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Comparison of Rheological Methods to Measure Grease Degradation

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Abstract: In a previous paper, methods that have been used to quantify grease mechanical degradation were compared, finding that crossover stress is a practical method for estimating the cone penetration value of a grease using a small sample. This paper covers techniques that have not generally been applied to modeling grease degradation and indicates their usefulness in characterizing the state of a grease. Three methods are examined, each using a different flow profile: rotation, oscillation, and normal force/extension. It is found that crossover stress is likely still the best choice for estimating cone penetration, and a fast, practical method is introduced here. In addition, a procedure for evaluating pull-off force is provided that describes some of the stretching behavior experienced by grease in a rolling contact; this method can also be used as an estimate of cone penetration. Finally, the applications of a "start-up yield" measurement are covered, providing details about the significance of wall slip as well as an independent way of estimating cone penetration.

Keywords: grease measurement; rheological test; grease consistency; grease degradation; crossover stress; cone penetration; pull-off force

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1. Introduction

Consistency is perhaps the most widely reported material property of lubricating grease. It generally refers to the suitability of grease for a wide variety of applications by describing its firmness, ability to form stable channels of lubricant, and tendency to stay in place despite externally forced motion. Measuring grease consistency [1] is achieved through the cone penetration method, where a cone penetrates into a cup of grease and the depth it reaches in 5 s is the result. A model is provided in Figure 1, with Equations (1)–(3) describing the cone area, A, as a function of penetration depth, Δ, in decimillimeters (dmm). Note that these numbers are approximate due to tolerances.

Due to the shape of the cone, the area is clearly a non-linear function of penetration depth [2]. In addition, the cup diameter also plays a role in determining the penetration value, especially when penetration values are high. These two geometric characteristics make cone penetration results somewhat difficult to accurately predict using alternative methods. Nevertheless, various authors have published equations for estimating cone penetration using measurements such as yield stress [2–5] and crossover stress [1].

In order to improve life prediction methods and perform remaining useful life estimates of grease, there is a need for techniques that can measure the state of a grease as it degrades so that degradation can be quantified and monitored. Generally, shear stability is understood to be a grease's resistance to consistency change from mechanical degradation and is calculated using methods such as the prolonged working method [6] or the roll stability method [7]. However, a large volume of grease is needed for these measurements, which is generally impossible for real applications.

$$A(\Delta) = \pi\left(\Delta\tan\left(\frac{\pi}{12}\right)\right)^2 \qquad \Delta < 150\ dmm \tag{1}$$

$$A(\Delta) = \pi(\Delta - 108)^2 \qquad 150 < \Delta < 455\,dmm \tag{2}$$

$$A(\Delta) = \pi(347)^2 \qquad \Delta < 150\,dmm \tag{3}$$

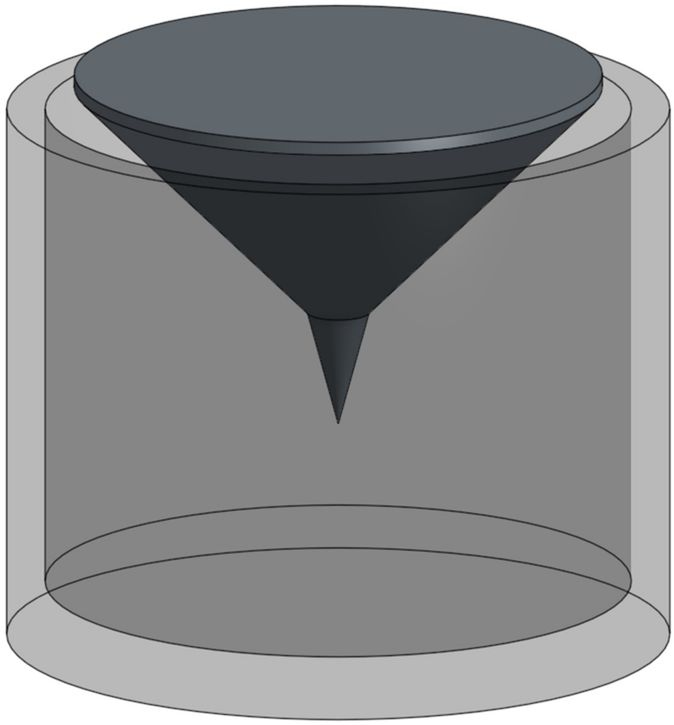


Figure 1. Cone penetration model using dimensions from ASTM D217 [6].

Many papers have been published over the years on techniques for measuring grease properties and using them to describe the state of a grease as it degrades. Examples of such techniques include yield stress measured in various ways [8,9], rheometer penetration [10,11], energy expenditure to reach the crossover point [12,13], and cone penetration [14]. Rheological techniques have the advantage of needing only a small sample to conduct measurements, though many of these tests were examined in previous work [1] and had important weaknesses. The factors that determine the overall quality of a test method may be a matter of debate, but one generally seeks a method that provides meaningful results with a simple procedure that is repeatable and reproducible. It was found that the crossover stress method meets these criteria, and even further evidence is provided here.

There are, of course, many other techniques that were not investigated in the previous study. Such techniques may involve rotational, oscillatory, and/or extensional motion with various different profiles, such as stepped flow, steady flow, and creep flow. From such tests, it is possible to obtain many different values that are generally a combination of a grease's material properties as well as a response to the test's specific inputs. Thus, obtaining meaningful measurements demands tests to be operated in a certain range and results to correlate with established meaningful characteristics.

A rheometer compression test was performed in a previous study [1], but the elongational response was not investigated. However, according to other studies, such as those by Achanta and Vargo [15,16], adhesion generally correlates with cone penetration. Therefore, a method was constructed based on the pull-off force concept used in various grease studies [15–19]. This type of test is quite simple to perform and can be conducted using equipment that is much simpler and cheaper than a rheometer.

Various definitions of yield stress exist and have been used to estimate grease consistency and structural degradation. This study covers one particular value obtained from a stress growth test at a very low shear rate. There are many issues (covered in Section 2.3) with considering the yield stress, but this measurement provides information about a balance of the properties of a grease. This balance of properties can be used to estimate cone penetration and also yields information about a grease's tendency to exhibit wall slip.

Overall, various methods of estimating cone penetration are useful to the grease industry because they will allow for the analysis of samples from real applications. Even in a broader context, many other industries (cosmetic, bitumen, petrolatum, etc.) use cone penetration measurements and any additional methods will also be useful there. However, it is not clear which techniques are best to use and various researchers use their own methods. Thus, this paper and the one preceding it [1] attempt to guide future researchers into adopting similar methods so that results may be compared. Some of those industries may also benefit from using these techniques to model degradation, as well as thermodynamics-based degradation models using these techniques.

2. Methods for Estimating Consistency

Though there are many types of rheological tests and values to select from each test, this study focuses on three methods with one singular value taken from each test. As will be noted in the Discussion section, other methods and values were considered, but the ones discussed were all found to work well as methods for estimating cone penetration values.

2.1. Crossover Stress

In previous work performed by the present authors [1], crossover stress was shown to correlate quite well with cone penetration. It can serve as a way of estimating cone penetration measurements using a much smaller sample than for the cone penetration test. Crossover stress is found by using an oscillatory amplitude sweep test.

There are many types of oscillatory rheological tests, but an amplitude sweep involves fixing a frequency and increasing the stress or strain amplitude. The choice of increasing the stress or strain value can make a difference due to the complex behavior of grease in oscillatory motion [20] and the way rheometers work in the two modes [21]. Therefore, this must be specified in the procedure. Typical results of an oscillatory strain sweep are shown in Figure 2.

