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Analysis of magnetorheological grease normal force characteristics in static and dynamic shear modes

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Abstract

In this paper, the normal stress behavior of magnetorheological grease (MRG) in a quiescent state and its dynamic shear modes were systematically studied. Three types of magnetorheological greases were prepared, MRG-30, MRG-50, and MRG-70 with 30%, 50%, and 70% carbonyl iron(CI) powder mass, respectively. Furthermore, static and dynamic shear tests were carried out using a rheometer, the effects of time, magnetic field strength, shear rate and temperature on normal stress were studied and analyzed under both static and dynamic conditions. In the static mode, the research results show that the normal force is related to the magnetic field strength and the content of magnetic particles in the MR grease, but has nothing to do with historical time. the static normal stress generates the evident fluctuation with changing the temperature when magnetic field strengths of 96 kA m\(^{-1}\) and 194 kA m\(^{-1}\). The fluctuation is very small when the magnetic field strengths are 0kA m\(^{-1}\), 391 kA m\(^{-1}\) and 740kA m\(^{-1}\). Similar to the static normal stress, the dynamic normal stress at the degaussing is greater than the normal stress during magnetic field loading. The dynamic normal stress are almost invariant with prolonging the time below 100 s\(^{-1}\) shear rate under the medium magnetic field strength of 194 kA m\(^{-1}\) whereas the stress is increased at the shear rate of 100 s\(^{-1}\). Besides, the dynamic normal stress is basically independent from the time above the shear rate of 1 s\(^{-1}\) at the high magnetic field of 740 kA m\(^{-1}\). Under other conditions, the normal force basically presents a stable value. At the absence of the magnetic field, there are quietly weak effects of temperature on the static and dynamic normal stress. Finally, when a magnetic field strength higher than 96 kA m\(^{-1}\) is applied to the material, the value of dynamic normal stress increases with temperature.

1. Introduction

The properties of Magnetorheological (MR) materials change continuously, rapidly and reversibly when a magnetic field is applied to it [1–5]. Generally, MR materials include MR fluid [6], MR elastomer [7], and MR gel [8]. Among them, MR fluid is one of the most used smart materials and has attracted great interest due to the outstanding adjustability of its rheological properties [1, 9]. As such, it has been widely used in mechanical engineering when designing clutches [10], brakes [11], dampers [12], and actuators [13]. However, the MRF also exhibits high sedimentation due to the density mismatch between magnetic particles and base fluid, which decreases the MR effect and its stability in mechanical application [14]. Aiming to resolve this problem, many studies were carried out [15, 16], such as Change the shape of magnetic particles [17], adding additives [18], introduced single-walled carbon nanotube (SWNT) in the magnetic particles [19], and so on. however, the settlement problem is still not entirely solved. Thus, a novel material called magnetorheological grease (MRG) was proposed.

MRG was discovered by Rankin et al [20] and was regarded as the analogue of MRF [21]. But unlike the magnetorheological fluid, magnetorheological grease has the advantages of low sedimentation rate, simple preparation, and good sealing performance [9, 22]. Therefore, extensive research on its rheological or physicochemical properties was carried out by many researchers. Mohamad N et al studied the dynamic
viscoelastic properties of MR grease made of plate-shaped carbonyl iron particles (MRGP). The experimental results showed that the storage modulus and loss modulus of MRGP depend on the fraction of plate-shaped CI particles [23]. Kejie Wang et al investigated the effects of silicone oil viscosity (SOV) and carbonyl iron particle (CIP) weight fraction and size on dynamic yield stress for magnetorheological (MR) grease, by comparing response surface analysis (RSA), confirming that the CIP score and SOV have a positive effect on the yield stress [24]. Mohamad et al experimentally studied the dynamic viscoelasticity of MR grease under the changing magnetic field and particle fraction. The results have shown that MR grease can withstand high yield stress and have MR effects [1]. Park et al used a mechanical stirring method to prepare MR grease and measured its rheological properties of various magnetic fields in static and dynamic modes. They confirmed that under zero field, MR grease will produce yield stress, and when a magnetic field is applied, MR grease will form a strong chain structure [9]. Wang et al investigated the effect of temperature on the MRG properties. The MR grease viscosity and flow curves under various temperatures and magnetic fields were discussed in detail. The authors found that the temperature has a predictable effect on the apparent viscosity and yield stress of MR grease. The MRG dynamic properties at various temperatures were also investigated. The results showed that as the temperature rises, the storage modulus increases, and the loss factor decreases [25]. Guo et al divided the properties of MR material into two categories; the shear action orthogonal to the field direction and the normal behavior along the field direction [26]. Thus, in addition to studies on shear properties, such as shear stress and shear modulus, research on the normal force behavior has also received great attention in recent years. Its normal force research is also helpful for designing actuators, sensors and other practical applications.

