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DEVELOPMENT OF GREASE TACKINESS TEST

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ABSTRACT

A test to evaluate the tackiness of grease has been developed using a standard tribometer. There is currently no standard test of tackiness. A preliminary study determined the test parameters to use in the subsequent experiments. Twelve different greases were tested and the results showed how the developed test method was able to differentiate between different greases. The results were linked to the application of grease to a rail using a scaled-wheel rig developed at The University of Sheffield. The developed test method showed the same relationship as the larger scale tests, leading to the conclusion that the developed method can be used to inform larger scale tests which are more costly and time consuming. The effect of

"working" the grease prior to the test showed that the working had a significant effect on the tackiness of the grease. The test method was shown to be sensitive to small changes in the grease by adding small amounts of tackifier additive (0.1 % increments) to the grease.

INTRODUCTION

Greases are widely used in many different applications to improve the performance of systems and protect components from failure. Understanding the properties of a grease and impacts of additives is vital to selecting the appropriate grease/additive to use.

This work uses the example of grease pick-up in the railway industry to apply context and validate the developed test method of tackiness. Grease is applied to the gauge corner of rail in curves. This reduces the friction and forces in the wheel-rail contact to reduce wear and rolling contact fatigue (RCF), prolonging the life of the rail and wheels. The grease transfers to the wheel flange from lubricators, often placed next to the rail in straight track. The wheel then carries it down the track lubricating the rail in the subsequent curves. How much grease gets picked-up and carried down the track is important to ensure adequate lubrication of the rail curves. The tackiness of the grease plays an important part in this transfer process, ensuring enough grease is transferred from the lubricator to the wheel and then from the wheel to the rail.

There is a large body of published research on the effect flange lubrication has on tribological performance of the railways, such as RCF [1-2], retentivity [3], friction and wear [3–10]. These tests predominantly use small-scale laboratory tests (either pin-on-disc or twin-disc). There are few papers that study the physical application of grease to the track. This is important as without understanding how much grease to apply, or how far down the track the grease is carried, it is unknown if the performance benefits seen in the laboratory actually take place during operation in the field. Grease pick-up has been investigated at the University of Sheffield using a bespoke test rig [11-12]. There have also been a few field studies of flange lubrication [14-15]. Whilst the tribological performance and application of flange lubrication is vital to study, there is no mention in the literature of how the properties of grease (e.g. base oil viscosity/shear stability) or different components of grease (e.g. different additives or thickeners) effects the performance or application.

Currently there are no standard tests for grease tackiness and very few studies of tackiness in the literature. A standard lab test is required for tackiness for quality control purposes and for greases to be optimised for their specific application.

Aim

The aim of this work was to devise a test method to quantify tackiness of grease. This test would be able to be used in the future to link tackiness to performance benefits. Initially test parameters had to be determined that would produce consistent, reproducible results. To prove the test method works, the effect of roughness on tackiness and different greases were tested. Also, the effect of “working” the grease pre-test was analysed. Finally, the effect of adding small amounts of tackifer additive on the tackiness was assessed.

Grease Tackiness Research

Tackiness is described as the ability of the grease to form strings or threads [16]. It is often confused with adhesion which refers to the ability of the grease to stick to a surface. Figure 1 shows how a grease forms threads when a train wheel rolls through a grease applicator site. The grease is pumped through an applicator bar, forming bulbs of grease on the side of the rail. The wheel flange contacts these bulbs, transferring the grease onto the wheel. Tackiness is important in the wheel-rail contact as a tackier grease will form longer strings, enhancing grease pick-up from the GDU to the wheel. More grease pick-up on the wheel leads to better lubrication of the contact in curves which improves the life of the rail and wheels. Tackiness performance of a grease can be improved by incorporating additives to it. Tackiness additives are often high molecular weight polymers, susceptible to breakdown by high shear rates [17].

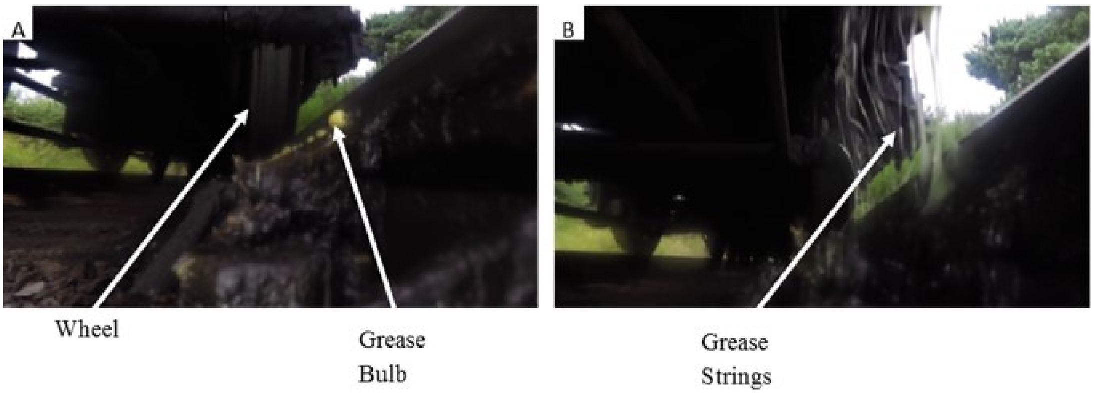

Figure 1- Still images captured from video camera A) before contact between first wheel of train and grease bulb B) After first wheel has passed through lubricator site but before second wheel

The simplest method of determining tackiness is a finger test. A blob of grease is squeezed between finger and thumb and then this finger and thumb are pulled apart. A similar test using a spatula is shown in Figure 2. This is currently used at the end of a manufacturing line during quality assurance to determine if the grease is an acceptable tackiness. The results from this test are based on observation, “feel” and are qualitative. For quality assurance purposes, a more standardised and quantative measurement would be extremely beneficial, also allowing comparison of different grease formulae.