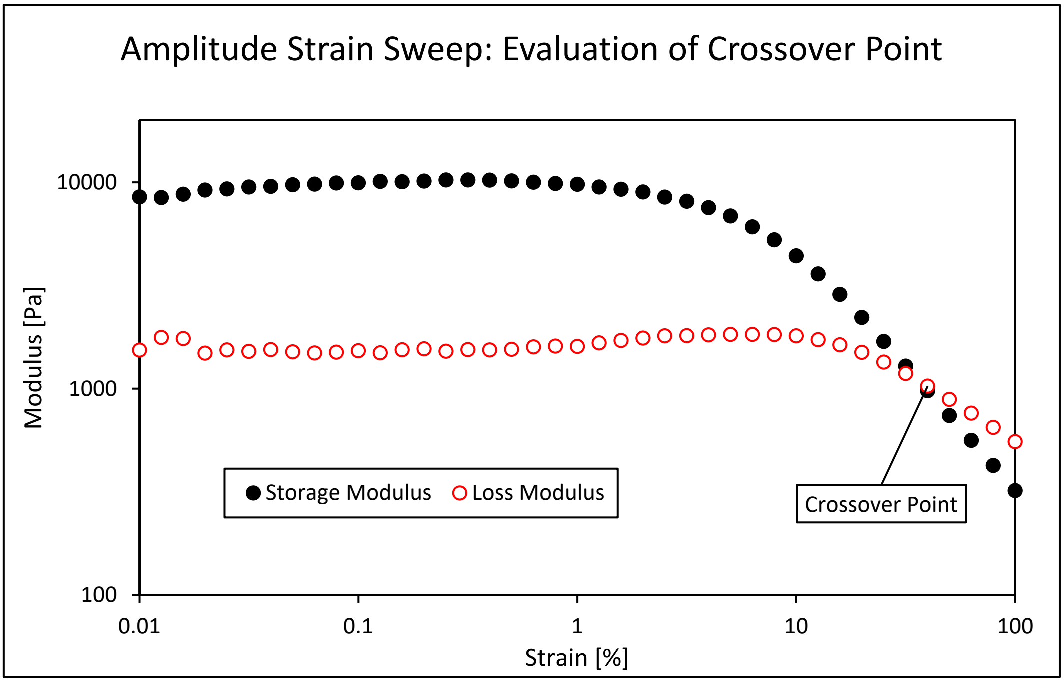


Figure 2. Typical results of an oscillatory strain sweep test with the crossover point marked.

Results are interpreted by calculating the complex modulus, G^*, using the ratio of maximum shear stress, τ_{max}, to maximum shear strain, γ_{max}, using Equation (4).

$$G^* = \frac{\tau_{max}}{\gamma_{max}} \quad (4)$$

This is then resolved into storage modulus, $G\prime$, and loss modulus, G'', depending on the phase shift, δ, between the test input and measured response according to Equations (5) and (6). The storage modulus represents the material's elastic response in phase with the input, thus quantifying the solid-like behavior. The loss modulus represents the viscous response, which lags 90° behind the input.

$$G\prime = G^* \cos(\delta) \quad (5)$$

$$G'' = G^* \sin(\delta) \quad (6)$$

Both storage and loss modulus are tracked as the amplitude of oscillations increases. When amplitudes are very low, the values are roughly constant, and this region is called the linear viscoelastic region. However, when amplitudes increase sufficiently, they both decrease and eventually cross over one another at the crossover point or flow point. This point corresponds to a phase angle of exactly 45°, signifying this is the point where behavior is exactly between a solid and fluid. The amplitude of stress corresponding to this value is the crossover stress.

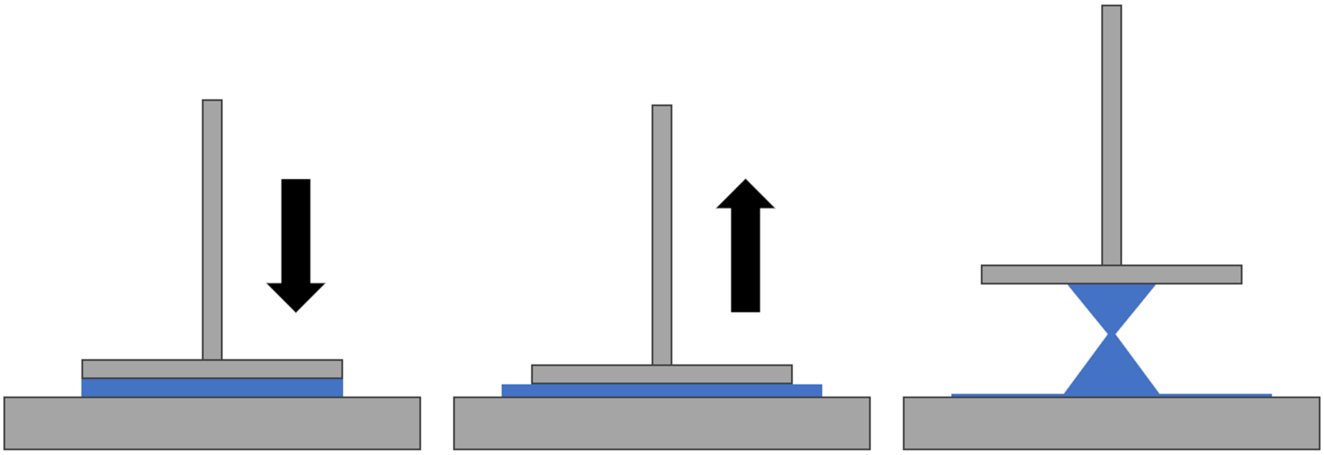
Within the linear region, properties are strongly dependent on sample preparation procedures. Hence, the degree of agitation of the sample before measuring it can significantly affect results. Because of this, a relaxation time must be imposed for measurements conducted within this region in order to allow the structure to rebuild itself. The crossover point, on the other hand, is not within the linear region and is less sensitive to small differences in sample handling. Nevertheless, it was found that surface roughness and measurement gap do not play a significant role in the linear range but can make a difference outside it due to wall slip [22]. Therefore, one should consider which region is more important and design the measurement procedure accordingly.

2.2. Pull-Off Force

Elongational flow is an important type of flow that is mainly used for high-viscosity substances such as polymer melts and concentrated solutions [23]. Such a flow profile demands a high apparent viscosity in order for the material to maintain adhesion to surfaces and cohesion within itself. This type of extensional flow gives rise to the concept of extensional viscosity, which is generally significantly different from the shear viscosity of a material. For Newtonian fluids, the ratio of extensional viscosity to shear viscosity (Trouton ratio) is 3. For non-Newtonian materials, there can be interesting behavior, such as shear-thinning but extension-thickening behavior [24]. Extensional viscosity, η_e, can be obtained by Equation (7), where force, F, filament radius, R, and strain rate, $\dot{\varepsilon}$, may change with time but surface tension, S, is a material property [25].

$$\eta_e = \frac{F}{\pi \dot{\varepsilon} R^2} - \frac{S}{R\dot{\varepsilon}} \quad (7)$$

Extensional flow behavior has been investigated for quantifying the adhesion and tackiness of grease using different kinds of indent–retract experiments [15,19], which are represented in Figure 3. Such experiments can measure the "pull-off force" as a measure of adhesion and possibly the thread length of a grease as an indication of tackiness [17]. However, the pull-off force is not a single value but is instead dependent on experimental conditions [18]. Notably, grease adhesion was found to be strongly dependent on the surface tension of the base oil, which is responsible for substrate wetting [15]. Therefore, other factors that affect wetting, such as material type and surface roughness, also play a role. There may also be important effects, such as cavitation and fibrillation, that influence results [26].



(**a**) (**b**) (**c**)

Figure 3. Schematic of the pull-off force procedure with (**a**) an initial trimmed gap of 1.5 mm, (**b**) a plate velocity reversal point with a 1 mm gap, and (**c**) the grease sample pulled into a thread once the plate has been raised sufficiently.

