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Thixotropy of MR shear-thickening fluids

Xianzhou Zhang[1], **Weihua Li**[1,3] **and Xinglong Gong**[2]

[1] School of Mechanical, Materials and Mechatronic Engineering, University of Wollongong, Wollongong, NSW 2522, Australia
[2] CAS Key Laboratory of Mechanical Behavior and Design of Materials, Department of Modern Mechanics, University of Science and Technology of China, Hefei 230026, People's Republic of China

E-mail: weihuali@uow.edu.au

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Abstract
Particle sedimentation is a key issue of conventional magnetorheological (MR) fluids. We recently fabricated MR shear-thickening fluids (MRSTF), which can work as novel MR fluids without particle settling. This merit of the material against particle settling is attributed to the thixotropy property. By using shear-thickening fluids as a base medium, a series of MRSTF samples was prepared and their rheological properties were tested. It was found that when the weight fraction of the STF base is above a threshold value of 15%, the MRSTF exhibits a significant thixotropy phenomenon, which greatly reduces the settling problem of MR fluids and consequently increases the stability of MR fluids. A theoretical approach was proposed to verify the experimental studies.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Magnetorheological (MR) fluids have attracted considerable interest for more than two decades. These materials exhibit an ability to change their flow characteristics by several orders of magnitude and in times of the order of milliseconds under the influence of an applied magnetic field. MR fluids have found important applications in a number of areas, including vehicle dynamics, aeronautics, smart structures, virtual reality, etc [1–7]. In MR fluid applications, a number of key issues should be taken into account. One issue is to enhance MR fluid field-dependent shear stress by investigating a number of influencing factors, like particle size, working mode, etc. Foister reported that MR suspensions composed of mixtures of magnetizable particles of two different diameters (1.25 and 7.9 μm) can provide a substantial increase in the field-induced yield stress without an increase in the viscosity of the mixture in the absence of a magnetic field [8]. Similar observations were reported by Weiss *et al* and Ulicny *et al* [9, 10]. Tang *et al* improved the yield stress of MR fluids by changing the fluid microstructure. Immediately after a magnetic field is applied, they compress the MR fluid along the field direction. The static yield stress can reach as much as ten times as high as that of the same MR fluid without compression [11]. Zhang *et al* studied the mechanism of the squeeze enhanced MR effect [12].

The second important critical issue is the low viscosity of MR fluids in the absence of a magnetic field. The difference between such off-state viscosity and on-state yield stress may be conveniently expressed as a 'turn-up ratio'. In MR fluids, the maximum force or torque 'on' is controlled by the yield stress while the minimum force or torque 'off' is controlled by the zero-field viscosity. The general aim in designing controllable fluid actuators is generally to maximize the turn-up ratio under given operating conditions [13].

The third, or perhaps the most important critical issue is the stability against sedimentation. Because of particle/carrier fluid density mismatch, the particle sedimentation is hard to remove. Particle sedimentation would greatly decrease the behavior of MR fluid, even cause failure. It is a vital shortcoming for MR fluid, especially for long-term inactive devices, such as an MR fluid damper for seismic control [14]. It is common for these dampers not to be used for tens of years under buildings, but when the earthquake comes they are likely to fail because of sedimentation of particles in the fluid, and this is a significant flaw.

A number of methods have been developed to reduce the sedimentation problem. These methods are classified into two categories: the modification of particles and the modification

[3] Author to whom any correspondence should be addressed.

of carrier fluids. For the first category, a simple method is to replace the micro-sized magnetic particles with sub-micro or nano-scale particles, such as nanotubes or nanowires [15–17]. This approach has greatly reduced the particle sedimentation. However, the MR effect is degraded significantly due to the particle size reduction. For example, the MR effect of nanotube based MR fluids is three or four orders lower than that of conventional MR fluids. Even for sub-micro particle based MR fluids, the MR effect is still one or two orders lower than in conventional MR fluids. To overcome this shortcoming, Fang *et al* [15, 18] have developed both polymer coating techniques and sub-size filler systems to fabricate a series of carbon iron based magnetic composite particles. The sedimentation ratio testing indicated that composite particle based MR fluids changed very little, from 100% to 98%, after 200 h while the sedimentation ratio of other conventional iron particle based MR fluids decreased quickly to 94% within 50 h. Also, the maximum yield stress of the composite MR fluids can be as high as 20 kPa.