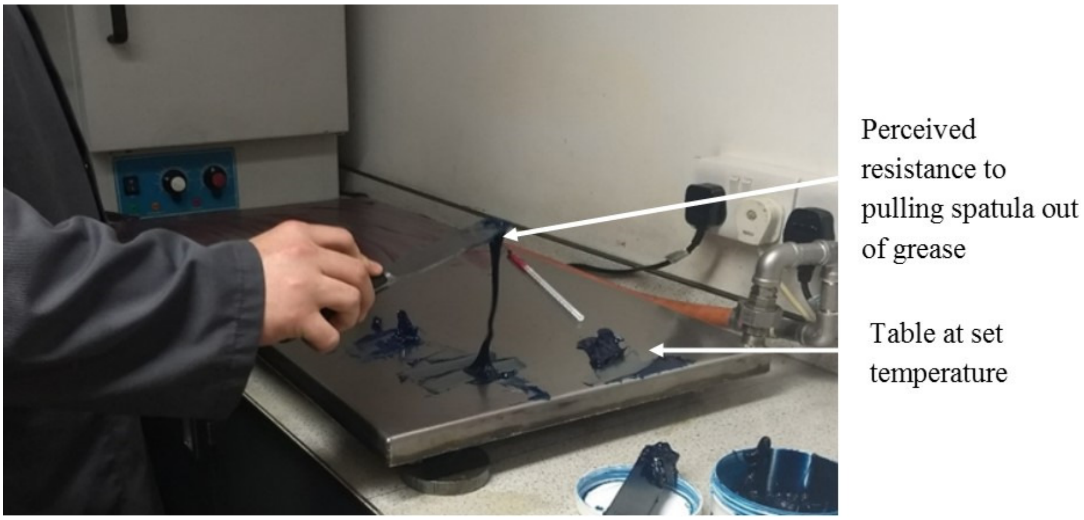

Figure 2- Current tackiness test

A more sophisticated method of measuring the tackiness is an open siphon technique described in detail in [17]. This method measures the length of a formed string of grease, the longer the string, the tackier the grease is said to be. Another test method is an approach/retraction method [16]. This method has been validated with cone penetration tests by comparing the cohesiveness results and has shown good correlation between the two methods. It uses a measured force during retraction to calculate the energy required to break the grease strings as a measure of tackiness. The final test method found uses a Capillary Break-up Extensional Rheometer to measure the string formed during retraction of two discs [18]. The diameter of the strand is measured via a laser as a function of time.

Work carried out by Strasburger et al. [19] showed different forms of separation according to the force trace produced during the test as seen in Figure 3. The results were obtained using an approach-retraction method. There were clear differences in the traces depending on the type of separation. Initially, the separation is characterised by the flow of the liquid where the force rises to maximum and then decays to zero as seen in Figure 3a. After a threshold value, cavitation causes a rapid decrease in force after the maximum value before a sharp turn and decaying to zero as seen in Figure 3b. Figure 3c shows a transitional period trace where there is both cavitation separation and separation by flow. The reason given by Strasburger et al. [19] for cavitation causing the rapid decrease in force is that at peak force, the tension in the liquid is released by bubbles which grow and join together to form a cavity. The expansion of this cavity causing the break of the fluid layer and the rate of decay of the force is related to how quickly this cavity expands. Cavitation causes the rapid break of any grease strings formed and so lowers the measured tackiness.

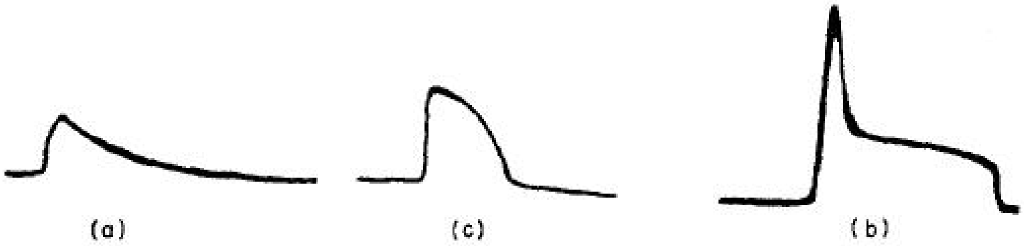

Figure 3- Typical force traces for approach-retraction experiment results. A) trace typical of separation by flow between two surfaces C) trace typical of transition region between flow separation and cavitation separation B) trace typical of separation by cavitation [19]

The polymer chains in greases must have the capacity to expand for a substance to be tacky and the important parameters governing tackiness are the molecular weight and the flexibility of the chains of molecules [17].

There is currently no one method that is universally accepted as a measure of grease tackiness. The only published research found describes the test methods rather than research into tackiness itself and focusses on oils rather than greases. Therefore, there is a need for development of the current available test methods for greases to ensure they are reproducible and produce consistent results. The approach-retraction method will be used as an existing tribometer can be modified to carry out this form of test.

TEST EQUIPMENT

A tribometer was used in this work as its modular construction meant it could be tailored to fit the requirements. A linear lower drive with a vice was used to hold the lower specimen in position. A 50 N load cell was used to provide a suitable level of resolution. A Bruker Universal Mechanical Tester (UMT) is controlled by creating a test script, defining the parameters which the computer then implements when the script is run. Figure 4 shows a typical test set-up. The upper specimen diameter is 29 mm; both specimens have a slight convex surface and machined from stainless steel 316. A video camera was used to record the test. A syringe was used to apply grease to the lower specimen using a mass balance accurate to ±0.0005 g to measure the amount of grease. A nominal amount of grease was pre-smeared on both specimens prior to each test starting. The grease used was a standard multi-purpose grease.

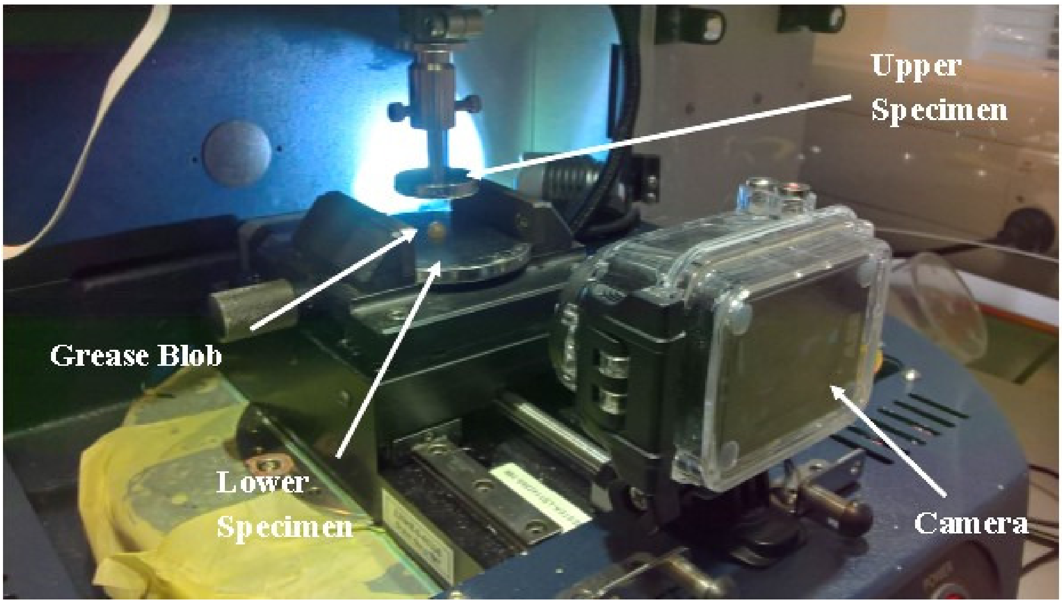

Figure 4- Typical test set-up

Test Method

The method is an approach-retraction type experiment. This method was chosen due to its simplicity to implement. All tests were carried out at room temperature with no temperature or humidity control.

