Prediction and analysis of thermal aging behavior of magnetorheological grease

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Abstract

Magnetorheological grease (MRG) is a new type of field-response intelligent material with controllable performance and excellent settlement stability, which is feasible to replace traditional materials. The heating phenomenon of magnetorheological (MR) devices is more common during operation and the influence law of continuous thermal effect (thermal aging) on the performance of MRG needs to be studied. In this article, the effect of thermal aging behavior on the rheological properties of MRG has been investigated. Accelerated heat treat the sample and test the shear stress under the condition of thermo-magnetic coupling. To reduce the time and cost during the study of MR materials, an improved and reliable artificial neural network (ANN) prediction model was developed to characterize and predict the relationship among temperature, aging time, magnetic field strength and the thermo-rheological properties of MRG. The test results of MRG before and after thermal aging show that thermal aging causes irreversible structural damage and the performance decreases with increasing aging time. The comparison of the ANN prediction results with the test results, the correlation coefficient R reached and exceeded 0.95. The results showed that the model had excellent prediction accuracy and could provide theoretical reference for the thermal aging behavior of MRG.

1. Introduction

MRG is a field-response intelligent material. It has gained great attention of researchers because of its salient controllable properties and potential applications to various fields such as automotive industry, aerospace and military sector [1]. MRG uses commercial grease as a carrier liquid, which is structural colloidal dispersion system with special rheological properties in the process of flow [2]. The unique soap fiber structure of lubricating grease can effectively solve the settlement problem of magnetic particles [3]. MRG can be used as a working medium for some special-purpose MR devices due to its unique structural system.

In fact, researchers have conducted extensive research on MR devices. MR devices are mostly energy-consuming devices in the working process, inevitable temperature rise effect in the actual service process of MR devices. MR material is a composite system and temperature is an important factor affecting the properties of polymer composites [4]. Temperature increase has a large impact on the rheological properties of the MR medium. The research work of Rabbani et al [5] confirmed that the shear stress of MR materials decreases significantly with increasing temperature. Chen et al [6] studies of magnetorheological fluid (MRF) have found that magnetic particle chain formation is strongly influenced by temperature. The rheological properties of MRG is also influenced by temperature. The studies of Sahin et al [7] showed that temperature has a significant effect on the yield stress and apparent viscosity of MRG. Wang et al [8] observed that temperature has an effect on the apparent viscosity, and this effect decreases with the increase of magnetic field strength. Pan et al [9] found that MRG and grease have similar thermal rheological characteristics. The yield stress and consistency coefficient decrease with the increase of temperature. Yang et al [10] found that the interaction of the grease soap fiber structure and magnetic chain will have an effect on the shear stability of MRG. We expect the MRG to
maintain excellent stability as a MR medium during long-term service. Especially in engineering applications, long-term stable and reliable service is an important indicator for MR devices [11]. What is not ideal is that Zheng et al. [12] found that long-term storage under high temperature causes changes in the viscosity of MRF and this phenomenon that constitutes the main failure modes of MRF. After long-term heat treatment, the degradation and structural damage of the base carrier liquid of MRG will occur [13], which have an effect on the whole MRG system. Combining previous research results, temperature is the main factors affecting the performance of MRG but the effect of long-term heat treatment on the performance of MRG needs to be further studied. MRG as a composite system inevitably faces structural deterioration and even thermal aging effects under long-term thermal effects. Aziz et al. [14, 15] experimentally evaluated effects of thermal aging on polymer composites under long-term elevated temperature exposure. Experimental results show that no significant changes of loss factor occurred at a low CIPs concentration, whilst the loss factor increased at a higher CIPs concentration [14] and over a long-term of thermal aging, the rubber becomes hard, thus influencing the properties of the materials [15]. However, the current research on MRG mainly focused on the influence of temperature and magnetic field, and the influence of continuous thermal effect (thermal aging) was ignored. Therefore, it is necessary to investigate the performance changes of MRG before and after thermal aging. This is valuable for assessing the stability and reliability of MRG for long-term service.