As a grease is subjected to this two-part (compression followed by tension) procedure, results of force vs. time will appear similar to Figure 4. Here, the peak tensile force is marked and is the pull-off force considered by this study. By convention, compression is treated as positive while tension is treated as negative, though for simplicity the pull-off force values reported in this paper are all reported as positive values.

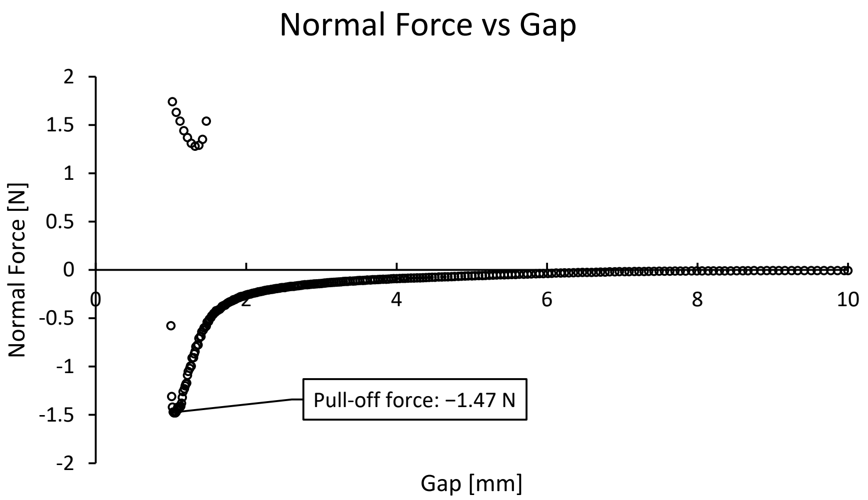


Figure 4. Plot of normal force vs. gap during pull-off force test with pull-off force marked; note that the peak tensile force is reported as a positive number.

Grease behavior in a pull-off force test can be complicated to describe since the extensional viscosity can change with extension rate and time. However, for a Newtonian fluid in a parallel plate plastometer, the force measured at instantaneous height, h, as plates separate at a constant velocity, U, is obtained by Equation (8) [23].

$$F = \frac{\pi}{2}\eta_e U \frac{R^4}{h^3} \tag{8}$$

The peak tensile force can be assumed to occur at the lowest point, but the effective radius value is not obvious since a significant amount of grease may be expelled from the measurement gap. Because grease is not actually pulled from the walls, the measurement of pull-off force as described here is more of a measure of cohesion (hence, extensional viscosity) than adhesion.

2.3. Start-Up Yield

While there are many ways of measuring yield stress [27–30], one must be consistent when comparing values, as different techniques can provide significantly different numbers [31,32]. Using a stress growth test is one method, and the term "start-up yield" (SUY) value will be used to refer to the peak shear stress value using this technique to avoid confusing terminology. A start-up, or stress growth, test involves shearing a sample at a fixed rate and observing its time-dependent behavior. When performed at a very low shear rate, one observes the gradual growth of stress within the sample, similar to a creep test. Especially for thixotropic materials with a yield stress, it is possible to have a maximum value of shear stress that corresponds to some combination of yield stress, thixotropy, creep behavior, and wall slip [29,33,34]. This maximum value is considered here as a parameter that may be used to estimate grease consistency. Figure 5 shows a plot of shear stress over time during a stress growth test with such peak values marked.

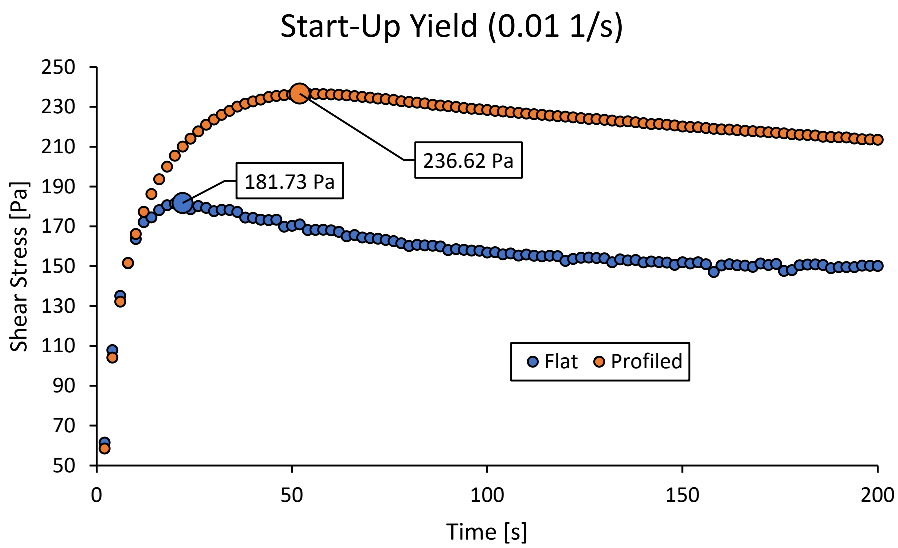


Figure 5. Start-up yield plots of shear stress over time comparing a flat plate to a profiled plate of the same diameter with peak values marked for each. This figure was obtained by shearing a grade 2 polyurea grease at 0.01 1/s with a 1 mm gap.

Even within the plot shown in Figure 5, there are numerous choices for what best resembles an appropriate yield stress value. One might consider the yield stress as the initial departure from linearity, the maximum stress reached, or potentially the steady-state stress value [30]. The maximum shear stress value reached is simply one choice that is investigated here and found to be a simple way of characterizing a grease. In fact, one can investigate wall slip behavior by comparing results obtained from a flat plate and a profiled plate (with no slip).

Performing a stress growth test at very low shear rates and small gaps is one of the simplest methods to observe a significant degree of wall slip in an experiment. Wall slip is characterized by the disperse phase migrating away (depletion) from system boundaries, causing an overall drop in measured apparent viscosity within a thin layer at the boundary [33]. Because this layer is generally orders of magnitude smaller than the size of particle structures within the sample, the sample–boundary interaction resembles solid–solid sliding and is often called slip. Though in some cases the sample does truly lose contact with the wall, the sample generally remains adhered to the boundary throughout.

Slip is influenced by the wetting characteristics of the materials involved. Roughening the surfaces reduces the contact energy between the sample and surfaces, improving wettability and possibly eliminating slip planes [35]. In addition, wall depletion is strongly dependent on the size of particles or flocs. Because floc size is lowest at low shear rate values, the effect of wall depletion is magnified at low shear rates [24]. For materials with a fibrous microstructure, including many greases, a smooth wall also disrupts the random orientation of fibers, partially aligning them with the direction of flow and leading to lower flow resistance.

Therefore, it is clear that measurement geometry characteristics play a large role in determining results using this method. Balan and Franco [22] investigated the influence of geometry on this method and found that gap and roughness play a major role in determining results. In a separate study, Delgado et al. [36] investigated the magnitude of the stress overshoot, finding that pre-shearing the sample accelerates steady state flow (eliminating overshoot) at the expense of altering the sample. Because this overshoot is a key parameter investigated here, it is clear that pre-shear should not be used when trying to measure it.

3. Equipment and Procedures

3.1. Sample Preparation

Grease samples were prepared starting with 11 different fully formulated greases with various thickener and base oil types ranging from grade 00 to grade 3. More details are provided in Table 1. These base samples were altered in various ways, including prolonged working with a grease worker, mixing in a grease worker with water, and mixing multiple greases together in a grease worker. There are two reasons for these alterations: obtaining more data points using only 11 greases and ensuring the correlations among all the methods remain valid despite mechanically degraded and contaminated greases. In total, 32 unique samples were measured for these experiments. However, because similar measurements were conducted for crossover stress in a previous paper [1], 45 additional points (making a total of 77 points) were used in the crossover stress portion of the results.

Table 1. Grease samples tested.