All test followed a standard procedure:

- Specimens pre-smeared with a nominal amount of grease
- Blob of grease applied to lower specimen via syringe using mass balance

- Test script run:
  - Lowers upper specimen until set force is reached for 10 seconds
  - Retract upper specimen at set speed until grease strings broken
  - Excess grease removed from upper specimen
  - Lower specimen re-weighed to measure the grease pick-up onto the upper specimen

Figure 5 shows a schematic of the different stages of the test:

- Stage 1: the upper specimen is lowered until a set force is reached
- Stage 2: the upper specimen is held in position at the set force for a set time
- Stage 3: the upper specimen is raised, pulling the grease upwards forming a grease “string”
- Stage 4: the upper specimen continues to raise, the grease “string” is broken and some of the grease is transferred to the upper specimen

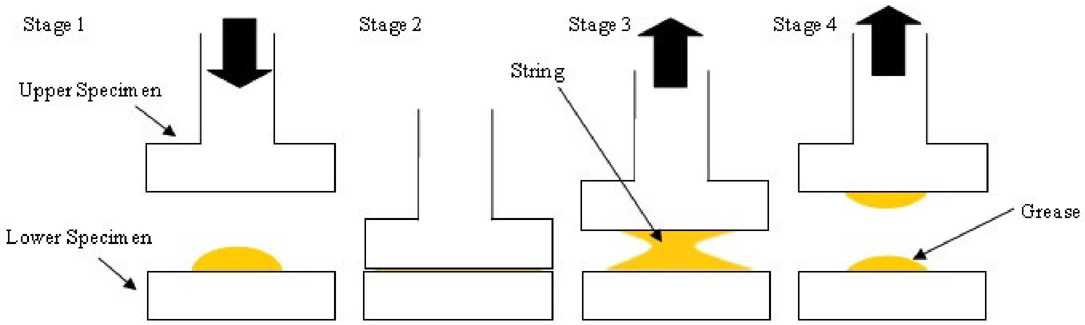

Figure 5- Schematic of test

Before carrying out testing, a number of parameters had to be determined:

- Initial compressive force
- Initial grease amount
- Retraction speed
- Repeatability of results

Each parameter was varied in turn in a preliminary study to analyse its effect and to decide what setting to use in the main investigation. All of the parameters used in all of the tests can be seen in Table 1.

Report Section Number	Initial Compressive Force (N)	Retraction Speed (mm/s)	Initial Grease Amount (g)	Distance of Retraction (mm)	Number of Working Steps	Specimen Type
3.1 Repeatability of Results	2	0.25	0.1	8	0	Smooth
3.2 Initial Compressive Force	1/2/5/10	0.25	0.1	8	0	Smooth
3.3 Initial Grease Amount	4	0.25	0.05/0.1/0.2/0.3/ 0.4/0.5/0.75/1.0	8	0	Smooth
3.4 Retraction Speed	4	0.25	0.1	8	0	Smooth
4 Effect of Specimen Roughness	4	0.25	0.5	10	0	Smooth and Rough
5 "Working" of grease	2	0.1	0.1	8	0/5/10/15/20/ 30/40/50/100	Smooth
6 Tackifier Additive	4	0.25	0.5	10	0	Smooth

Table 1- Summary of all test conditions

Data Analysis

The test rig outputs a text file with the time, distance upper specimen moved and force inside it. Figure 6 shows an example of a typical force distance graph produced from data. This graph is just for the retraction period and not the loading period of the test. The force tends to zero as the grease strings break. This shape of graph is typical for slow rates of separation caused by the grease flowing between the two test specimens [19]. From work carried out by Achanta et al. [16] the three distinct regions represent different properties of the grease:

- Region A- the work done against the grease resistance
- Region B- the work required to start separation
- Region C- the work required to break the grease strings. This region allows tackiness to be calculated.

The area in Region C and the maximum force is calculated by implementing a MATLAB script.

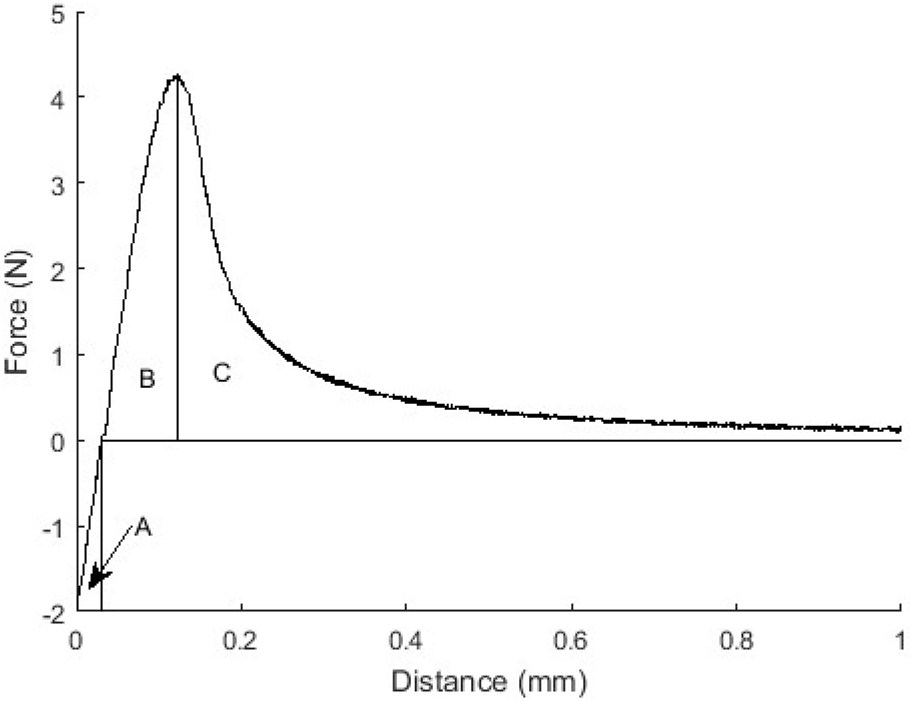

Figure 6- Example force response graph

PRELIMINARY STUDY RESULTS

Figure 7 shows the work done to break the grease strings for each of the preliminary tests. The ideal case is to maximise the work done to break the grease strings as this leads to a reduction in error overall. There is a clear linear correlation between initial grease amount (Figure 7C) and work done to break the grease strings. At the higher grease amounts, the relationship starts to change as some of the grease is squeezed outside the specimen circumference, therefore not effecting the results. There is also a linear correlation between retraction speed and work done (Figure 7B). In Figure 7A, there is a clear increase in work done as force increases with the work done dropping once cavitation effects start occurring. Cavitation effects are more severe at higher compressive forces. Figure 7D shows that the method produces repeatable results.