Until recently, studies on the normal force were primarily focused on MR fluid (or MR elastomers). Yao et al used a rheometer to test the normal force of the magnetorheological fluid (MR) in the porous metal foam. The results found that the magnetic field has a significant effect on the normal force of the MRF in the metal foam in the oscillating shear mode. while time and frequency have little influence on the normal force. In addition, the dynamic normal force changes with the shear rate in three stages: it is stable at first, then decreases to a minimum, and finally shows an upward trend [27]. Liao et al studied the influence of monotonic magnetic field load, particle distribution, temperature and magnetic field cyclic load on the normal force of MR elastomers under compression status. The experimental results demonstrate that the normal force increases with the increase of the magnetic field and the pre-compression force. In addition, the normal force first increases as the temperature increases, and then decreases [28]. Guo et al analyzed the quiescent state and normal dynamic force characteristics in MR5 with different compositions under constant various magnetic fields. The study has shown that if the magnetic field is greater than the critical point, the normal stress has a positive value. This value increases with the magnetic field strength and converges to the saturation value [26]. Moreover, the author also observed that the normal force have a low dependence on temperature. See and Tanner reported that the normal stress direction of the MR suspension is parallel to the magnetic field. The authors found that upper and lower plates of the rheometer could be separated in the presence of normal force under no shearing; the shearing value increases with the magnetic flux density but is inversely proportional to shear strain [29]. Ju et al studied the effects of time history, strain amplitude, frequency, shear rate, and temperature on the MR gel normal stress at both variable and constant magnetic field using an advanced rheometer. The results have shown that the magnetic field had the most significant impact on normal force behaviors when compared to other factors. In the constant magnetic field, remaining factors have the weak effect on the normal stress [21]. Laun et al compared the difference between the normal force of the first and second MRF for the MRF with a volume fraction of 50%, concluding that the normal stress in static condition increases without shearing, according to power law: $F_N \propto B^{2.4}$. Moreover, two normal stresses are positive under stable shear at 10 s$^{-1}$; the normal stress is almost five times greater than the shear stress [30]. Based on the above-mentioned literature investigation, it can be verified that researchers mainly focused on the viscoelastic properties and shear stress of MR grease, normal stress behavior in the MR fluids, MR elastomer and MR gel. However, researches on the normal force of MR grease in static and dynamic modes are considerably rare.

In this study, the authors studied the physical properties (microstructure, magnetic features) of MR grease, Additionally, by using a rheometer, the effect of time, magnetic field strength, shear rate, and temperature on the normal stress was measured and analyzed in the quiescent state and dynamic modes. The research results in this paper provide theoretical guidance for researchers in this field and guidelines for the design and manufacture of engineering machinery equipment.

2. Sample preparation and experimental principle

2.1. Materials preparation

MR grease is manufactured by mixing carbonyl iron powder and commercially available grease. This preparation method makes MR grease almost no sedimentation. In this paper, carbonyl iron particles with an
average particle size of 6 μm produced by BASF and lithium grease (carrier medium) made in China are chosen. The detailed physical characteristic of the grease are presented in table 1, while the prepared MR greases with specific compositions are shown in table 2. As can be seen from table 2, the CI particles weight fraction were selected to be 30%, 50%, 70%.

First, the lithium-based grease is poured into a vial, and then use a mechanical stirrer to stir at 500 rpm for 30 min while keeping the temperature at 60 °C. After the mixing is complete, the CI particles are added to the grease base in proportion. Finally, a stirrer device is used to mix the MR grease ingredients until the grease and CI particles are completely fused.