Sample Number	Thickener Type	Initial Cone Penetration	Procedures Used
1	Polyurea	269 dmm (Grade 2)	60 strokes, 100,000 strokes, 5% water, 10% water
2	Lithium Complex	420 dmm (Grade 00)	60 strokes, 300,000 strokes
3	Lithium Complex	369 dmm (Grade 0)	60 strokes, 100,000 strokes
4	Lithium Complex	346 dmm (Grade 1)	60 strokes, 100,000 strokes
5	Lithium Complex	297 dmm (Grade 2)	60 strokes, 100,000 strokes
6	Calcium Sulfonate	285 dmm (Grade 2)	60 strokes, 100,000 strokes
7	Lithium	292 dmm (Grade 2)	60 strokes, 100,000 strokes
8	Calcium	278 dmm (Grade 2)	60 strokes, 100,000 strokes
9	Aluminum Complex	270 dmm (Grade 2)	60 strokes, 100,000 strokes
10	Lithium Complex	247 dmm (Grade 3)	60 strokes, 100,000 strokes, mixed with Sample 1, mixed with Sample 2
11	Calcium Sulfonate Complex	285 dmm (Grade 2)	60 strokes, 100,000 strokes, 5% water, 10% water

One method used to obtain different cone penetration values using the same grease was to perform prolonged working in an ASTM standard grease worker in accordance with ASTM D217 [6]. Generally, each grease was measured after 60 strokes and 100,000 strokes using this cone penetration procedure.

Another method for obtaining different penetration values using the same grease was to mix greases with water and with other greases. To mix water into grease, both were measured on a scale and the appropriate weights added to a grease worker cup, which was then worked for at least 1000 strokes. Similarly, mixing two greases together involved measurements on a scale, the addition of the desired amount into a grease worker cup, and working for at least 1000 strokes.

3.2. Rheological Procedures

All rheological measurements were performed with an Anton Paar MCR 301 model rheometer using 25 mm diameter flat plates. One of these was a PP25/TG plate with a flat surface coated with chromium (III) oxide, while the other was a PP25/P2 D profiled plate with 0.5 mm tall pyramid-shaped profiling spaced 1 mm apart that was made of stainless steel. The flat plate was used for all measurements except where clearly stated otherwise. All measurements used the DIN 53018 R_{max} method for conversion between torque and shear stress values, not the 2/3 R method, which would yield significantly different values.

Unlike existing rheological standards and common practice [37], where there is generally some relaxation time given for grease, this study performed rheological measurements more similarly to the cone penetration method, where a grease is immediately subjected to the measurement after being worked in the grease worker. Before measuring, the grease was briefly mixed by hand in a container and then loaded into the rheometer. The top plate was lowered, the sample was trimmed at 0.025 mm above the measurement gap, and the measurement gap was set. Measurement procedures began immediately after this step, commencing within one minute of loading the sample.

Such a procedure would be generally inappropriate for measuring properties within the linear viscoelastic range since significant structural rebuilding occurs after a sample is loaded. However, with the possible exception of the start-up yield value, none of the properties measured in this study are within the linear region and they are less sensitive to structural rebuilding. Accelerating the procedure allows for more practical methods while maintaining high repeatability.

Crossover stress was measured using a strain sweep from 1% to 100% but was stopped after reaching the crossover point to speed up the procedure. Points were spaced according to the software used, with 10 points per decade. The measurement gap was 1 mm, and 1 Hz was selected as the frequency because of its use in previous relevant studies [28,36,38].

Pull-off force was measured starting with a 1.5 mm measurement gap. From this point, the top plate was set to lower at a rate of 0.1 mm/s for 5 s and was then set to turn around immediately and travel upwards at 0.1 mm/s. The peak tensile force was recorded and presented as a positive number.

The start-up yield was measured using a 1 mm gap and a constant shear rate of 0.01 1/s, similar to the shear rates from previous studies [22,32,36]. The test was set to run for 5 min but was stopped after the measured shear stress decreased from its peak value by approximately 10%. This was implemented to save time since some samples reached a peak value within 30 s of initiating the test.

Overall, all measurements were conducted at 25 ± 1 °C and were performed three times. The average value is presented with error bars corresponding to one standard deviation.

4. Results

4.1. Test Comparison

The main results of this study are plots of cone penetration compared with the three other rheological measurements. These results are given in Figures 6–9. Figures 6 and 7 also contain data from a previous study [1].

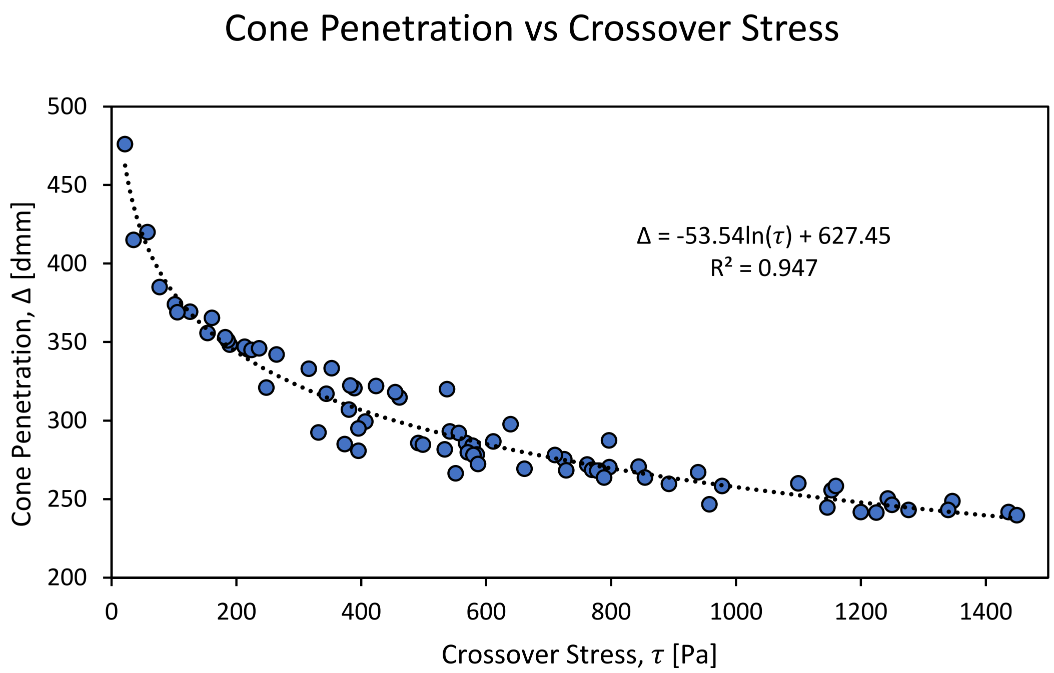


Figure 6. All data (77 points) collected by the present authors, including points from a previous study [1].

