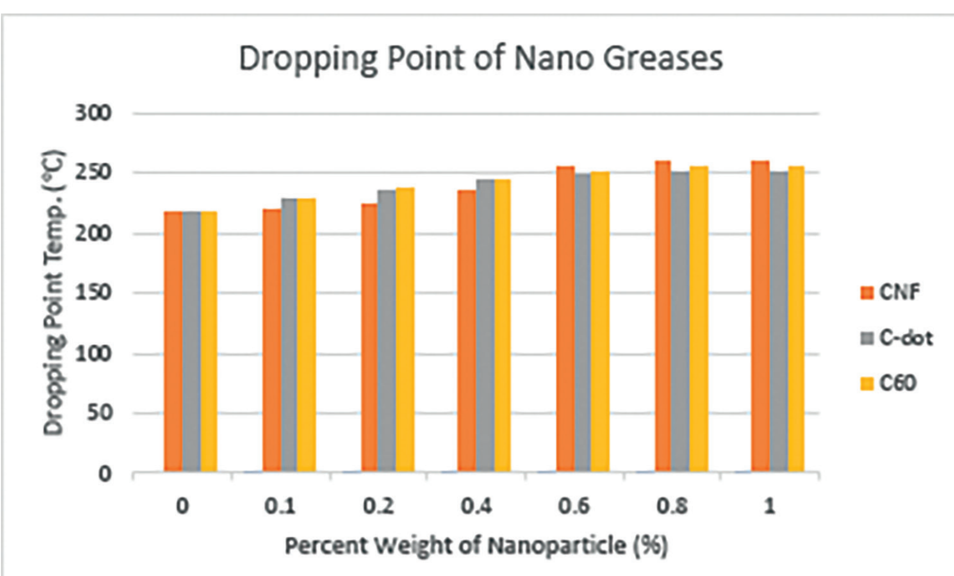

Figure 6. Dropping Point of Pprepared Nano Greases at Different Percent Weights. Adapted from [9]

Retention of consistency is integral to the quality of a grease and its longevity throughout service applications. This retention longevity is highly desired for a grease, and research into additives that enhance the retention of grease consistency areare heavily funded. An additive that has specifically received a large amount of attention is carbon nano fibers (CNFs). CNFs have been found to greatly improve the dropping point of a grease,, or the lowest temperature at which a drop of material falls from the grease [9]. Research by Sadeghalvaad et al. has found that CNFs additionally decrease penetration in both worked and unworked lithium grease. In this study, doped lithium greases with nano fullerene (C60), carbon dot (C-dot) nanoparticles and carbon nano fibers were studied for tribological behaviors [9]. These greases contained the respective nanoparticles at percent weights of 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 % [9]. Improvement in both penetration and dropping point was observed among all nano greases, with CNF nano greases experiencing the greatest increase in these properties [9]. The decrease in worked and unworked penetration is shown in Figures 4 and 5, respectively.

This heightened improvement in the consistency of greases containing CNFs is believed to be due to CNF's structure. Cylindrical in structure, these nanostructures layered with graphene sheets form fibrous networks that interweave and cross link with each other [10]. These interlinked fibers occur in the grease and improve its consistency, in turn increasing the force needed to penetrate it. This increased resistance to penetration directly decreases the penetration value of the grease when compared to the base lithium grease tested under the same conditions by ASTM D-217. This newly interlinked grease also experiences an improved dropping point for the same reason, as the grease is more resistant to deterioration and material is held in the grease at higher temperatures [9]. The improved dropping point is shown in Figure 6 below, which shows the dropping points of the base grease (0% wt) and the prepared nano greases.

As stated previously this increase is observed due to the interwoven fibers caused by the cylindrical structure of the CNFs. Therefore, not only do CNFs improve the dropping point of a lithium grease, but also the resistance of a grease to penetration. Moreover, carbon's hydrophobic behavior, working synergistically with the CNF's ability to become interwoven, improves the nano grease's resistance to water penetration and corrosion [11]. Thereby, this additive should be highly sought after as it improves many tribological characteristics required in lubrication, specifically for engine and bearing lubrication.