2.2. Experiment apparatus and methods
Three tests were carried out to determine the MR grease characteristics, focusing on microstructure, magnetic properties, and mechanical properties. A scanning electron microscope (Model Quant 250FEG, SEM) was applied to measure the microscopic morphology of CI particles and MR grease. Vibrating sample magnetometers (Lakeshore Cryotronics Inc. model 7404 series VSM) was used to test the magnetic features of three MRGs; the test was conducted at room temperature. Normal stresses of MR grease were measured using a rheometer (Physica MCR 301); the device was installed with a water bath temperature control system (JULABO F25) and a current accessory system. In this experiment, the rheometer is equipped with related accessories to control the temperature and magnetic field. The sample is placed between two parallel disks with a diameter of 20mm and a spacing of 1mm, and the magnetic field passes through the sample perpendicularly. The normal force data is obtained by a sensor placed on an air bearing in the rheometer. The sensor has a range of −50 N to 50 N, with an accuracy of 0.03N.

During the experiment, the Anton Paar MCR301 rheometer was used to test the rheological properties of the magnetorheological grease. The experimental principle is shown in figure 1. The sample needs to be pre-sheared before each test to ensure the accuracy of the experimental data. In this paper, the shear rate was set to 1 s⁻¹, aiming to keep the sample well dispersed. After stopping the shear, the magnetic field was set to 60 s to ensure that the sample forms a stable structure. It should be noted that, except for the experiment on the influence of temperature on the normal force, all experiments in this paper are carried out at a temperature of 25 °C.

3. Results and discussion

3.1. Material features
SEM was applied to independently observe the carbonyl iron powder and MR grease microstructures in the absence of magnetic field. The gray in figure 2(a) and (b) indicates the position of carbonyl iron powder, while the black background indicates the position of grease. Obviously, figure 2(a) shows that the carbonyl iron powder is spherical, and because the van der Waals force between the carbonyl iron particles is large, there are many small particles adhering to the surface of the larger particles. In addition, it can be seen from figure 2(b) that the CI particles are well dispersed in the grease matrix.

The magnetic properties of the three MR grease samples were measured by VSM, and the results are shown in figure 3. When the applied magnetic field strength reaches 700 kA m⁻¹, MRG-70 showed the highest saturation magnetization, 135.8 emu g⁻¹. Moreover, as the content of pure carbonyl iron powder in the grease
Figure 1. Testing principle for normal force of magnetorheological grease.

(a) Pure carbonyl iron powder          (b) The microstructure of MR grease

Figure 2. (a) The microstructure picture of the carbonyl iron powder and (b) the picture of MR grease as observed by SEM.

Figure 3. Magnetization curves of magnetorheological grease of different samples.
increases, its magnetization value will also increase. The reason for such behavior is because research has shown that pure CI particles have a high saturation magnetization \(206 \text{ emu g}^{-1}\) \[7\]; therefore, if the MR grease contains a higher share of CI particles, its magnetization becomes higher—as reported by Mohama \[1, 31\].

3.2. Static normal force without shearing

3.2.1. The influence of testing time on normal stress

As shown in figure 4, the relationship between normal force and time was measured in the no-shear mode. In the initial stage, the value of the normal force with time fluctuates around zero. When the magnetic field intensity is increased, the normal stress will increase sharply, and once the magnetic field is removed, it will drop quickly to practically zero. The normal stress was appeared because of the ordered structures created in response by applied magnetic field strength. When the magnetic field strength increased, more chain-like and columnar structures formation resulted in the intensification of normal stress \[27\]. After losing the magnetic field response, the magnetic particles in the MR grease will be arranged disorderly, and the normal force value at this time is almost close to zero.