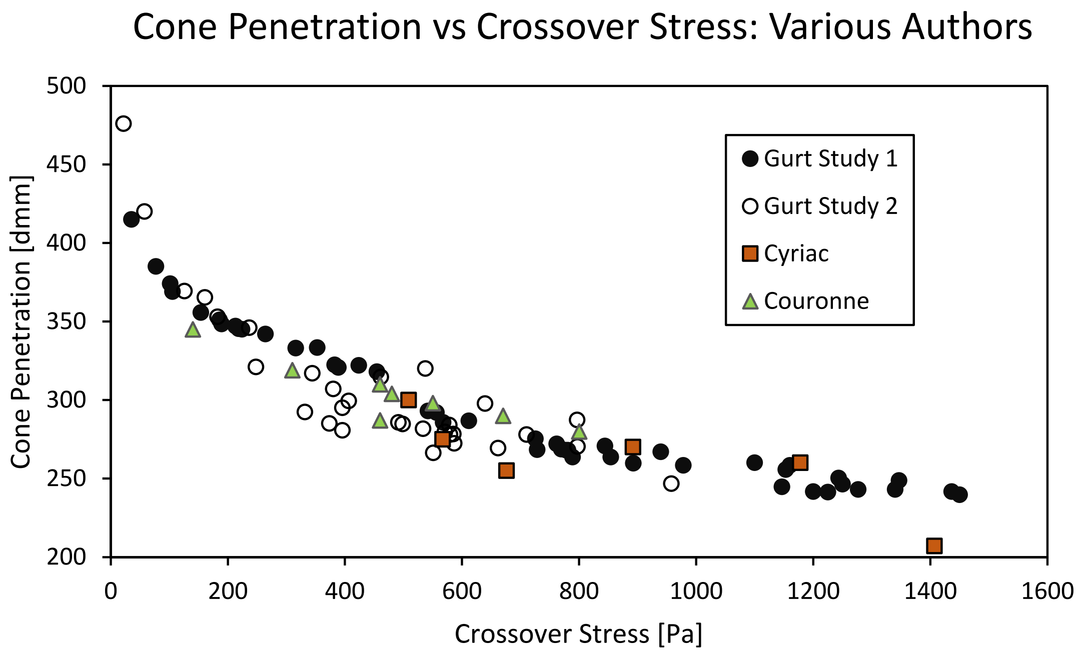


Figure 7. Collection of cone penetration vs. crossover stress data from various authors [1,28,39] showing similar results.

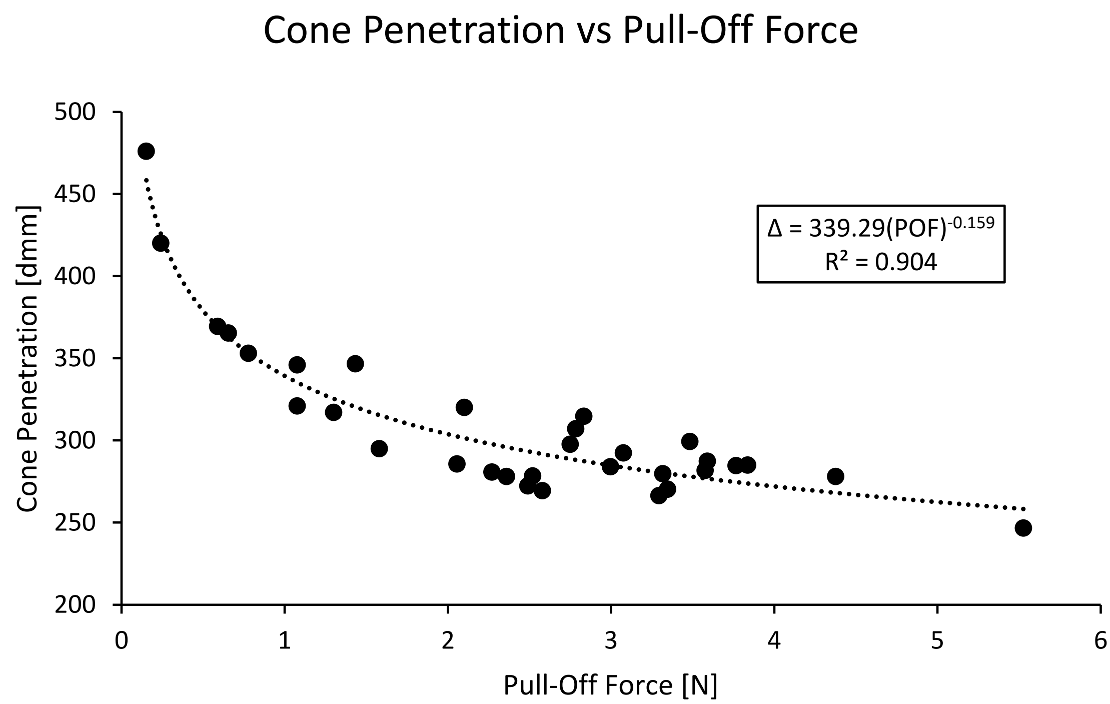


Figure 8. Plot of cone penetration vs. pull-off force showing a reasonable degree of correlation.

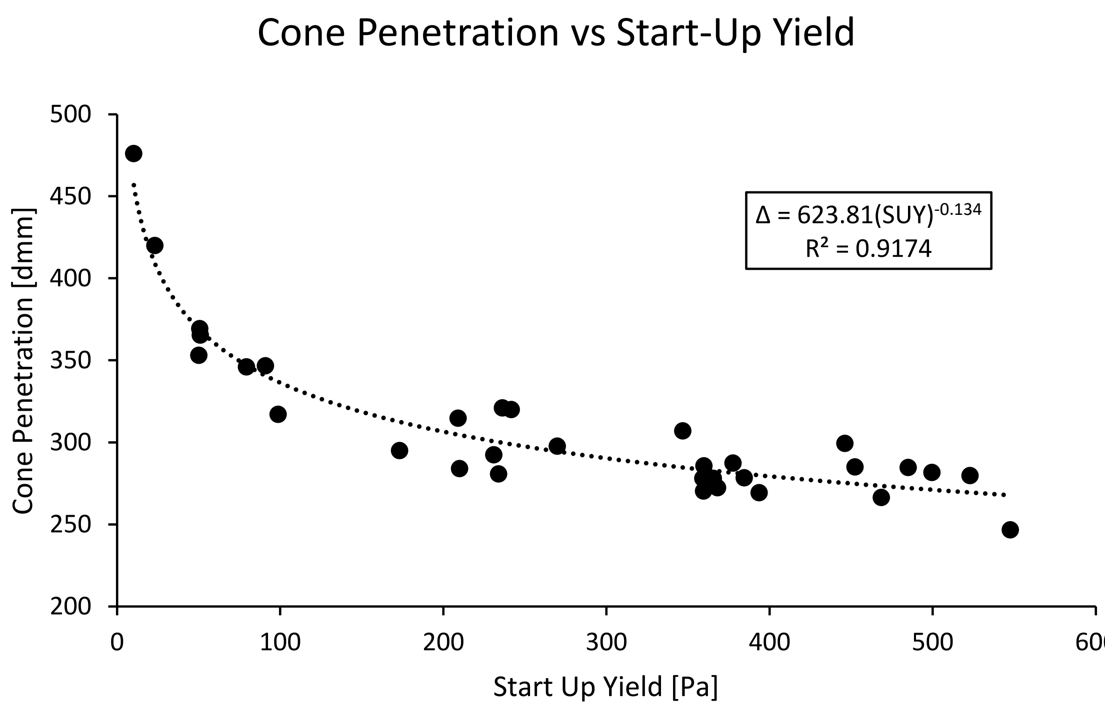


Figure 9. Plot of cone penetration vs. start-up yield showing a reasonable degree of correlation.

Figure 6 demonstrates that crossover stress can be used to estimate cone penetration with a reasonable degree of accuracy. In fact, out of 77 averaged points, every measured value of cone penetration is within 10% of the value from Equation (9). This equation is close to the one reported in the previous study [1].

$$\Delta = -53.54\ln(\tau_c) + 627.45 \tag{9}$$

Figure 7 shows the same data but also includes results from other authors who performed similar measurements using the same parameters. The results are clearly quite similar, indicating that the results obtained for this study are reproducible.

Figure 8 shows that pull-off force may also be used to estimate cone penetration values. The correlation is not as strong as the one for crossover stress and there are no available data suggesting a high degree of reproducibility, but the measurement technique is fast, simple, and can be performed on equipment other than a rheometer. Therefore, this method should be investigated more in the future.

Figure 9 shows similar results, indicating the start-up yield value may also be used to estimate cone penetration. This technique is relatively quick and simple, but the results may depend on sample handling and the exact type of plate used.