The experimental test is the main research means in the research process of MRG, it takes plenty of time, manpower and financial resources. ANN is a computational model with self-adaptability, self-organization and self-learning properties [16], and its output depends on connection mode, weight value and incentive function. In its application, Erdil et al. [17] used ANN model to predict meteorological variables and achieved the reliable prediction of meteorological value through the training and testing of ANN model. Actually, in the research field of carrier liquid grease of MRG mentioned in this study, Lijesh et al. [18] proposed a method to evaluate the hydrophobicity of grease to save time and materials, and quantified the hydrophobicity of grease by using the contact angle of water droplets on the surface of grease. Osara et al. [19] derived the basic formula for the instantaneous nonlinear response of grease to load according to the law of thermodynamics. The grease model obtained is close to 100% agreement with the nonlinear data measured by uncontrolled experiments. Son et al. [20] evaluated the life of ball bearing grease according to the changes of temperature, speed and load, and developed and theoretically verified the ball bearing grease life testing machine. Rezasoltani et al. [21] applied the theory of irreversible thermodynamics to study the mechanical degradation of grease under shear. The verification results show that there is a linear relationship between degradation rate and entropy production, which can be used to estimate the life of mechanically degraded grease. In the existing studies, the grease model and grease life estimation obtained based on thermodynamic theory have a certain degree of confidence. Zheng et al. [12] used the improved CGP algorithm to study the effect of high temperature on the rheological properties of grease-based MRF and predicted its lifetime. Bahiuddin et al. [22] proposed a constitutive model of MRG to predict the variation of shear stress and dynamic yield stress with temperature. In the follow-up research, they proposed a model based on machine learning method to characterize and predict the relationship between the rheological properties of MRG and shear rate, magnetic field and its constituent elements [23]. Saharuddin et al. [24] proposed a new constitutive model for the viscoelastic behavior of MRE. The machine learning method is used to predict the stiffness and damping characteristics of MRE varying with the magnetic field. With regard to the effect of thermal aging time on the rheological properties of MRG, there are few literatures about the effect of thermal aging time on the rheological properties of MRG. And the nonlinear regression model of multi-feature input and single-feature output does not fully cover the thermal aging time and magnetic field strength.

According to the above research work, introduce the corresponding prediction model to predict the rheological properties of MRG is valuable. The prediction model can save a lot of time and cost for research related to MRG. The purpose of this paper is to propose an ANN prediction model to predict the shear stress of MRG after thermal aging based on the rotational rheometer test results. The results of the study can provide reference for MRG performance prediction and evaluation after being thermal aged.

### 2. Experiments and methods

#### 2.1. Materials Preparation

The MRG in this study is prepared in the laboratory, using grease as carrier liquid and CI particle as magnetic particles. The specific preparation method is in accordance with the method proposed by Mohamad et al. [25]. The exact weight of grease was weighed and placing it in a beaker. Then the grease was stirred with a mechanical stirrer at a temperature of 80 °C (ten minutes, 500 rpm). Subsequently, CI particles with a weight fraction of 30% were added to the grease and stirred with the same mechanical stirrer (800 rpm) until the grease and CI particles were fully and evenly mixed. MRG for experiment can be obtained after cooling.
A lithium grease was selected as the matrix to suspend the CI particle, which was manufactured by Lubricant Tianjin Company, Sinopec Lubricating Oil Co., LTD (China). The main components and technical parameters of the lithium grease were listed in table 1. The weight fraction of CI particle is 30%. The CI particle (type MRF15) was purchased from Jiangsu Tianyi Ultrafine Metal Powder Co., LTD (China). The parameters for the MRF 15 CI particle were given in table 2.

2.2. Experimental method
2.2.1. Static thermal aging
For the static thermal aging process of MRG, the MRG was smeared on the inner wall of the beaker with a capacity of 150 ml (thickness is 1 ∼ 3 mm). Placed at heating and drying oven (DHG-9623A from Shanghai Jing Hong Laboratory Instrument Co., LTD, China) and heated at 120°C for 4 h, 8 h and 24 h. The thermal aging samples were numbered MRG-0h, MRG-4h, MRG-8h and MRG-24h.