As shown in figures 5(a) and 5(b), when no magnetic field is applied to the MR grease, the magnetic particles are free to move in the carrier liquid; when the magnetic field is applied to the MR grease, magnetic particles will instantly form a long chain in the magnetic field direction. The formation of different long chains will push away the rotating disk and increase the normal force. Moreover, as shown in figure 4, as the intensity of the magnetic field increases, the normal stress also increases. A similar phenomenon can also be found for MR gel \[21\] and MR suspensions \[26\]. While the added magnetic field strength is 96 kA m\(^{-1}\) and 194 kA m\(^{-1}\), the normal force decreases slightly with the test time. Once the test magnetic field intensity is 391 kA m\(^{-1}\), the normal stress slightly increases with the test time. However, at a magnetic field intensity of 740 kA m\(^{-1}\), the normal force...
produces minor fluctuations as time progresses. This conclusion is consistent with Yao’s research on MR fluid [27]. Said fluctuations are caused by the sudden breaking and recombination of the MR grease particle chains under high magnetic fields strength. At the same time, the increase in the magnetic field promotes the generation of long chains, inevitably leading to a struggle between the interaction forces of CI particles and viscous forces of MR grease. When the relationship between the viscous force and particle interaction reaches equilibrium, the normal force becomes a stable value [32].

3.2.2. Effect of the magnetic field on normal stress
The value of MR grease normal stress in the absence of shear, as a function of magnetic field strength, is shown in figure 6. In the same MRG sample, the normal stress value was obtained by scaling the magnetic field intensity from the 0 kA m\(^{-1}\) to 740 kA m\(^{-1}\), followed by a drop to 0 kA m\(^{-1}\). Compared with the normal force value measured when the magnetic field intensity increases from 0 kA m\(^{-1}\) to 740 kA m\(^{-1}\), the normal force value is higher when the magnetic field is descended from 740 kA m\(^{-1}\) to 0 kA m\(^{-1}\) (figure 6). It can be seen from figure 6 that the larger the content of carbonyl iron powder particles, the larger the resulting hysteresis area. This is because the strength of the generated magnetic chain is related to the magnetic particles. This phenomenon is caused since the increase in iron powder content produces both the higher number and more durable particle chains. Furthermore, when the magnetic field intensity reaches 740 kA m\(^{-1}\), the normal force values of samples MRG-30, MRG-50, and MRG-70 reach the maximum values of 7.77 N, 10.4 N, and 14.72 N, respectively. This shows that the higher magnetic field and larger particle content induced the greater normal force. The similar results were confirmed that the normal stress would be increased with enhancing the particle fraction and magnetic fields [26, 32].

3.2.3. The effect of temperature on the normal stress
In some cases, the temperature range is an important parameter when assessing MRG performance; it directly determines the application range and application effect of various MR devices [33]. Additionally, MR grease mechanical properties are directly related to the temperature, meaning that its performance varies significantly as the temperature changes (i.e., normal force). Thus, it is essential to investigate the effect of temperature on the normal force. The experimental temperatures range between 10 °C and 70 °C, and the values of MRGs normal force were measured at various magnetic field strengths, as seen in figure 7.

Figure 7 shows that in the absence of a magnetic field, the normal force is stable as the temperature changes. Once the magnetic field was applied, the normal stress value fluctuates with the change in temperature. Exactly speaking, the normal stress generates the evident fluctuation with changing the temperature when magnetic field strengths of 96 kA m\(^{-1}\) and 194 kA m\(^{-1}\), but, the fluctuation is very small when the magnetic field strengths are 0 kA m\(^{-1}\), 391 kA m\(^{-1}\) and 740 kA m\(^{-1}\). This phenomenon is caused by the balancing between the Brownian force and magnetic stress under the combined action of the magnetic field and temperature; the breakage and reconstruction of the magnetic particle chain cause the fluctuation in normal force. The magnetic particles in the material will generate long chains under the action of magnetic force. When the temperature is higher, the viscosity will be lower and the Brownian motion will become violent, which increases the chance of changing the position of the particles and the interaction between the particles, thus leading to an increase in the normal force. Moreover, it was shown that at a certain temperature, normal stress increases with the increase in the magnetic force.
field intensity, which is consistent with section 3.2.2 results. As the magnetic fields increasing, the repulsive force among magnetic particles might be strengthened and lead to the larger normal stress to push away the plates.