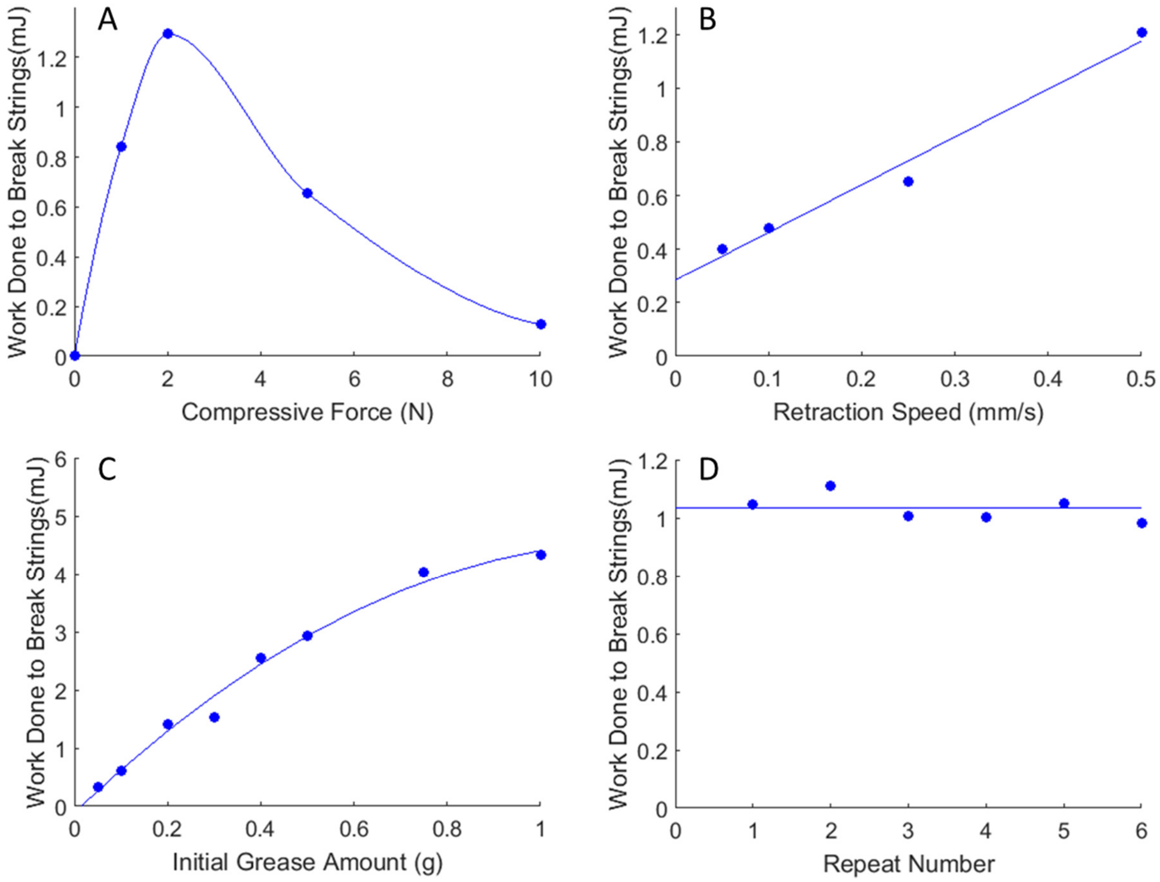

Figure 7- Work done to break grease strings for A) different compressive forces B) different retraction speeds C) different initial grease amounts D) repeats of same parameters

The force curve is influenced by cavitation effects, the parameters detailed in Table 2 to take forward to further tests were chosen to avoid cavitation.

Compressive force (N)	4
Retraction speed (mm/s)	0.25
Initial grease amount (g)	0.5

Table 2- Chosen parameters from preliminary study

These parameters are not representative of wheel-rail contact as usually there is more grease present, there is a greater compressive force, the speed is much faster and the wheel rolls over the grease rather than a straight vertical retraction. However, the parameters chosen will allow repeatable, reproducible results so that comparisons can be made and this method can be used to inform future tests in more realistic conditions.

EFFECT OF SPECIMEN ROUGHNESS AND DIFFERENT GREASES

To investigate what effect the specimen roughness had on tackiness two specimen pairs were made, one with an Ra of 3 µm and a smoother pair with a Ra of 0.6 µm. Twelve different greases were tested on both pairs of specimens using the standard test method described in Test Equipment section. Some of the greases are different formulations of the same grease.

Results

Figure 8 shows the results from the testing with different greases using the different roughness specimens. It clearly shows that the rough specimens require more work to break the grease strings with a couple of exceptions. It is unclear why the roughness causes the tackiness to increase. It could be due to the rougher surface having larger asperities which are a better shape for forming and holding onto the grease strings. This highlights the importance of the surface condition that is being lubricated as changes to the roughness changes the work done to break the grease strings. The graph also shows that the test method is able to differentiate between different greases/different formulations of the same grease.