As another important approach, the idea of the modification of the carrier fluid is to increase the viscosity of the carrying fluids. A general case is to replace the fluid carrier with a gel carrier. This kind of MR material is called an MR gel [19]. If the 'carrier fluid' is replaced with a solid status material, the fabricated MR material is called an MR elastomer [20]. Both MR gels and MR elastomers have a rather lower MR effect that that of MR fluids. The high 'viscosity' of MR gels and MR elastomers can greatly reduce or eliminate the sedimentation, but the high viscosity in the absence of magnetic field also decreases the relative MR effect.

The original idea is from our investigation of shear-thickening fluids (STFs). STFs are concentrated colloidal suspensions composed of non-aggregating solid particles suspended in fluids, and show a marked increase in viscosity beyond a critical shear rate. In addition, this increased viscosity is seen as being both 'field activated', due to the dependence on shearing rate, as well as reversible [21]. In our previous work [22], the thixotropic phenomenon of STF was observed. If the MRF is fabricated based on an STF, the iron particles may be held on the STF media while the MRF is not sheared and sedimentation can be avoided. The viscosity in the absence of magnetic field can also be reduced by shearing the fluid. This is our basic starting point.

Many earlier studies have focused on determining this 'field activated' viscosity and other rheological properties of STF under steady shear and oscillatory shear flow. Steady shear research indicated the common feature of the materials' rheograms is a sharp increase in viscosity that decays at higher shear rates, often referred to as a discontinuity. This increase occurs at a critical shear rate. In addition, the suspension behaves as a 'Newtonian' fluid during a large spectrum of shear rates, except for the transition shear rate period that causes the fluid's specific critical shear rate transition. These types of fluids have been characterized as behaving as quasi-Newtonian fluids with a low viscosity before the critical shear rate transition, and, again, with a quasi-Newtonian behavior but with a higher viscosity after the transition. There have been some researches into the dynamic properties of STFs. Laun *et al* reported the critical strain amplitude for dynamic shear thickening at a fixed angular frequency of a polymer latex dispersion [23]. Raghavan *et al* investigated the shear-thickening response of fumed silica suspension under steady and oscillatory shear [24]. Fischer *et al* investigated the dynamic properties of STF based on a vibrating sandwiched beam. Strain thickening refers to a sharp increase in the complex viscosity observed at critical combinations of strain amplitude and frequency [25]. However, the thixotropic phenomenon of STF is not considered to be an important kind of behavior. This is the major motivation of this work.

2. Experimental details

2.1. The fabrication of the STF base

The STF used was based on fumed silica (S5505, from Sigma-Aldrich) which has a primary particle size of 14 nm and a specific surface area of approximately 200 $m^2\ g^{-1}$. The carrier fluid was ethylene glycol [$HOCH_2CH_2OH$] with a density of 1.113×10^3 kg m^{-3} (102466, ReagentPlus®, from Sigma-Aldrich). In each case, the carrier fluid was added to the powder, and a blender was used to mechanically mix the two components for 1 h. The resulting suspensions were then placed in a vacuum chamber for several hours to eliminate any bubbles. Solid contents of 0%, 5%, 10% and 15% w/w (weight fraction) were selected for study.

2.2. The fabrication of the MRSTF

Carbonyl iron particles (Carbonyl iron, C3518, Sigma-Aldrich Pty Ltd) with 5 μm mean particle size were chosen as the magnetic particles in the MRSTF. The carrier phase is the prepared STF base. The preweighed solid iron microspheres were added in weighed amounts to form four MRSTF samples with weight fractions of 0%, 5%, 10%, and 15%, respectively. The particles were thoroughly mixed with the STF base under high shear conditions for approximately 10 min. The air bubbles in the resultant mixture were removed in a vacuum chamber before testing. The weight ratio of the iron particles and STF were 1:1.