### 3.3. Dynamic normal stress in steady shear mode

#### 3.3.1. The effect of test time on the normal stress

Effect of time on the normal stress in MR grease with 70% weight content of CI particles is observed at different constant shear rates (figure 8). The total test time was 1020 s and the magnetic field strength is 740 kA m\(^{-1}\). The shear rates were 0.01 s\(^{-1}\), 0.1 s\(^{-1}\), 1 s\(^{-1}\), 10 s\(^{-1}\) and 100 s\(^{-1}\), respectively.

It can be observed from figure 8 that the normal force is practically zero in the absence of a magnetic field. Once the magnetic field is applied, the value of the normal stress will instantly increase. Surprisingly, when the magnetic field is eliminated, the normal stress practically reaches zero once again. Such behavior is caused by the lack of long-chain structures in the direction of the magnetic field (since there is no field). Thus, the normal force diminishes significantly; on the other hand, when the magnetic field is applied to the sample, it rapidly forms a chain structure, generating the normal force. This phenomenon is similar to the static normal force. It can also be observed from figure 8, the dynamic normal stress is only increased with prolonged the time at 100 s\(^{-1}\) shear rate under the magnetic field strength of 194 kA m\(^{-1}\) and at 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) shear rate under the magnetic field strength of 740 kA m\(^{-1}\); however, an increase in shear rate does not necessarily increase the normal stress. For example, the normal stress at a shear rate of 1 s\(^{-1}\) was higher than the normal stress at 10 s\(^{-1}\) and 100 s\(^{-1}\). The reason for such behavior the elimination of the MR grease chain structure under a high shear rate; therefore, the

![Figure 7](image1.png)  
**Figure 7.** The trade-off between the static normal force and temperature in MRG-70 under various magnetic field intensities.

![Figure 8](image2.png)  
**Figure 8.** Diagram of the relationship between normal force and the time of MRG-70 at constant shear rates. (a) \(H = 194\) kA m\(^{-1}\); (b) \(H = 740\) kA m\(^{-1}\);
normal stress was diminished. Once a certain threshold was met, the chain was re-formed with the addition of a magnetic field, finally reaching equilibrium value.

3.3.2. The effect of magnetic field on the normal stress
The relationship between the normal force and the magnetic field strength of the three samples at a shear rate of 50 s\(^{-1}\) was observed. The magnetic field strength increased from 0 kA m\(^{-1}\) to 740 kA m\(^{-1}\) and then decreased to 0 kA m\(^{-1}\) (figure 9). According to the figure, it can be observed that the normal force value increases with the increase of the magnetic field strength. The normal stress under removing magnetic field is slightly higher than the stress at the applied magnetic field. This behavior is similar with the static normal behavior. In addition, the hysteresis area of the normal dynamic force is smaller than the hysteresis area of the static normal stress. This is because under the action of dynamic shear, its magnetic force is much higher than the viscous force, so the structure of the particles is relatively stable, which will reduce the hysteresis. The smaller the hysteresis would occur in the greater shear rate [34]. Moreover, in figure 9, MR grease dynamic normal force increases as the CI particle concentration and magnetic field intensity increase. Yao X [27] explained the reason for this behavior, that is, under the action of a magnetic field, MR grease will produce chain-like structures. If the concentration of magnetic particles increases, the chain structure will increase, and the chain structure will appear to push the rheometer plate apart, thereby increasing the value of the normal force. In turn, that causes the normal stress generated by the test sample to push the rheometer plates apart. Compared with samples with high mass scores, compared with samples with higher quality scores, the overall upward trend of samples with lower quality scores is more stable, because samples with lower quality scores require a lower magnetic field to reach saturation.

The relationship between the magnetic field and the normal stress for MRG-70 (under various shear rate) is shown in figure 10. Obviously, the normal stress increases with the magnetic field strength at different shear rates. In addition, the normal force in steady-state shear does not consistently increase with the shear rate. It can be seen from figure 10 that in the low magnetic field, the shear rate has little effect on the normal force. Because the value of the normal force mainly depends on the strength of the magnetic field, and the shear rate is not enough to make the material structure in the MR grease change greatly. Under the medium magnetic field intensity, the normal force increases with the increase of the shear rate, while under high magnetic field strength, the normal force values of shear rate at 10 s\(^{-1}\) and 100 s\(^{-1}\) are almost the same. This is because under the influence of high magnetic field and high shear, the particle chain of the material is more stable, that is to say, the breakage of the material particle chain during the shearing process is balanced with the formation of the chain structure under the action of the magnetic field. so the normal force value does not change.