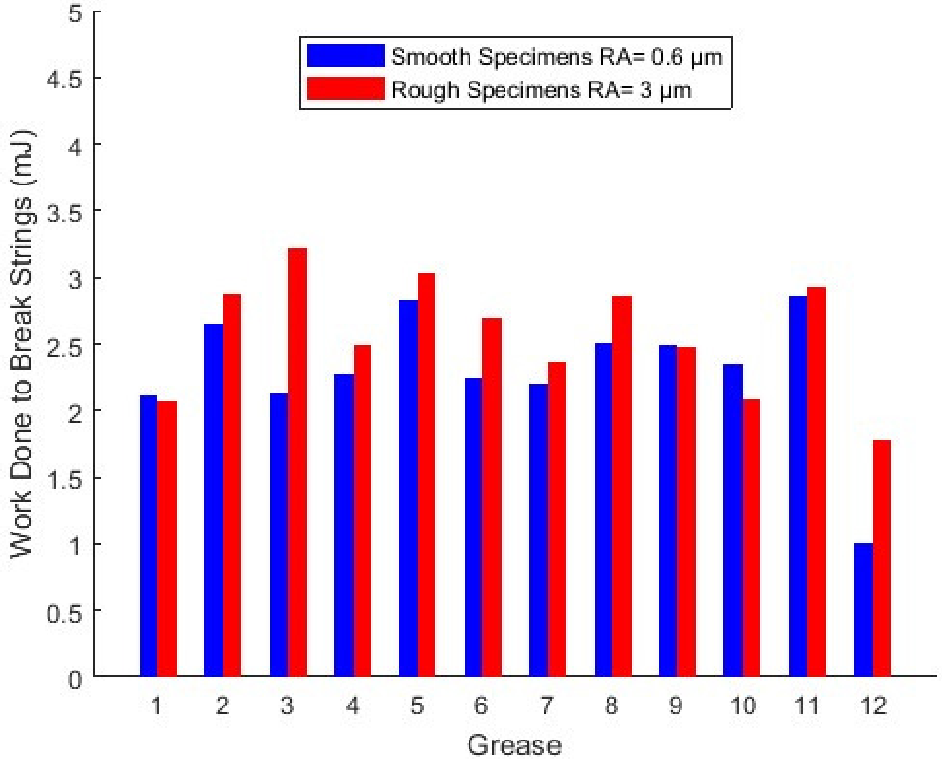

Figure 8- Effect of roughness on the work done to break grease strings

Figure 9 shows the percentage of grease picked-up on the upper specimen. There are no consistent conclusions that can be drawn from this graph. This is because the smooth specimens pick up more grease on some occasions and the rougher specimens pick-up more grease on other occasions. This could be due to poor grease homogeneity as the greases were all manufactured on different dates and stored for different lengths of time.

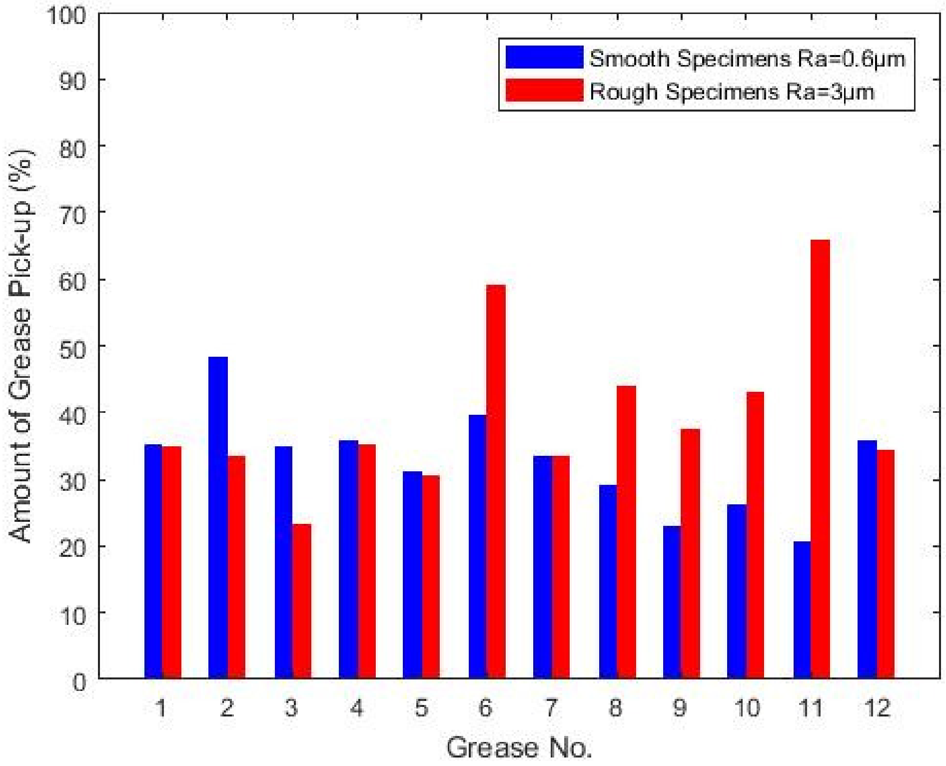

Figure 9- Amount of grease pick-up on upper specimen

Figure 10 compares the pick-up of two greases on a wheel from previous work done at the University of Sheffield. The tests used a scaled-wheel on a short section of rail with two different grease applicator bars tested. Figures 8 and 10 support the hypothesis that the tackier the grease, the greater the pick-up. This is because the tackiness will cause longer strings to form which will cause more of the grease to transfer to the upper specimen. It is difficult to know why this hypothesis is not supported in Figure 9. It is expected that this is because the contact conditions and motion is completely different between moving two conical plates vertically and rolling a wheel along a rail.

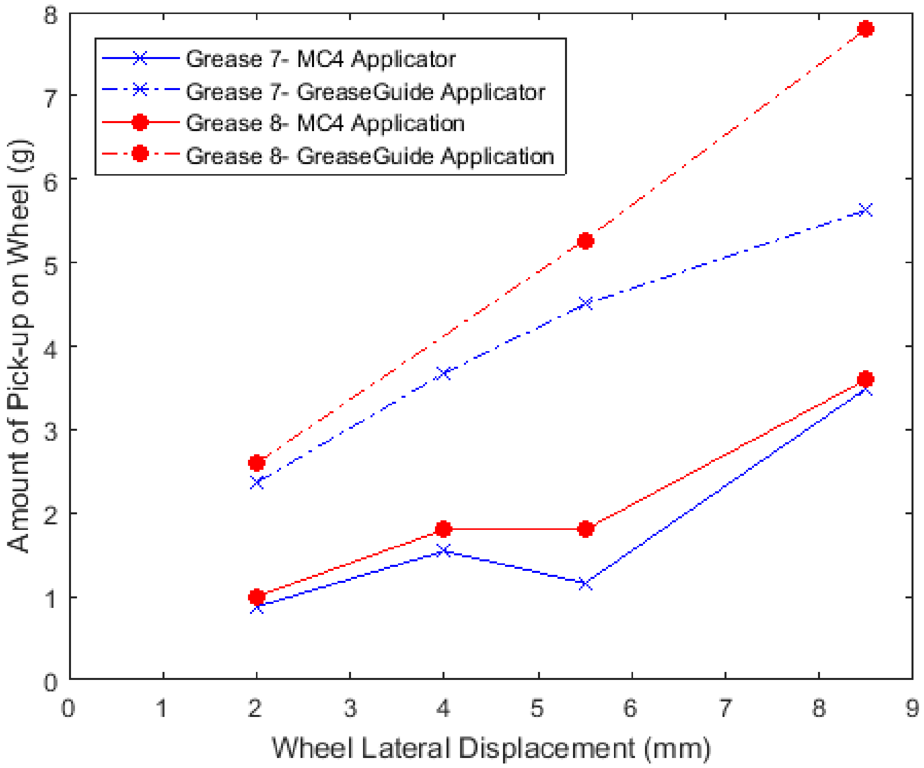

Figure 10- Comparing pick-up of grease on a wheel from a scaled-wheel rig at the University of Sheffield for two different grease and two different grease applicator bars.

Observations

This section of work has shown that rougher specimens cause more work to be done to break the grease strings and hence mean the grease is seen as more tacky. This has not translated to a higher pick-up in the tribometer tests and it is unknown why this is. The tests have also been able to differentiate between many different greases and even between different formulations of the same grease. This has been further extended to a more realistic wheel-rail test rig where the grease with a higher tackiness has resulted in a higher pick-up of grease onto the wheel.