Table 1. Main components and technical date for lubricating grease.

| Properties                        | Specification          |
|-----------------------------------|------------------------|
| Thickener type                    | Lithium 12-hydroxystearate |
| Thickener % (w/w)                 | 9.3                    |
| Base fluid                        | Mineral                |
| Lubricating oil viscosity (mm²·s⁻¹) | 100                   |
| ASTM D-445 (40 °C)                |                        |
| Dropping point (°C) ASTM D-566    | 178                    |
| Worked penetration (dmm) ASTM D-217 | 325                |
| Consistency NLGI grade            | 1                      |

Table 2. Technical parameters for MRF 15 CI particle.

| $D_m$ (average diameter)/μm | Particle size distribution |
|-----------------------------|---------------------------|
|                             | $D (\leq 2.5 \mu m)$ | $D (\leq 4.5 \mu m)$ | $D (\leq 8 \mu m)$ |
| 3 ~ 5                       | 10%                      | 50%                    | 90%                    |
2.2.2. Rheological property
Rheological measurements of MRG were performed in a controlled-shear and controlled-strain rheometer (Physica MCR 302 from Anton Paar, Germany) by using a plate-plate geometry (PP20/MRD, 20 mm diameter, 1 mm gap), as shown in figure 1. The magneto-controllable accessory (MRD 180) and temperature control unit (JULABO F25) were used to control magnetic field strength and temperature during the process of rheological experiment, respectively. The magnetic field of MRD 180 accessory was generated by controlling the current of experimental system, and the values 0 A, 1 A, 2 A, 4 A, 5 A are corresponding to 0 mT, 220 mT, 440 mT, 880 mT, 1100 mT, respectively. The magnetic sweep test, at different temperatures (25 °C, 45 °C, 65 °C, 85 °C), were carried out in an electricity range comprised between 0 A and 5 A, with a constant shear rate 10 s\(^{-1}\) for 300 s. The shear stress of MRG before and after thermal aging was tested at different temperatures with the variation of magnetic field strength.

2.3. Experimental results
The relationship between shear stress and magnetic field strength of MRG before and after thermal aging at different temperatures is shown in figure 2. It can be seen that the MRG shear stress increases with the increase of magnetic field strength, and tends to be smooth after the magnetic field reaches saturation. The operating temperature during service is a key factor affecting the performance of MRG, and the shear stress decreases significantly as the test temperature rises. At 25 °C, the shear stress of MRG is significantly higher than other temperature conditions, which may be due to the high entanglement of grease soap fibers at low temperature, which hinders the movement of magnetic particles. In addition, the magnetic field scanning curve of MRG-24h at 25 °C were different from other samples at the same temperature, and similar to those at 85 °C, indicating the thermal aging at 24 h caused irreversible damage to the structure of MRG.

After the magnetic field strength reaches 440 mT, the trend of the magnetic field scanning curve tends to be smooth, which is close to the magnetic field saturation. The shear stress varying with the magnetic field strength reached a maximum value and remained constant. This phenomenon is mainly due to the magnetic particles in MRG are formed into magnetic chains along the magnetic field direction under the action of the magnetic field, and the number of magnetic chains reaches the maximum after the magnetic field is saturated. In the case of magnetic field saturation, there was no obvious shear yield phenomenon in MRG-0h, but the shear yield
phenomenon began to occur owing to the structural damage caused by thermal aging, among which MRG-24h had the earliest shear yield and was the most obvious.

3. Prediction model of thermal aging properties of MRG

To obtain the relationship between temperature, aging time, magnetic field strength and shear stress of MRG, an ANN prediction model was established. The model can be approximately regarded as a nonlinear continuous function. The nonlinear function can be expressed as the function of the variation of the shear stress of MRG with the magnetic field strength at different experimental temperature and aging time, as shown in equation (1):

\[
F \approx f_{T,m}(t)
\]

where \(F\) is shear stress, \(t\) is aging time, \(T\) is experimental temperature, and \(m\) is magnetic field strength.