3.3.3. The effect of shear rate on the normal stress
The effect of shear rate on the normal force in MRG-70 specimen is illustrated in figure 11. Five magnetic fields strengths (0 kA m\(^{-1}\), 96 kA m\(^{-1}\), 194 kA m\(^{-1}\), 39 1 kA m\(^{-1}\), and 740 kA m\(^{-1}\)) are applied on MR grease. The shear rate scan is used to evaluate the normal stress under various magnetic fields strengths with a shear rate ranging from 1 s\(^{-1}\) to 100 s\(^{-1}\) (rotational shear mode is used). As showed in figure 10, the dynamic normal stress closely relies on the magnetic field strength under the shear. Additionally, the authors observed that the normal force remained constant at magnetic field strengths of 96 kA m\(^{-1}\) and 0 kA m\(^{-1}\). When the magnetic field
strength is greater than 96 kA m$^{-1}$, the value of the normal stress will fluctuate. This is because when shear exists, the magnetic particle structure will be destroyed or reconstructed under the action of shear force and magnetic force. When the magnetic field strength is relatively small, this relationship will be balanced. Finally, it is worth noting that this phenomenon is inconsistent with the results of the MR gel study of Ju [21].

### 3.3.4. Effect of temperature on normal stress

When the magnetorheological device operates, the material undergoes constant shearing movement, causing the temperature to increase, and magnetorheological grease mechanical properties have a direct relationship with the temperature [35]. Thus, the material performance under depends on the temperature conditions, affecting the performance of magnetorheological devices. Therefore, it is necessary to consider the effect of temperature on the normal force in dynamic shear mode (figure 11). The experimental temperature range is 10 °C to 70 °C and the shear rate is 50 s$^{-1}$.

As shown in figure 12, the research results of temperature on the normal force of MR grease are similar to those of MR gel [21], when the magnetic field intensity was 0 kA m$^{-1}$ and 96 kA m$^{-1}$, the normal stress value hardly changes with the temperature. However, once the magnetic field intensity reaches 194 kA m$^{-1}$ and 391 kA m$^{-1}$, the normal stress increases with the temperature. This is primarily caused since higher temperature decreases the material viscosity; therefore, more particles form chains, enhancing Brownian motion and increasing the normal stress. When the magnetic field strength reaches 740 kA m$^{-1}$, the normal force fluctuates with the increase of temperature; This is because as the temperature increases, the interaction between iron
particles becomes larger, and the influence of magnetization will be weakened. In addition, the temperature effect will cause the magnetic particles to produce brownian motion, the competition between brownian motion and magnetism will cause the normal force to fluctuate. Comparing with figure 7 and figure 12, it can be observed that the static and dynamic normal force are not affected by temperature in a zero field. This behavior might be due to the absence of magnetic chains at off-state magnetic field. In the medium and high magnetic fields, the dynamic stress generated more obvious fluctuation than the static stress with changing the temperature.

4. Conclusions

This paper mainly introduces the normal force characteristics of MR grease in static and dynamic modes, and studies the influence of time, magnetic field, shear rate and temperature on the normal force of MR grease. It was found that the static normal force is related to the magnetic field strength, but has nothing to do with time. In addition, when testing the effect of temperature on the normal force, it is found that the normal force is in a stable state when the normal force is in a zero field. After the magnetic field is applied, the value of the normal force will fluctuate with temperature changes. In the steady-state shear conditions, dynamic normal force is related to magnetic field strength, particle content, time and shear rate. Among them, the relationship between normal force and magnetic field is similar to the static mode, but the difference is that the hysteresis area of the normal dynamic force is smaller than the hysteresis area of the static normal stress. Furthermore, the temperature has no effect on the normal force in a low magnetic field, but it is higher than 96 kA m$^{-1}$, the value of dynamic normal stress increases with temperature.

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