2.3. Rheological testing

Both static and steady shear tests were conducted by using a parallel-plate strain-controlled rheometer (MCR 301, Anton Paar Companies, Germany), with a constant gap of 0.5 mm used in all tests. An electromagnet kit was embedded in the rheometer to generate a magnetic field, perpendicular to the shear flow direction. In this experiment, the rheometer operated in steady-state strain rate sweeps mode from shear rate at 0–100 rad s^{-1}. Also, quasi-static experiments with the strain ranging from 0% to 100% were conducted to study the stress–strain relationships. Seven different magnetic field strengths of 0, 0.5, 1, 1.5, 2, 2.5 and 3 ampere (A) were applied, where the magnetic flux density is represented as $B = 220I$ (mT).

Figures 1–4 show the steady shear and quasi-static shear results of the MRSTF sample based on the STF with solid contents from 0% to 15% w/w. From these experimental results, both static and dynamic yield stresses of the MRSTF

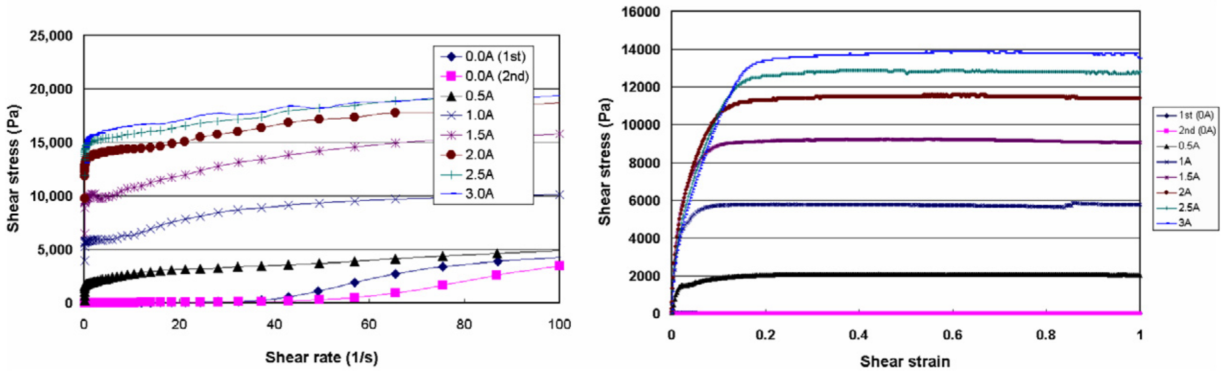

Figure 1. The steady shear and quasi-static shear results of the MRSTF sample based on 15% w/w STF.

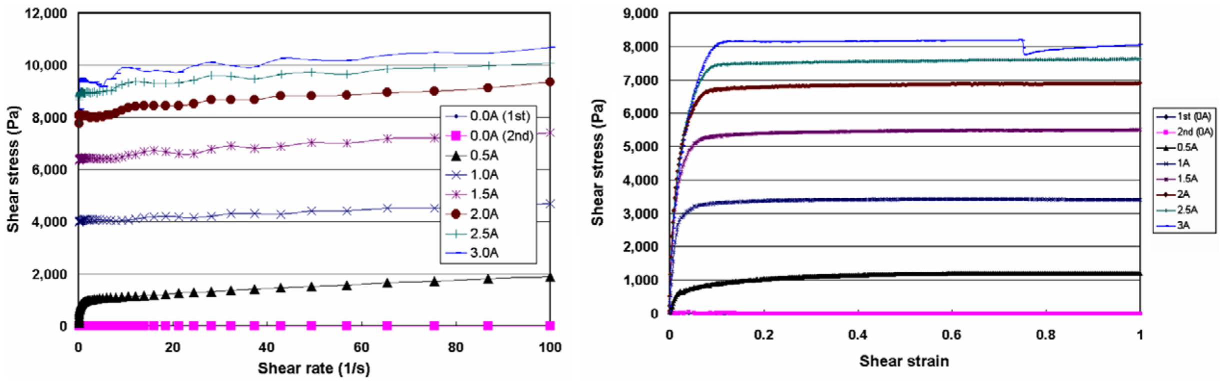

Figure 2. The steady shear and quasi-static shear results of the MRSTF sample based on 10% w/w STF.