Finally, Figures 10–12 show the three other possible plots comparing the measured parameters to each other. Interestingly, the correlation among these is quite poor and demonstrates that although crossover stress may be used to estimate cone penetration reasonably well, it cannot be used to predict pull-off force or start-up yield with a reasonable degree of accuracy. Nevertheless, these plots show that the three techniques are independent ways of estimating cone penetration.

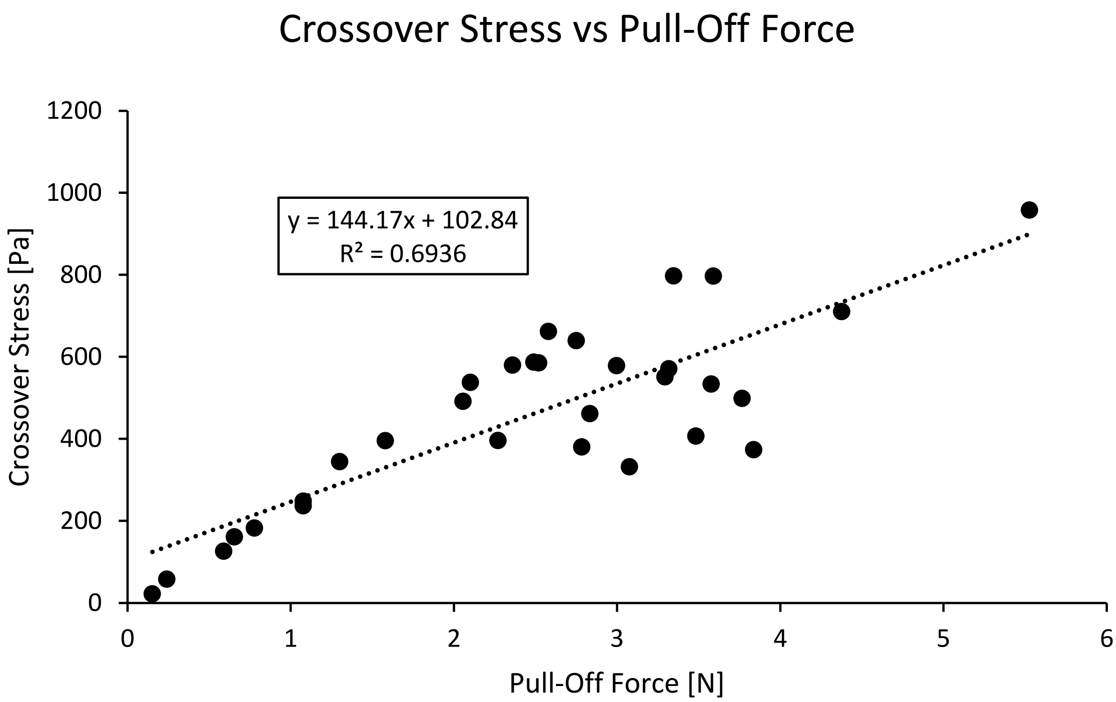


Figure 10. Plot of crossover stress vs. pull-off force showing a general positive correlation but high variance.

4.2. Analysis of Methods

A basic analysis of the aforementioned procedures was conducted by measuring 14 grease samples using the three methods with both a flat plate and a profiled plate. This was performed to examine the sensitivity of each method to surface effects since structured geometry can provide different flows and act as a higher effective gap [40]. Results are provided in Figures 13–15.

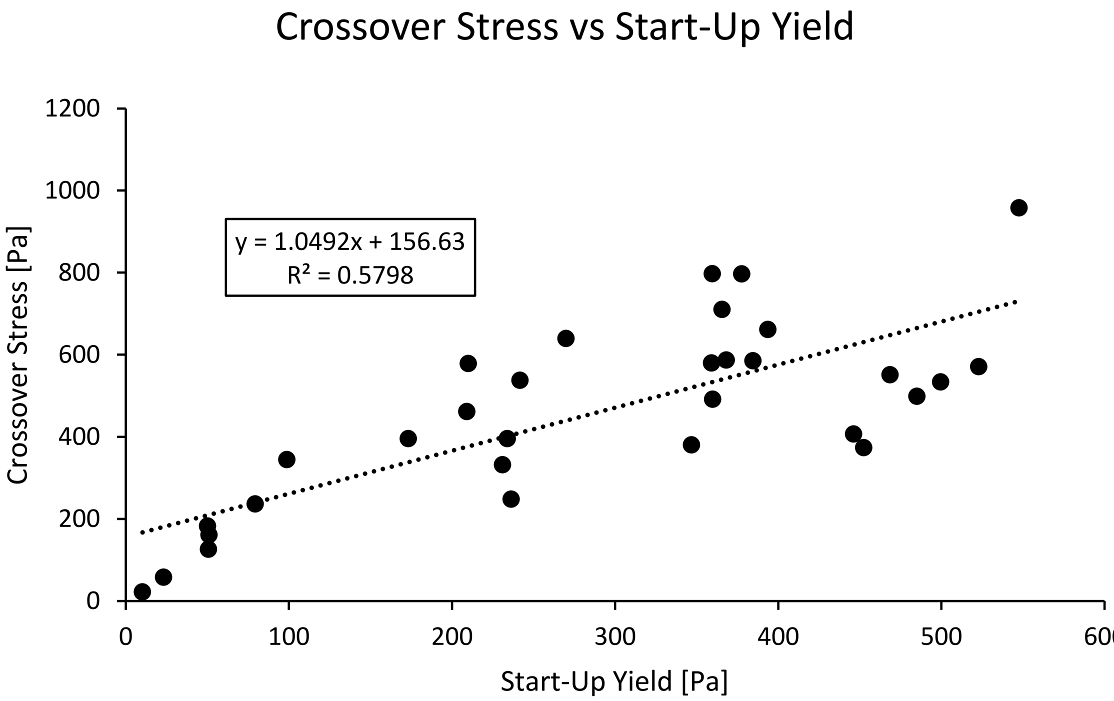


Figure 11. Plot of crossover stress vs. start-up yield showing a general positive correlation but high variance.

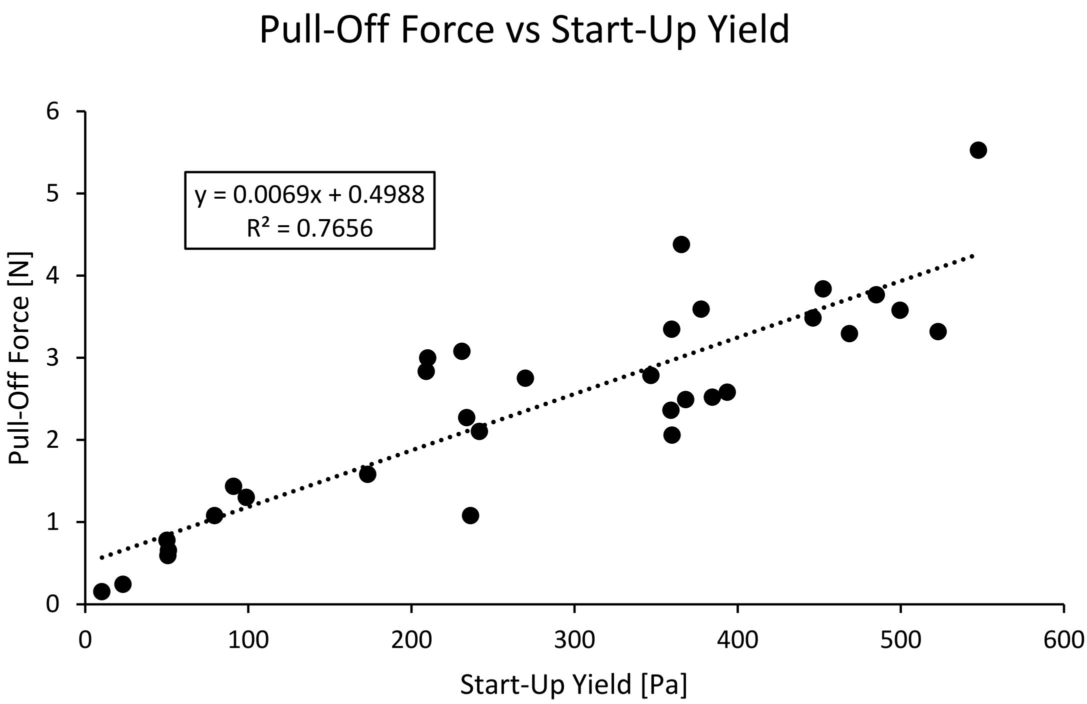


Figure 12. Plot of pull-off force vs. start-up yield showing a general positive correlation but high variance.