“WORKING” OF GREASE

There has been anecdotal evidence that “working” of a grease prior to carrying out a tackiness test changes the tackiness of the grease. “Working” of the grease in this study means applying small amounts of compressive force to the grease before starting the separation test.

Modifications to Test Method

To investigate if this does occur and to attempt to quantify the effect, a modification to the standard test script detailed in the Test Equipment section was made (changes to script are highlighted in bold text below). A “working” stage was included prior to carrying out the “test” stage

- Specimens pre-smeared with a nominal amount of grease
- Blob of grease applied to lower specimen via syringe using mass balance
- Test script run
    - **Working stage**
        - **Lower upper specimen until force of 2N is reached for 2 seconds**

        - **Retract upper specimen at set speed for a small distance as shown in Table 3**
        - **Repeat previous two steps a set number of times**
    - Test Stage
        - Lower upper specimen until force of 2N is reached for **2** seconds
        - Retract upper specimen at set speed until grease strings broken
- Excess grease removed from upper specimen
- Lower specimen re-weighed to measure the grease pick-up onto the upper specimen

There were two types of "working" investigated with the parameters detailed in Table 3:

- Type A- peak force is reached during working stage, but strings are not completely broken
- Type B- peak force not reached during working stage

Type	Distance Retracted (mm)	Speed of Retraction (mm/s)
A	0.1	0.1
B	0.05	0.05

Table 3- Parameters used during the working stage of script

Figure 11 shows how the two types of working are different as it can clearly be seen that in type A, the peak force has been reached before the grease is compressed again. There is a slight overshoot of force as the grease is compressed, it is not expected that this makes a difference to the results. The graph shows five working steps.

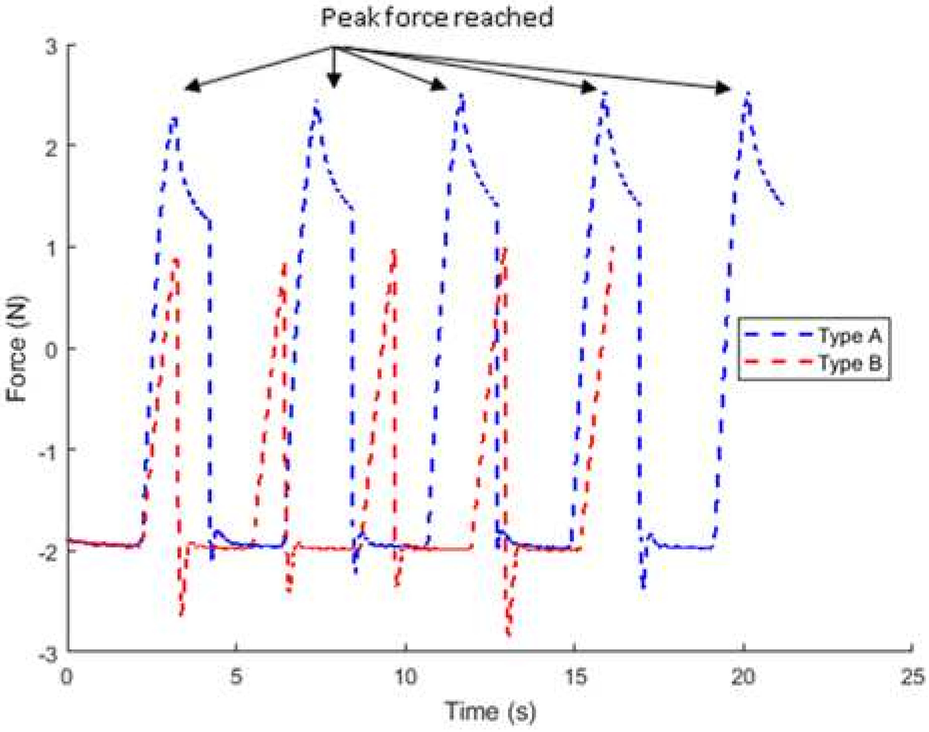

Figure 11- Force time graph of five working stages

Results

To show the effect of prior working of the grease, Figure 12 shows the peak pull-off force reached against the number of steps the grease was worked for before the test was carried out. For type A working the reduction in tackiness is linear as the number of working steps increases. Whereas for type B working, the relationship is increasing for number of working steps and quadratic.

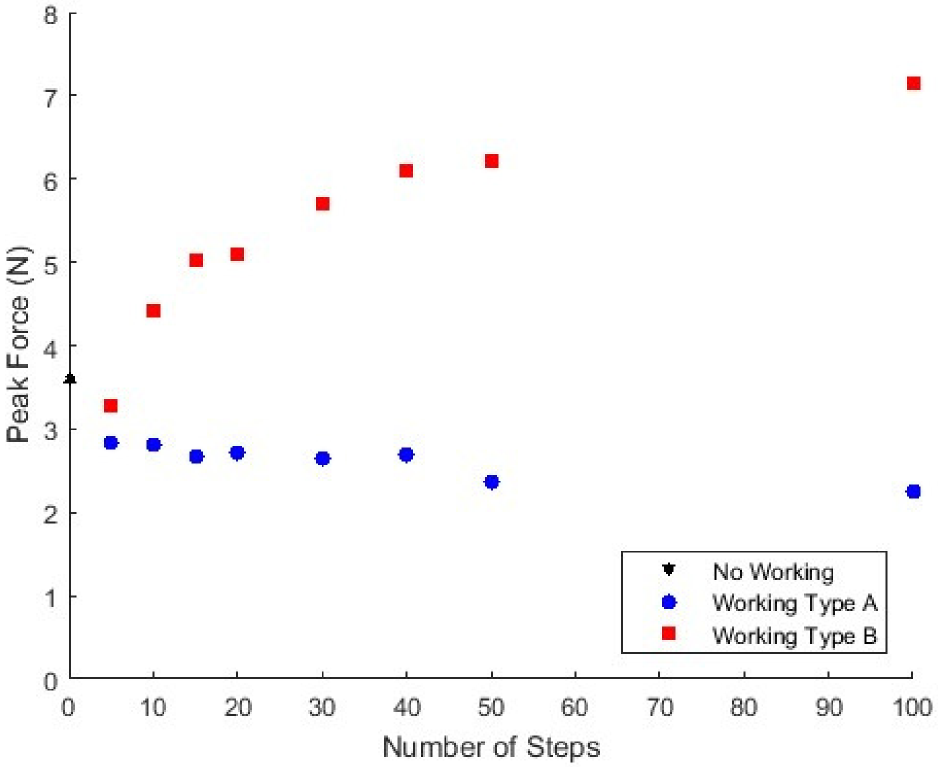

Figure 12- Peak force reached during test for differing number of worked steps prior to test being carried out

Figure 13 shows the work done to break the grease strings. Type B working required more energy than type A working to break the grease strings due to an increase in tackiness with this type of working.