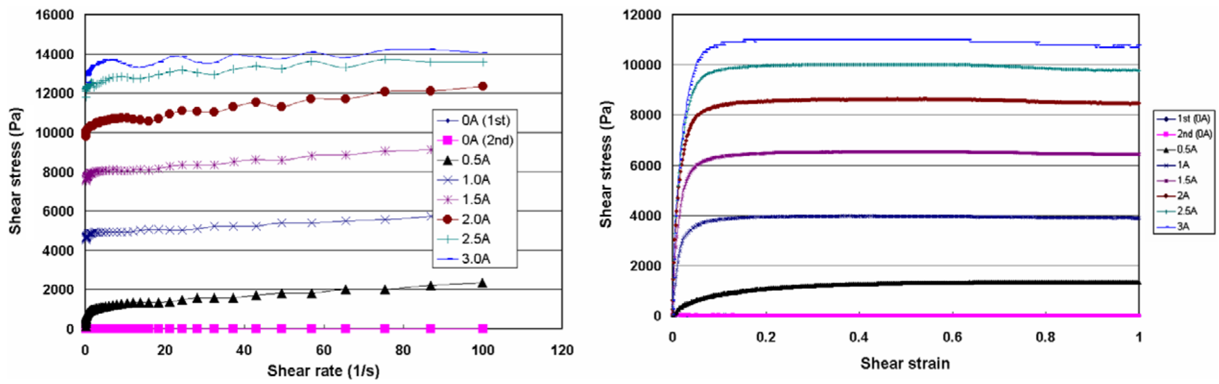

Figure 3. The steady shear and quasi-static shear results of the MRSTF sample based on 5% w/w STF.

sample with different w/w percentages were obtained and are shown in figure 5. It can be seen from this figure that the static yield stress is higher than the dynamic one, which is similar to conventional MR fluids. When the weight percentage is lower than 10%, the yield stresses show a decreasing trend with weight percentage. For example, the static yield stress of the MRSTF dropped from 26 kPa at 0% w/w to about 12 kPa at 10% w/w. Further increasing the w/w percentage to 15%, the static yield stress of the MRSTF increased up to about 19 kPa. The reason for this phenomenon is the different influences of silica in different concentrations. The silica in the low concentration STF is a kind of interference. Because of the low concentration, the silica particles are not sufficient to form a stable structure, they can only interfere with the magnetic particle chain formation process and decrease the MR effect.

However, the MRSTF made with 15% STF has a similar MR effect to MRF. (The first and second are the testing results of the first time shear and second time shear just after the first time shear. The different results indicate whether the fluid has the thixotropy phenomenon or not.)

According to the steady shear results, when the weight ratio of silica is over 15%, an obvious shear-thickening phenomenon can be observed. Applying a magnetic field, the shear-thickening phenomenon disappeared, and the MRSTF only shows an MR effect. The reason for this may be that the iron particle chains in the magnetic field prevent the

