Study on Rheological Properties of Different Brands of Grease

Yuanyuan Sun1,2,*, Yuexing Wang1,2
1Jiangsu XGMG Constuction Machinery Research Institute Ltd. Xuzhou, China
2State Key Laboratory of Intelligent Manufacturing of Advanced Construction Machinery. Xuzhou Construction Machinery Group, Xuzhou, China

*Corresponding author e-mail: sunyuanlculc.2009@163.com

Abstract. The rheological properties of seven kinds of lithium greasewere tested by Anton-paar MCR 302 rotational rheometer, and the Herschel-Bulkley rheological equation \( \tau = \tau_0 + \eta^n \) gives the calculation formula of apparent viscosity and analyzes its rheological properties. The results show that the rheological property of lubricating grease decreases with the increase of shear rate and decreases with the increase of temperature. Different kinds of lubricating grease show different elastoplasticity under the same experimental conditions, and composite lithium-based grease shows the best performance.

1. Introduction

Rheology is a discipline that studies the flow and deformation of materials from aspects of stress, strain, temperature and time [1]. Grease is a typical non-Newtonian fluid with viscoelasticity and thixotropy. In the performance evaluation of lubricating grease, parameters such as dropping point, cone penetration and oil separation amount have been widely concerned by the public [2], but they cannot well reflect the actual performance of lubricating grease.

However, the rheological property of grease can reflect the lubricating state of grease under certain working conditions, so it is very great significance to establish and study the rheological property of grease for understanding the actual use performance of grease [3]. In 1956, Ering H [4] used Ree-Eyring non-Newtonian model to describe the rheological phenomenon of grease. Sasaki [5] and others used Bingham model to describe the rheological properties of grease for 60 years, and Herschel-Bulkley [6] established a three-parameter H-B rheological model based on this model, which can well simulate the rheological properties of grease at low speed.

Lithium-based grease is the most important one in soap-based grease. It is generally prepared from hydrogenated castor oil, 12-hydroxystearic acid and stearic acid. It has excellent shear stability, water resistance and high dropping point. Thixotropy of lubricating grease is a phenomenon that the consistency of lubricating grease subjected to shearing action decreases with the increase of shearing time at a certain shearing rate, and the consistency starts to rise again after the shearing action stops, and the structural part of lubricating grease recovers. Through the thixotropic ring area to study the speed of grease structure recovery, analyze the relaxation time of different greases to measure the structural stability of grease. Yield stress refers to the minimum shear force at which the colloidal structure inside the grease is destroyed, which can reflect the strength of the network structure and is related to the tightness of the grease.
2. Experimental materials and methods

2.1. Basic physical and chemical test indexes of lubricating grease

Seven different brands of lithium-based grease were selected for index test, numbered a, b, c, d, e, f, g.

| Test properties                        | a     | b     | c     | d     | e     | f     | g     | Test Methods |
|----------------------------------------|-------|-------|-------|-------|-------|-------|-------|--------------|
| Soap-base                              | single lithium | single lithium | single lithium | single lithium | single lithium | single lithium | composite lithium | —            |
| Viscosity of base oil (40℃) / mm²s     | 220   | 150   | 150   | 220   | 150   | 150   | 220   | GB/T 265     |
| Dropping point /℃                      | 193   | 192   | 192   | 213   | 195   | 285   | —     | GB/T 3498    |
| Extension of working taper 0.1 mm      | 289.7 | 299.7 | 287   | 324.3 | 313.3 | 313.3 | 285.3 | GB/T 269     |
| Steel mesh oil separation (100℃, 24h)/% | 0.958 | 1.963 | 1.685 | 1.393 | 1.629 | 2.433 | 1.045 | SH/T 0324    |
| Bearing rust prevention (52℃, 48h)/%   | Class I | Class I | Class I | Class I | Class I | Class I | Class I | GB/T 5018    |
| Evaporation (99℃, 22h)/%               | 0.98  | 1.62  | 1.15  | 1.01  | 0.83  | 1.09  | 1.27  | GB/T 7325    |
| Damp heat experiment (49℃, 14d)        | pass  | pass  | pass  | pass  | pass  | pass  | pass  | ASTM D1748   |
| Grinding spot diameter, mm             | 0.554 | 0.418 | 0.692 | 0.461 | 0.412 | 0.577 | 0.494 | SH/T 0204    |

Through the basic physical and chemical tests and analysis of seven kinds of greases, it is found that there is not much difference between domestic greases and imported greases, and the selected greases all meet the requirements of national standard.