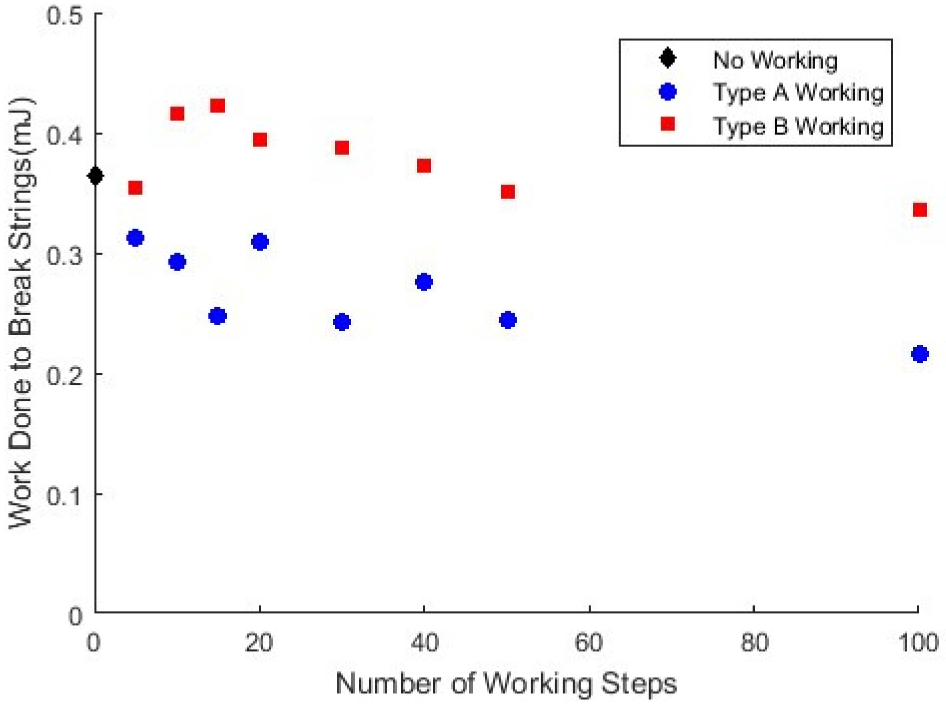

Figure 13- Work required to break grease strings for both types of working

Discussion

Figures 12 and 13 support each other. Both figures show a decrease in tackiness and adhesion for type A working and an increase in tackiness and adhesion for type B working. However, in Figure 13 there is a decrease in tackiness after 15 working steps. This is explained by understanding how the chains of molecules in the grease respond to the two different working types. For the type A working, the maximum pull-off force is reached which shears some of the long chains of molecules into smaller chains. This would happen at each working step so the more steps, the smaller the chains of molecules which need less force to cause separation when the upper specimen retracts fully. These shorter chains are also less elastic than longer chains causing the reduction in tackiness. This explains the linear relationship seen in Figure 12 and 13 for type A working. For type B working, the maximum pull-off force is not reached and the working steps have the effect of aligning the chains of molecules in the grease. This has the result that when the upper specimen is retracted fully, more chains of the molecules share the load and extend elastically, hence more force is required to pull the specimens apart and a corresponding increase in tackiness.

Two things working together cause the quadratic relationship seen in Figure 12 for type B working. First, the cavitation effect places an upper limit on the adhesion of the grease [19] and the more working steps that occur the greater the cavitation effect. Secondly, the working of the grease is aligning the molecules, but they can only be aligned by a finite amount. Initially, the chains are aligned randomly. Once working of the grease starts the chains get more aligned as the number of working steps increases. Therefore, once the majority of the molecules are aligned, further working of the grease has little effect. In Figure 13 tackiness starts reducing after 15 working steps. This is caused by cavitation occurring breaking the grease strings quicker. This shows that whilst increasing the number of working steps beyond a certain amount increases the adhesion of the grease, it reduces the tackiness.

Observations

This section of work has shown that working the grease prior to testing, changes the response of the grease. If the maximum pull-off force is not reached during working then the peak force seen increases with the number of steps the grease is worked for. If the maximum pull-off force is reached during working the opposite occurs. This is due to changes in the arrangement and length of the chains of molecules in the grease. The work done to break the grease strings shows that if maximum pull-off force is not reached during working the tackiness increases until cavitation effects occur and start to decrease the tackiness. If maximum pull-off force is reached during working of the grease then tackiness is reduced.

TACKIFIER ADDITIVE

To test how sensitive the test method is, small amounts of a tackifier additive were added to the grease in increments of 0.1% by weight starting at 0%.

Modifications to Test Method

The standard test method described in Test Equipment section was used in these tests. The initial grease amount was 0.5g, the retraction speed was 0.25mm/s and initial force was 4N. The samples were mixed using the following method:

- Base grease added to mixing pot (~70g)
- 0.1% by weight tackier additive added to mixing pot
- Mixed by hand for 3 minutes
- Sample removed via syringe (4-6g)
- Previous steps repeated to get all 5 samples
- Syringes placed in dry ultrasonic bath for 10 minutes and heated to 40°C
- Samples prepared 48 hours before testing

Results

Figure 14 shows the effect of adding the tackifier additive on the tackiness of the grease. The overall trend is for an increase in tackiness although there is large amounts of scatter in the results. The scatter comes from the difficulty in ensuring that each test sample has the exact ratio of additive to base grease due to the small quantities involved. This highlights the fact that when adding additives or mixing greases together for this test careful preparation is required. Otherwise, the effects of the additive can be lost in variability of the results.