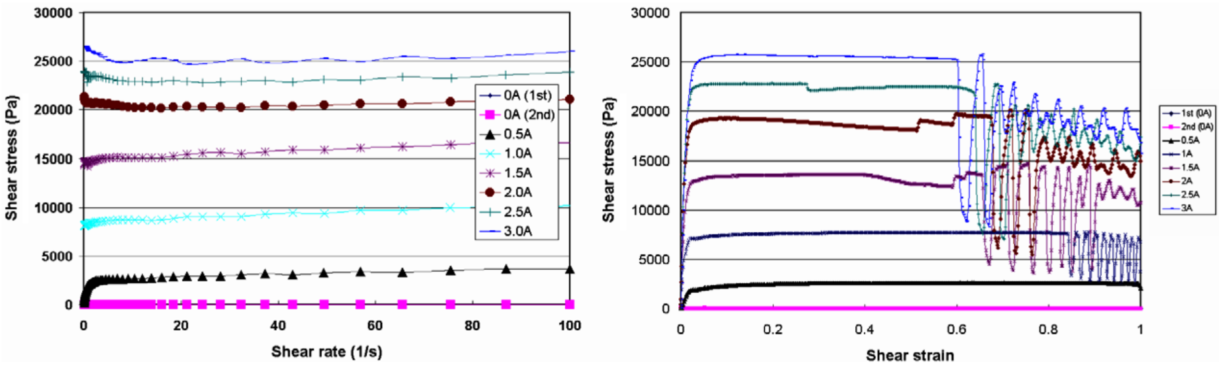

Figure 4. The steady shear and quasi-static shear results of the MRSTF sample based on 0% w/w STF.

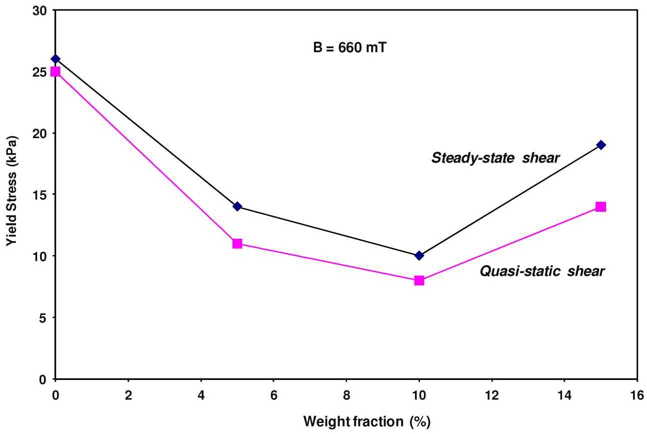

Figure 5. Yield stresses as a function of weight percentage.

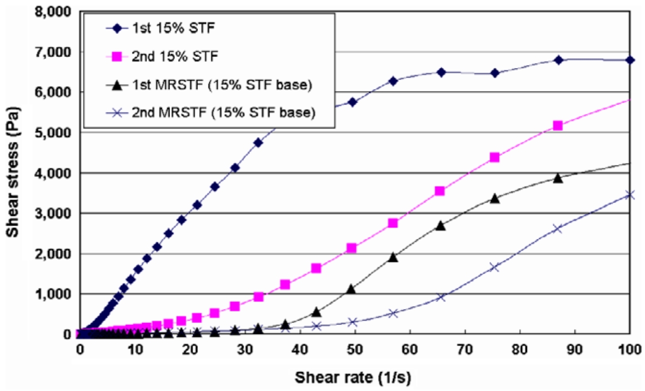

Figure 6. The steady shear results of the MRSTF sample based on 15% w/w STF and the STF sample.

hydrocluster of silica particles. The MRSTF based on low concentration STF have no shear-thickening phenomenon.

Figure 6 shows the steady shear results of the MRSTF sample based on 15% w/w STF and the STF sample. Comparing the shear experiments for the MRSTF and STF base, it can be found that the addition of micron size iron particles reduces the viscosity of the original STF to make it closer to the general characteristics of the MRF. The viscosity of the STF can be decreased obviously by mixing particles with different sizes. The shear-thickening phenomenon can even completely disappear in some samples, which has been reported in our previous work [22]. In this paper, the mixture of micron size iron particles and nano-size silica powder shows the same phenomenon.

According to the quasi-static shear results, the addition of silica can improve the stability of the iron particle chain structures of the MRF in a magnetic field. Even at a high shear strain, the shear stress can still remain stable. At high magnetic field strength and large strain, for the MRF based on pure polyethylene glycol, its internal chain structures are broken and reorganize continuously during the shearing, and the shear stress oscillation phenomenon can be observed.