2.2. Rheological experiment of lubricating grease

2.2.1. Experimental Instruments. Anton-paar MCR302 rotary rheometer was used in the experiment, and PP system was selected for testing. The rotor was 0.5 mm from the flat plate. Main indexes of rotary rheometer: minimum torque of oscillation mode is 1 nN·m; The minimum torque in rotation mode is 10 nN·m; The maximum torque is 200 mN·m; Torque accuracy is 0.1 nN·m; The rotating speed is 10⁻¹ r/min~3000 r/min; Angular velocity 10⁻³ rad /s~628 rad /s; Strain amplitude 1 μrad~∞; Normal stress is 0.005 Pa~50 Pa; Controlled temperature -40℃~200℃.

2.2.2. Experimental Contents. Steady-state rheological experiments under shear rate control: ① Under the shear rate control mode, steady-state experiments were used to test the change process of viscosity and shear stress of seven lithium-based greases with shear rate (0~3000s⁻¹) at 30℃. ② Under the mode of controlling shear rate (0.1s⁻¹), the change process of viscosity and shear stress of seven kinds of lithium grease with temperature at 30℃~ 200℃ was tested.
3. Results and discussion

3.1. Steady-state experiments are used to test the changes of viscosity and stress of seven kinds of greases with shear rate at 30 °C. Rheological curves conform to Herschel-Bulkley model and show yield shear.

According to Herschel-Bulkley rheological equation\( \tau = \tau_y + \eta \gamma^n \), where \( \tau \) (Pa) and \( \gamma \) (s\(^{-1}\)) are shear stress and shear rate respectively, rheological limit \( \tau_y \), plastic viscosity and plastic index \( n \) are the three rheological parameters of grease. The shear stress value measured when the test system rotates at an extremely low speed close to zero is \( \tau_y \).

The plastic viscosity and plastic index were obtained by least square fitting. From the rheological equation \( \tau = \tau_y + \eta \gamma^n \), \( \ln(\tau - \tau_y) = \ln \eta + n \ln \gamma \) can be obtained.

If \( y = \ln(\tau - \tau_y) \), \( a = \ln \eta \), \( b = n \), \( x = \ln \gamma \). Then the fitting model is \( y = a + bx \).

Test \( n \) times, \( y \times x \) is linear, according to the principle of least squares, there are

\[
\begin{align*}
\tau &= \tau_y + \eta \gamma^n \\
\ln(\tau - \tau_y) &= \ln \eta + n \ln \gamma \\
\ln \eta &= a + b \ln \gamma \\
\end{align*}
\]

According to the formula:

\[
\begin{align*}
L_{xx} &= \sum_{i=1}^{n} x_i^2 - n \overline{x}^2; \\
L_{xy} &= \sum_{i=1}^{n} x_i y_i - n \overline{x} \overline{y}; \\
L_{yx} &= \sum_{i=1}^{n} x_i y_i - n \overline{x} \overline{y};
\end{align*}
\]

So \( b = \frac{L_{xy}}{L_{xx}} \), \( a = \overline{y} - b \overline{x} \). The calculation results are shown in table 2.

| NO. | H-B | Correlation coefficient r |
|-----|-----|---------------------------|
| a   | \( \tau = 169 + 1374 \gamma^{0.521} \) | 0.930 |
| b   | \( \tau = 138 + 1409 \gamma^{0.711} \) | 0.957 |
| c   | \( \tau = 166 + 1482 \gamma^{0.646} \) | 0.933 |
| d   | \( \tau = 281 + 1678 \gamma^{0.594} \) | 0.959 |
| e   | \( \tau = 111 + 1145 \gamma^{0.421} \) | 0.917 |
| f   | \( \tau = 114 + 1149 \gamma^{0.597} \) | 0.949 |
| g   | \( \tau = 88.6 + 1061 \gamma^{0.725} \) | 0.918 |

Test of Regression Effect (Correlation Coefficient Test Method)

Calculation formula: \( r = \frac{L_{xy}}{\sqrt{L_{xx} L_{yy}}} \), \( L_{yy} = \sum_{i=1}^{n} (y_i - \overline{y})^2 = \sum_{i=1}^{n} y_i^2 - n \overline{y}^2 \).