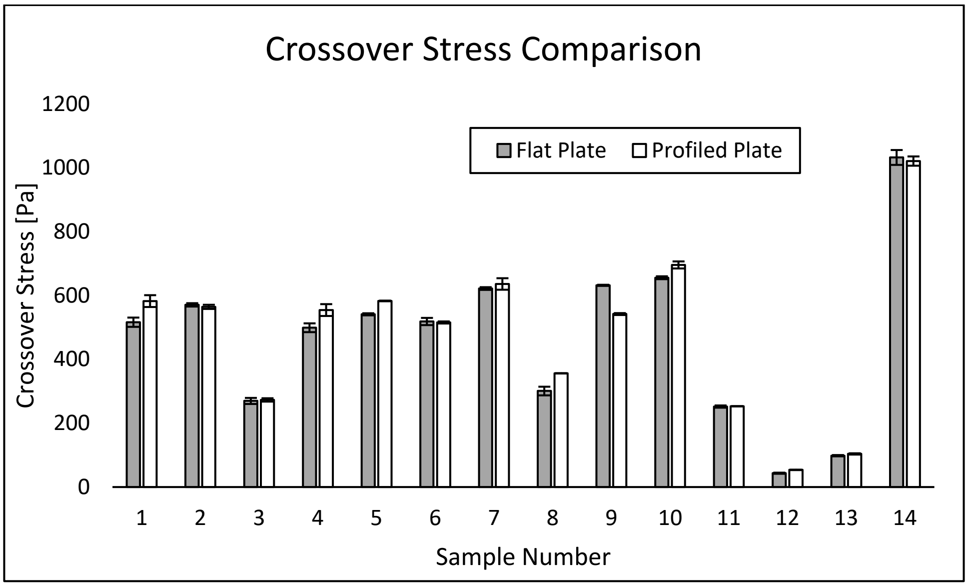


Figure 13. Comparison of crossover stress values obtained using a flat plate and profiled plate to measure the same sample showing approximately the same values.

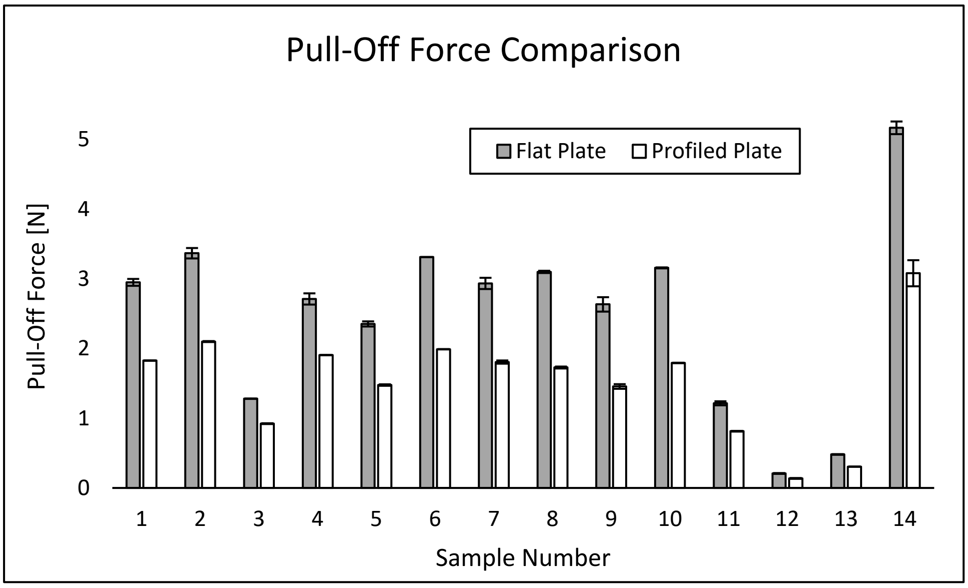


Figure 14. Comparison of pull-off force values obtained using a flat plate and profiled plate to measure the same sample showing significantly higher measured forces with the flat plate.

Figure 13 shows that crossover stress is not particularly sensitive to the plate surface. In addition, the standard deviation of measurements is quite reasonable, meaning that this technique is robust.

Figure 14 shows that pull-off force is very sensitive to the plate surface, as results using flat plate are approximately 1.6 times the value of those using the profiled plate. This may be explained by different mechanics, such as flow disruption caused by the profiling. Therefore, this test is not strictly testing adhesion.

Figure 15 shows that, as expected, start-up yield is almost always higher when measured with the profiled plate due to reduced slip. Surprisingly, two measurements actually show slightly higher values with the flat plate, though the values are quite close.

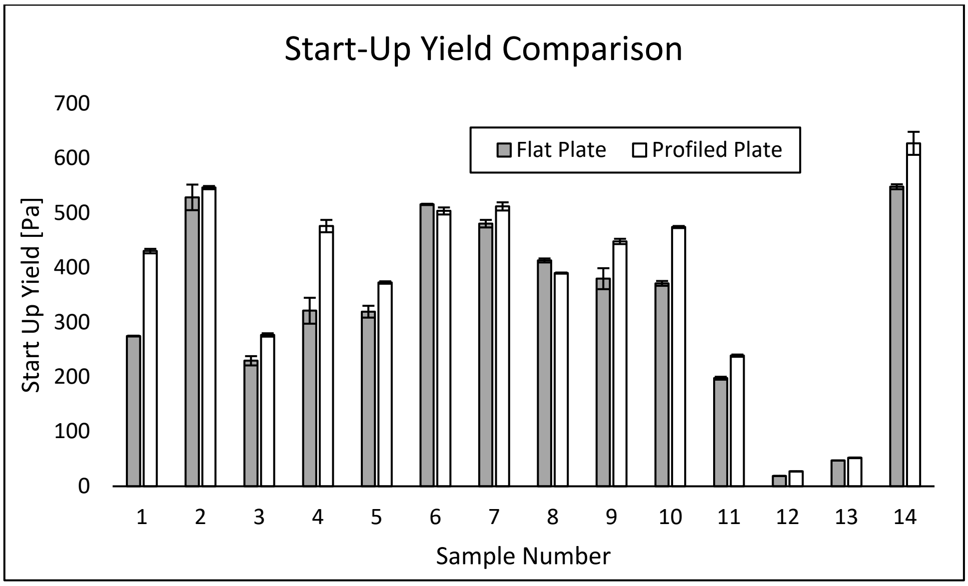


Figure 15. Comparison of start-up yield values obtained using a flat plate and profiled plate to measure the same sample showing higher measured values with the profiled plate.

Because slip behavior is important to understand for greases, the net start-up yield (profiled plate value minus flat plate value) was examined for a relationship with cone penetration, as the factors that generally lead to higher cone penetration (higher thickener concentration, larger particle size) were expected to show more slip. A plot of cone penetration vs. net start-up yield is provided in Figure 16.