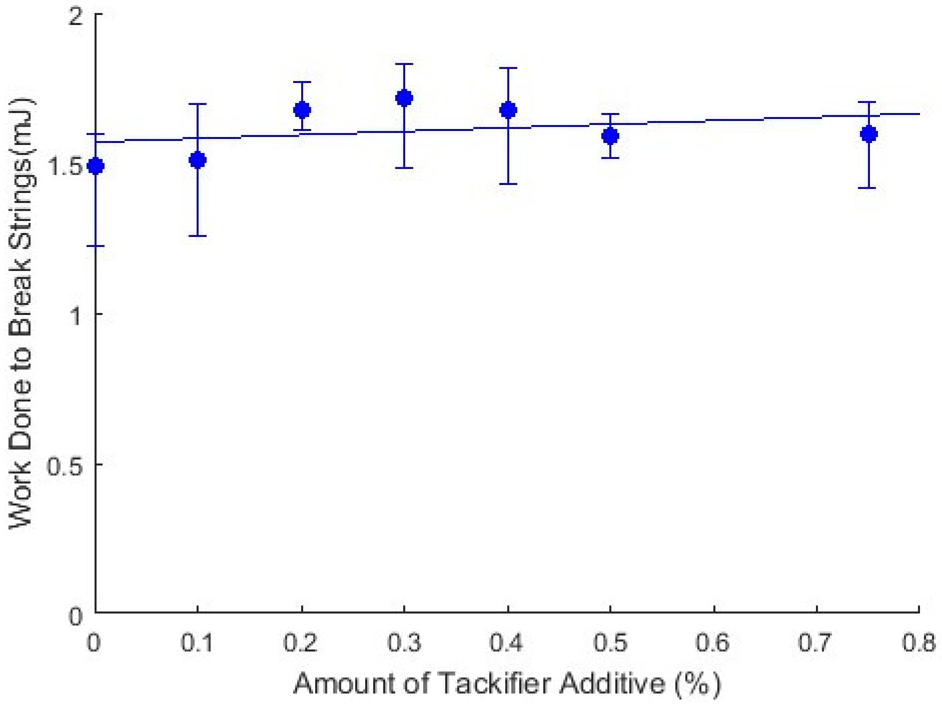

Figure 14- Work done to break grease strings with different amounts of tackifier additive- improved grease mixing method used

Figure 15 shows the mean peak force. It again shows an increased peak force for increasing tackifier additive. Figure 16 is an enlarged section of Figure 15 to show the results more clearly. It is important to note the y-axis scale.

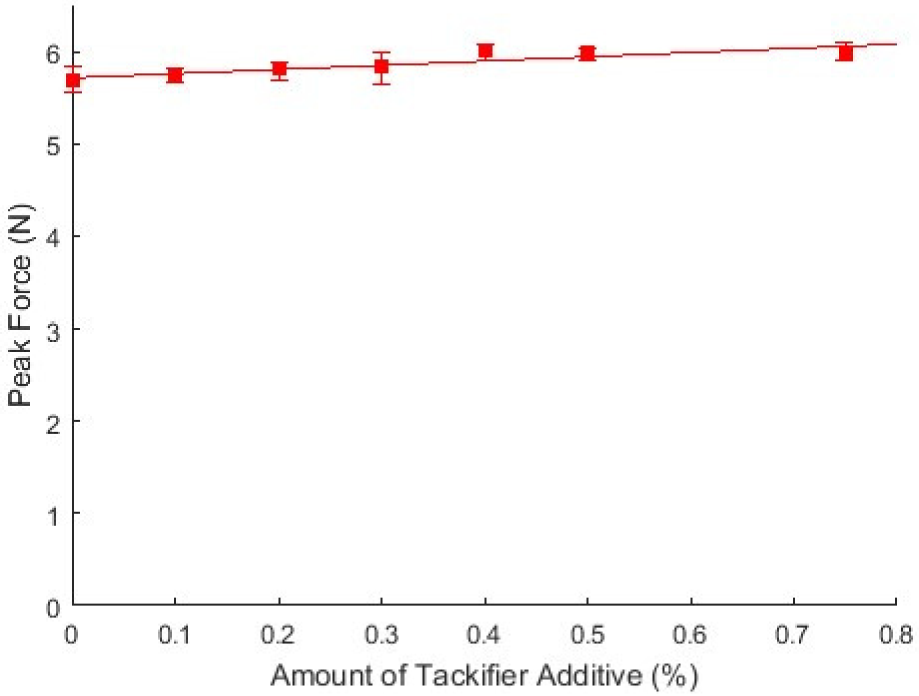

Figure 15- Peak force vs amount of tackifier additive for both mixing methods

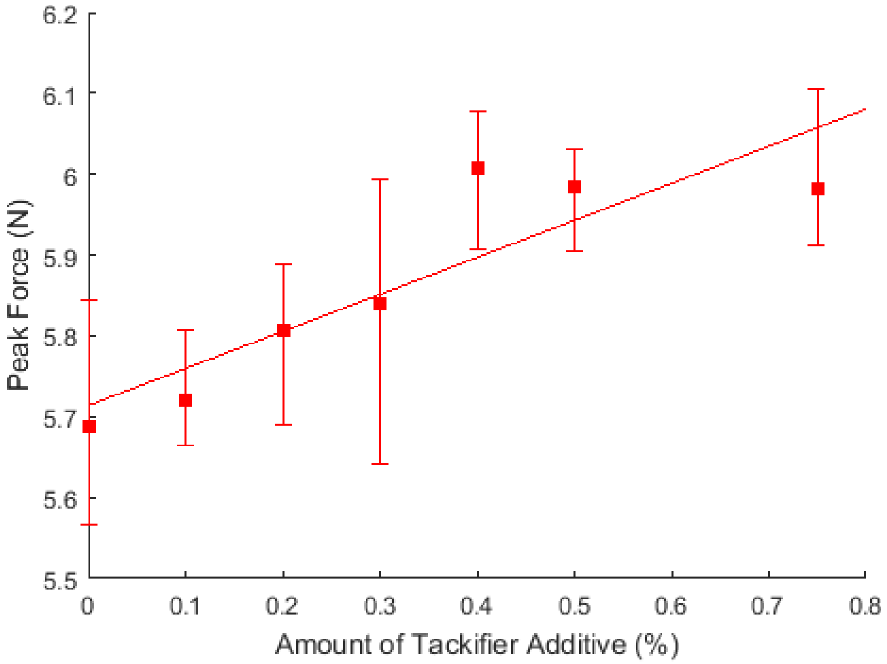

Figure 16- Zoomed in peak force vs tackifier additive graph

Observations

This section of work has shown that the test method does detect small changes in the grease but careful preparation of samples is important to ensure any experimental error is not greater than the measured changes in tackiness.

CONCLUSIONS

A test method using a standard tribometer has been developed using an approach-retraction method. Initially, different test inputs were investigated to understand how the parameters affected the results. Parameters were chosen to limit the cavitation effects seen in results. The test method has been shown to be able to differentiate between different greases and so can be used as a benchmarking method. The wide range of results seen shows that the test method can be used for a large range of products and applications increasing the relevance of this test method. The effect of adding small amounts of a tackifier additive has shown that whilst it is difficult to get the right quantities of additive onto the test specimen, the effect of the additive can be seen in the maximum pull-off force results and tackiness of the grease. Working the grease prior to testing has an effect on the response of the grease and can be incorporated into the test method if required. This enables this test method to be modified to suit a particular application, further increasing the applicability of this method. The results from this tackiness test have been compared to a larger scale wheel-rail grease pick-up test and have shown to have the same relationship between greases for both of the test methods. This shows that this new test method can inform the results of larger, more complicated tests.

Further Work

The test conditions can be expanded to cover different humidities/temperatures with an addition of an environmental chamber to the tribometer. The test could also be used with the conditions matched to a specific application. This would show if a better performing grease using this test method relates to a better performance in the real application.

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