ANN has a strong ability to approximate nonlinear functions, good accuracy can be obtained after data training [26]. Therefore, it is proposed to use ANN to complete the approximate fitting of this function, so as to provide relevant reference data for future MRG research and engineering applications. To further explain the working principle and implementation of the ANN prediction model, the implementation flow chart of the ANN prediction model is given, as shown in figure 3. Firstly, the original data need to be obtained through the rheological experiment on MRG after thermal aging. The architecture of ANN should be designed based on the characteristic of the experiment data, and the appropriate activation function and loss function should be selected [27]. The preliminary construction of the overall architecture of the network has been completed and the neural network will calculate the shear stress prediction value after input data and compare it with the experimental date.
3.1. Network architecture

In the process of setting the neural network architecture, the number of neuron nodes in the input layer is set to 3, because the input characteristics include temperature, aging time and magnetic field strength. The output is characterized by the predicted shear stress of MRG, so the number of neuron nodes in the output layer is set to 1. In addition, according to the Kolmogorov theorem \[28\], the formula for calculating the reference value of the number of nodes in the hidden layer is shown in equation \((2)\):

\[ P = 2n + 1 = 2 \times 3 + 1 = 7 \]

where \(P\) is the reference value of the number of nodes in the hidden layer, \(n\) is the number of neuron nodes in the output layer.

In addition, in order to improve the generalization ability of the network and make it adapt to the complex objects, an additional hidden layer is added. The architecture of ANN is shown in figure 4 as: \(3 \times 7 \times 1\).

3.2. Training ANN

Input part of the data from the magnetic field scanning experiment into the ANN, randomly select 70% of the data for training and 30% of the data for verification and testing. As the data is substituted into the ANN, the model will derive the predicted value and compare it with the experimental value, and then calculate the mean square deviation by the loss function (as shown in equation \((3)\)), making it lower than the given value. When the value is higher than the given value, the mean square error is used to modify the weights in the neural network. After the loss value is lower than the given value or reaches the given iteration period, the training is finished and the image of the loss value with the iterative period can be obtained at the same time, as shown in figure 5. It can be found from the chart that the mean square error of the ANN prediction model tends to be fixed after the number of training reaches 20 times.

\[ L(Y|F(x)) = \sum_{N} (Y - F(x))^2 \]

3.3. Prediction results

The ANN prediction model was trained by using the experimental data of temperature, aging time and magnetic field strength as input features and the experimental data of shear stress as a single output feature. The established model takes the shear stress as a single prediction result to characterize the performance of MRG under different temperature and magnetic field strength after thermal aging. The experiment date which is not trained by the ANN prediction model is used as the verification experiment, and the experimental conditions (temperature, aging time and magnetic field strength) of the verification experiment are used as input characteristics to predict the shear stress with the trained ANN model, and the predicted shear stress is output.
The correlation coefficients $R$ under different experimental conditions are shown in Table 3, and Figure 6 shows the experimental and prediction results. The results of the magnetic field scanning are given in the figure. In general, it can be seen that the shear stress of the MRG increases as the magnetic field increases, and the shear stress reaches a constant value when the magnetic field strength exceeds a certain value (440 mT). This outcome is observed mainly due to the MR effect of MRG. The stronger the magnetism is, the higher the number of magnetic chains will be, thereby eventually reaching saturation. The predicted model shows a good correlation with the experimental values, especially under the unsaturated magnetization.

Comparing the predicted value with the experimental value, it can be seen from Table 3 and Figure 6 that the ANN model proposed in this work can predict the shear stress with the $R$ accuracy up to 0.99. The prediction results strictly show the theoretical trend of magnetic field scanning. The experimental results decrease slowly under saturation magnetization, but then recover to the original level. Especially, the trend of graph at 24h–65°C displays a different trend from other groups. The predicted progress of MRG at magnetic field strength from 0 to 1100 mT shows excellent predictions at 0 h, 4 h, and 8 h, but there is a great discrepancy between the predicted value and the experimental value at 24 h. Further discussion will be given as following.