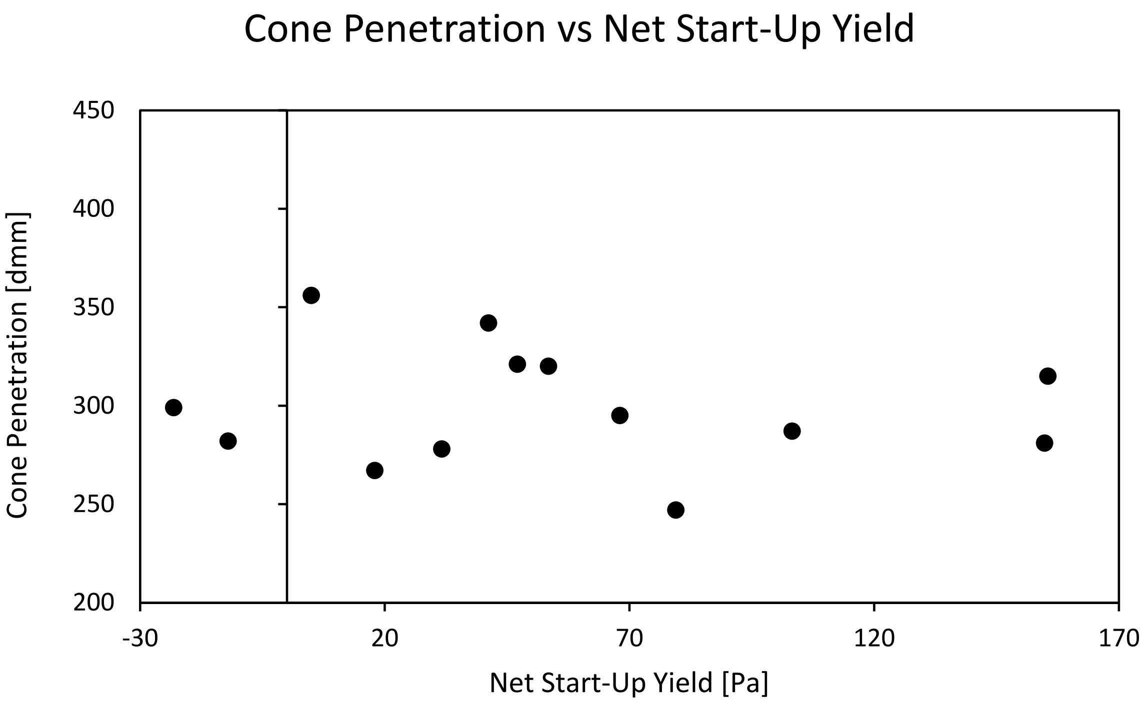


Figure 16. Plot of cone penetration vs. net start-up yield (difference between profiled plate and flat plate) showing that a higher consistency does not mean a higher degree of wall slip.

It was expected that greases with higher consistency (lower cone penetration) would have a more pronounced difference between start-up yield values obtained from the flat and profiled plate due to generally larger particle size and decreased slip. This plot shows that there is actually no obvious relationship between the two and that wall slip cannot be predicted by considering cone penetration measurements. However, this assumes the net start-up yield value directly indicates wall slip.

5. Discussion

Many rheological tests are available to measure grease; this study only covers three techniques because these three were found to work well when estimating cone penetration. Many other tests can be used to track the degradation of grease, though their core issue is that they may only be able to provide relative values for a single grease. The methods cannot be broadly applied to other greases and cannot compare the degradation of one grease to the degradation of another. The methods covered here all have the advantage of being a single number that can apply to any grease and can therefore be used to track its degradation in absolute terms.

Some examples of methods that were not found to work well for estimating cone penetration include the value of the storage modulus in the linear viscoelastic region, the value of strain at the crossover point, the value of moduli at the crossover point, the average force value from die extrusion testing, thread length (see [17]), yield stress, and rheometer penetration [1]. However, the change in these values as a grease degrades may still yield valuable information about a grease. There are also methods that have been used in the past to measure grease degradation that were not investigated, such as complex viscosity, complex modulus, and expended energy to reach the crossover point [12,13].

The comparison of profiled plate start-up yield values to flat plate values was conducted to investigate slip behavior. Upon further analysis of the net values, there are no simple relationships between the net value and any other parameter investigated. Thus, the degree of slip cannot be predicted using anything covered in this paper.

Perhaps some of the differences in the test methods and an explanation for the poor correlations in Figures 11–13 are due to material properties such as surface tension that are difficult to factor into results. The pull-off force is clearly dependent on adhesion, which depends on surface wetting due to the base oil type [15]. Surface tension is also a factor that can influence the formation of air pockets within grease samples that lead to potentially significant measurement errors. This is often an issue with cone penetration measurements.

Ultimately, all tests are arbitrary and only work so long as parameters are defined within useful ranges. An attempt was made to use common numbers and equipment to facilitate future use of these methods. In order to compare measurements, the exact same numerical inputs must be used. Even different scales of cone penetration tests are hard to compare because of complex differences arising from arbitrary geometry. Nevertheless, these methods are suitable for standardization and an overall summary is provided in Table 2.

Table 2. Summary of tests.

Test	Advantages	Disadvantages
Crossover stress	Commonly performed, standardized by DIN [41], excellent correlation with cone penetration, small sample	Depends on frequency, common procedures take a long time for a single measurement
Pull-off force	Quick test, can be performed with a basic setup (actuator and load cell), small sample	Lower correlation with cone penetration, sensitive to geometry/materials, possible issues with sample ejection and becoming detached from walls
Start-up yielding	Shows yielding behavior, can possibly quantify wall slip, small sample	Depends strongly on wall behavior and gap, result is not meaningful yield stress
Cone penetration	Simple test on inexpensive equipment	Requires a large sample, tests combination of parameters that may not resemble performance

6. Conclusions

The results of this study confirm the results of the previous study, showing that crossover stress is a valuable method for estimating cone penetration measurements using very small samples of grease. The methods also appear to be reproducible based on a comparison with additional data from other authors. A faster, more practical method for measuring crossover stress at room temperature is provided here. Despite the relatively quick procedure, all measurements taken were within 10 percent of the values obtained from Equation (9). Therefore, this technique will be used in future studies to understand consistency and grease degradation.

The pull-off force method described here may be an important method for estimating cone penetration in the future. As grease is used in rolling contact, it generally stretches between surfaces, meaning extensional/tackiness behavior is quite important. This method also shows promise in terms of practicality since the test takes a short time and does not need a rheometer. This test can be performed simply with a load cell and an actuation system and provides a rough estimate of cone penetration. Therefore, it should be investigated further and considered a standardization candidate.

The start-up yield test is interesting because it can provide information about the tendency of a grease to slip. Though this study did not find an accurate way of predicting the degree of slip of a sample, this is an important topic across a wide range of situations. Wall slip is a major source of error when performing rheological studies and also manifests itself as a grease is used in practice. Situations such as grease flowing through pipe have a significant amount of wall slip that must be understood to accurately model flow. Thus, the comparative procedure described here can be studied and considered.

The methods covered in this paper are a simple way of characterizing the state of a grease because they provide one number as a result. However, grease behavior is complicated and needs more sophistication to be thoroughly understood. Generally, one needs to consider numerous situationally dependent rheological parameters to properly describe a grease. Overall, consistency as an all-encompassing grease property may be replaced in the future by more specific information/methods or perhaps replaced with standardized rheological tests.

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Nomenclature

Symbol	**Meaning**
A	Area
Δ	Cone penetration
G^*	Complex modulus
τ_{max}	Maximum shear stress value
γ_{max}	Maximum shear strain value
G'	Storage modulus
G''	Loss modulus
δ	Phase angle
η_e	Extensional viscosity
F	Force
R	Radius
$\dot{\varepsilon}$	Strain rate

S	Surface tension
U	Velocity
h	Gap between surfaces
τ_c	Crossover stress
POF	Pull-off force
SUY	Start-up yield

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