The correlation coefficient \( r \) of the test results is close to 1. The test results have significant correlation, which indicates that the model is reliable.
Fig. 1 is a graph showing the variation trend of apparent viscosity of seven lubricating greases with shear rate under the motion state of shear rate from 0.01 s\(^{-1}\) to 3000 s\(^{-1}\). The test results show that the apparent viscosity of lubricating grease gradually decreases with the increase of shear rate. b has the largest variation range. When the shear rate reaches 6, the apparent viscosity of b and c tend to be equal. When the shear rate reaches 6.5, the shear rate and a tend to be consistent. When the shear rate reaches 8.5, c and a have the same viscosity and then separate. 16.5 The apparent viscosity of Volvo gradually exceeds f. The trends of a, c, g is basically the same. There is yield stress in lithium grease. With the increase of shear rate, there is obvious shear thinning phenomenon. As shown in the figure, the apparent viscosity measured at room temperature of 30°C is d > c > b > a > e > f > g. Lithium-based grease has the smallest apparent viscosity, the largest flow index and the smallest yield stress, and is Carter composite lithium-based grease. The larger the shear rate is, the stronger the shear action is, and the smaller the measured viscosity value is, that is, the apparent viscosity of several lubricating greases decreases with the increase of the shear rate, showing obvious shear thinning performance and typical non-Newtonian fluid characteristics. At the same time, it is shown that the apparent viscosity of grease has a great relationship with the shear rate, and the shear effect has obvious influence on rheological properties at this temperature. Although the viscosity values of several kinds of greases are different, the change law is similar, that is, with the increase of shear rate, the viscosity of greases decreases, but the degree of decrease gradually weakens. At high shear rate, the viscosity of greases hardly changes with the change of shear rate, while the non-Newtonian behavior weakens with the increase of shear rate. The viscosity of lubricating grease changes with shear rate, which makes it have certain selectivity in actual use. When the speed is higher, the grease with lower viscosity is selected, and then the grease structure is destroyed so that the skeleton structure moves directionally and the viscosity becomes lower. When the speed is lower, choose grease with higher viscosity. The viscosity of grease will not change too much at low shear speed.
3.2. In the steady-state experiment, the change of apparent viscosity with temperature in the range of 30 °C to 200 °C at a low shear rate of 0.1s⁻¹ is tested.

Figure 2 is a graph showing the variation trend of apparent viscosity with temperature of seven lubricating greases tested from room temperature of 30 °C to 200 °C. Apparent viscosity measured at room temperature of 30 °C is d > b > c > a > f > e > g. It is consistent with the results of adhesive shear test. Apparent viscosity of b and c drops sharply with the change of temperature, c and f have two intersections, which coincide for the first time at about 35 °C and for the second time at 70 °C. c tends to rise slowly between 90 °C and 110 °C, d tends to rise slowly between 130 °C and 150 °C. After 115 °C, g > e > b > f, after 148 °C, the apparent viscosity of g is greater than a, and after 185 °C, the apparent viscosity of g is the largest. The abrupt change off at 115 °C may be related to the wall slip of lithium grease at this temperature. The first step appears at 80 °C-120 °C for c and slowly rises at 130 °C-150 °C for d. After 115 °C, g > e > b > f, after 185 °C, g apparent viscosity is the largest. The apparent viscosity decreases with the increase of temperature, and the change trend of apparent viscosity decreases with the increase of shear rate. The strength limit of grease decreases with the increase of temperature.

3.3. apparent viscosity
As a typical pseudoplastic non-Newtonian fluid, the apparent viscosity of lithium grease shows a decreasing trend with the increase of shear rate, and the decrease of apparent viscosity of lithium grease is related to temperature. When the shear rate is in the range of 10⁻¹-100s⁻¹ and the temperature is 70 °C-100 °C, the apparent viscosity of lithium-based grease changes stably, forming the first platform area of apparent viscosity. When the temperature is > 135 °C, lithium-based grease enters the second apparent viscosity platform. The first platform area can be understood as the temperature section where the grease can normally work, while the second platform area corresponds to the obvious structural change of grease.
3.4. Temperature
As the higher the temperature is, the more easily the contact points between thickener particles or soap fibers are damaged, the more unstable the formed structural framework is, and the smaller the shear stress required for lithium-based grease from flowing to starting flowing is, thus the smaller the yield stress is. And the higher the temperature, the smaller the resistance of the long-twisted lithium 12-hydroxystearate soap fiber to directional flow under directional shear, the smaller the apparent viscosity and the larger the flow index. The higher temperature, the more effect.

4. Conclusion
1) Under steady-state experimental conditions, the rheological properties of seven different lithium-based greases were investigated and rheological models were given.
2) With the increase of temperature, the yield stress, apparent viscosity and flow index of lithium grease decrease and increase.
3) Lithium-based grease belongs to yield pseudoplastic fluid, which can recover its original shape within a small strain range. When reaching the yield stress, it has obvious shear thinning phenomenon and shows viscous fluid characteristics after reaching the flow point.

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