Conversely, one additive has been found to increase the penetration values of grease. Zinc dialkyl-dithiophates (ZDDP) were first used as a lubricant additive in the 1940's [12]. As V8 engines with overhead valves and increased compression ratios were introduced in the late 1940's-early 1950's, stresses on valve train components became apparent [12]. Valve train components include the camshaft, valves, rocker arms, and valve springs [13]. It was then that ZDDP was found to provide excellent antiwear properties [12]. This additive was used extensively throughout the late 20th century up until 1994, when it was discovered that ZDDP had a negative effect on the performance of the catalytic converter [14]. Used almost exclusively in engine oil, ZDDP had not been tested in lithium-calcium based grease (LCBG). Tiejun Shen et al.'s research on the ZDDPs effect of ZDDP on lithium-calcium-based greases, involved the formulation of two greases, along with a base grease. Grease A and Grease B were formulated using two distinct mineral oils containing a ZDDP additive at different percent weightsadditve. Both greases were formed from a 3:1 weight ratio of steric acid and 12-hydroxystererarc acid to mineral oil, followed by the addition of lithium and calcium hydroxide solution and saponification, with adjusted acidity via steric acid, followed by the introduction of the additive ZDDP at different percent weights [15]. Upon testing for the rheological properties of both greases, Grease B was found to have reduced viscoelastic functions, shear stress and mechanical stability as the percent weight of ZDDP increased [15]. Shear stress was found to decrease due to the breaking of fibrous links found in the grease, and the interaction of these fibers with ZDDP. ZDDP denied these fibers the ability to return to their interwoven state, and this decrease in shear stress was experienced greater in Grease B than A. Viscoelasticity refers to the exhibition of both elastic and viscous behavior of a substance upon the introduction of stress or deformation and is occasionally used to represent a substance's simultaneous liquid and solid behaviors, such as viscosity [16]. This reduction in mechanical stability is shown in Table 1 below, where unworked, worked and worked after roll stability penetration values are shown, as well as penetration change among both Grease A and B.

Note that an increasingly positive penetration change was observed, as the percent weight of ZDDP was increased for both test greases. This means that the penetration values increased after rolling and the grease softened during the roll stability test. Grease A experienced less of a penetration change as it was found the fibers formed a larger network, with more interlinking observed than in Grease B. Therefore, the breaking of these links took more time, and as Grease A and B were in the roll apparatus for equal amounts of time, Grease A deteriorated less than Grease B. It is important to note that for this experiment, a roll stability test adaptation was used, where the grease was rolled at 75 degrees C [15]. This heightened temperature was used due to specific service applications (engine/bearing lubrication)

Table 1

Parameters of Mechanical Stability for Lubricating Greases A and B. Adapted from [15]Adapted from [15]

Percent Weight ZDDP	0%	1%	2%	3%	4%	5%	6%	7%
Grease A pre-tested penetration	258.75	260.25	264	270.25	279	280.75	296.5	299.5
Grease B pre-tested penetration	270.25	286.5	295.25	298.75	299.75	306.75	308.75	311.75
Grease A penetration post roll stability	269.25	272.5	276.25	283.75	295.5	300.5	334.25	339.75
Grease B penetration post roll stability	292.75	324.5	327.75	332.5	336	350	357.75	373
Grease A penetration change	10.5	12.25	12.25	13.5	16.5	19.75	37.75	40.25
Grease B penetration change	22.5	38	32.5	33.75	36.25	43.25	49	61.25

and yielded the unstable nature of Grease B at temps above 60 degrees C. This instability was determined to be caused by the fibrous variability of the thickener of Grease B caused by ZDDP [15]. During the rolling process, both weak cross-linked fibers and thickener fibers were broken and ZDDP was absorbed [15]. This absorption of ZDDP denied the newly broken fibers the ability of restoration, leaving the consistency of the grease softer and more susceptible to penetration [15]. Thus, when compared to Grease A under the same test conditions, Grease B had the greater penetration values. Effectually, the introduction of ZDDP reduced the rheological and tribological features of the grease, causing advanced deformation of the grease. It is then determined that this additive makes the grease unstable in behavior and could cause issues in service, making it undesirable in the expensive, harsh conditioned lubricating systems of engines. This additive is thereby not to be used in grease as it depresses the performance of the grease and yields reduced quality of lubrication in both stability and tribological characteristics.

In all, these two additives had contrasting effects on the performance and quality of grease. While CNFs vastly improve the mechanical stability as well as the tribological characteristics of grease, ZDDP caused a deterioration in the quality and stability of the semi-solid. Penetration and roll stability are thereby influential indicators of the quality of a grease and its range of service applications. By understanding a grease's consistency and behavior over extended periods of stress, proper areas of implementation can be determined, and the grease can be classed as to a type. Therefore, the penetration and roll stability ASTM test methods are integral in both the classification of a grease as well as its quality control across batch production.

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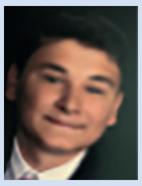
William Streiber is part of a thriving internship program at Koehler Instrument company and a student of chemical engineering at State University of New York, Stony Brook, where Dr. Shah currently heads the External advisory board of directors.



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Dr. Raj Shah teaching the NLGI course at Koehler Instrument Company's headquarters - Nov. 2022

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