Table 3. The accuracy of the predicted shear stress at different experimental conditions for ANN.

| Test conditions | $R$ (ANN) |
|-----------------|-----------|
| Test temperature (°C) | Thermal aging time (h) | 0 ~ 440 mT | 0 ~ 1100 mT |
| 65 | 0 | 0.989 | 0.992 |
| | 4 | 0.995 | 0.992 |
| | 8 | 0.996 | 0.994 |
| | 24 | 0.996 | 0.840 |

Figure 6. Comparison of experimental and predicted results.
The structural recovery properties of MR materials are more dependent on magnetic field strength and temperature [29]. The test temperature in figure 6 is 65 °C. When the magnetic field strength reaches saturation, the error between the experimental and predicted values of MRG-24h shear stress increases with the further increase of magnetic field strength. Theoretically, as the magnetic field increases to saturation, the shear stress should remain constant. The predicted results are exactly this trend. MRG uses grease as a carrier liquid which shows excellent settling stability due to the soap fiber structure. The soap fibers create the link between the magnetic chains. As the thermal aging time increases, the soap fiber entanglement will be changed. Shen et al [30] discovered that the stability of the grease structures deteriorated with prolonged ageing time, and the net structures of fiber rebuilt slowly after shearing. For MRG, the link between the magnetic chains is weakened along with shear stability variation of the grease structure. Therefore, the shear thinning trend of graph at 24h-65°C is mainly attributed to the change of grease fibrous entanglement. With the thermal aging time increased, the damage degree of MRG structural increased. The MRG samples showed fracture phenomenon and shear thinning with increase of shear time. Thus, the experimental results are lower than the predicted values.

In addition, Wang et al [31] observed that under the action of magnetic field, the nonlinear behavior of MRG is related to the shear frequency. Therefore, in the follow-up research work, test variables such as shear time and shear rate should be considered as input features and the prediction model should be further optimized.

4. Conclusion

Through the continuous heat treatment of MRG at 120 °C for 4 h, 8 h and 24 h, the plate test head of rotational rheometer was sheared at a constant rate under different temperature and magnetic field strength and the change of shear stress was observed. An ANN prediction model was established to predict the rheological properties of MRG after thermal aging treatment. The following conclusions can be drawn:

1) The effect of thermal aging behavior at 120 °C on MRG is mainly manifested in the reduction of the degree of entanglement of the base carrier liquid grease soap fiber structure, the reduction of shear flow resistance, and the weakening of structural recovery performance. At relative low temperature, the entanglement degree of the carrier liquid grease soap fiber is high, which hinders the movement of magnetic particles. In the case of magnetic field saturation, there is no obvious shear yield phenomenon in MRG-0h, nevertheless, shear yield begins to appear owing to the structural damage caused by thermal aging, in which shear yield occurs earliest and most obvious in MRG-24h. Thermal aging causes irreversible structural damage to MRG, and the effect of this damage is further aggravated with the increase of shear time.

2) The ANN prediction model is introduced to predict the shear stress in the working process of MRG. The training task of the model is completed by taking temperature, aging time and magnetic field strength as input features. The comparison between the prediction results and the experimental results of the saturated magnetic field (440 mT) shows that the model has excellent accuracy in predicting the shear stress of MRG, ANN prediction model is feasible in practical applications.

3) The variation of MRG shear stress with heat treatment time is the main study of this paper. The output data of the ANN prediction model are consistent with the experimental data, indicating that the model can be used for the prediction of MRG rheological behavior and the development of related MR devices. However, the actual working conditions are more complex and involve frequent shearing and media loss, etc. In summary, the model can be further improved by introducing other input parameters, studying the normalization of data and activation functions. With the continuous improvement and optimization of the ANN prediction model, it is expected that future research will provide more informative prediction data for MRG materials and MR device design.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
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