For each sample, the first shear and the second shear results are displayed in the same figure for comparison. It can also be observed from figure 6 that the MRSTF has a static solidification phenomenon (thixotropy). After shearing, the viscosity of the material could be reduced to one order of magnitude. In the solidification state, the MRSTF shows a viscoelastic fluid behavior, and maintains a semi-solidified state. This phenomenon cannot be observed in the MRSTF based on a low concentration of STF, the difference between the two shear results cannot be distinguished. This behavior is an important reason for there being no sedimentation of the MRSTF based on high concentration STF. In most MRFs, the liquid media can only provide a drag force to prevent particle sedimentation. The drag force is dependent on the moving speed. It is not able to avoid the particles' movement absolutely. In a high concentration STF based MRF, when it is in the solidification state, the iron particles are fixed in their original position by the elastic force provided by the matrix. And the movement is restricted.

During the experiments, it can be observed obviously that in the static state, the MRSTF based on high concentration STF (silica weight ratio over 15%) has a solidification phenomenon with relatively high viscosity. After shearing, its viscosity will be decreased and it will be re-flowable again. Without shearing, its viscosity will increase again. i.e. it shows a significant thixotropy phenomenon.

2.4. Stability observation

Figure 7 shows the stability testing of these four samples. Without silica (sample A), the iron particles deposit on the bottom of the bottle very rapidly. In a couple of minutes, the particles for sample A had totally settled. For samples B and C

Figure 7. Particle settling testing with different weight fractions. A: 0%; B: 5%; C: 10%; and D: 15%.

with silica weight percentages of 5% and 10%, respectively, the particle sedimentation is still clear, though there is an obvious improvement compared with sample A. This can be seen from figure 7 where samples B and C were observed after 24 h. For sample D where the weight ratio of silica in the STF is 15%, no sedimentation was observed after a few weeks.

3. Theoretical analysis

3.1. The resistance of the matrix

When a ball moves in a Newton fluid with a low Reynolds number ($Re \leqslant 1$), the resistance of the matrix can be expressed by the Stokes equation:

$$F_D = 6\pi \eta a_0 \dot{x} \tag{1}$$

where η is the viscosity of the matrix, a_0 is the particle's radius and $\dot{x}$ is the particle's speed.

It is noted that the carrier fluids for fabricating MR fluids are not Newtonian fluids. For example, the MRSTF still has a low original shear stress even without applying a magnetic field, which is because of the thixotropic phenomenon. In this study, the MRSTF is the Bingham plastic fluid, whose constitutive equation is given by

$$\begin{aligned} \tilde{T} &= (\eta + \tau_0/\mathrm{II})\tilde{A}_1, \qquad [(\mathrm{tr}\tilde{T}^2)/2]^{1/2} > \tau_0 \\ \tilde{A}_1 &= 0, \qquad [(\mathrm{tr}\tilde{T}^2)/2]^{1/2} \leqslant \tau_0 \end{aligned} \tag{2}$$

where η is the plastic viscosity, τ_0 is the critical yield stress, $\tilde{A}_1$ is a first order Rivlin–Ericksen tensor, $\mathrm{II} = [(\mathrm{tr}\tilde{T}^2)/2]^{1/2}$ is the second invariant of $\tilde{A}_1$.

The general Reynolds number can be expressed as

$$Re_B = \frac{2\rho a_0 \dot{x}}{\eta(1+\varepsilon)} \tag{3}$$

where $\varepsilon = \frac{2\tau_0 a_0}{\eta \dot{x}}$. The experimental drag coefficient expression is

$$C_D = 19/Re_B \qquad Re_B < 10. \tag{4}$$

The resistance of the matrix can be expressed as

$$F_D = \tfrac{19}{4}\pi a_0[\eta\dot{x} + 2\tau_0 a_0]. \tag{5}$$

The particle's volume fraction also influences the resistance during the particle's movement. According to the Guth–Gold equation, the viscosity of the matrix and the particle mixture can be expressed as

$$\eta' = \eta(1 + 2.5\varphi + 14.1\varphi^2) \tag{6}$$

where η is the viscosity of the matrix and φ is the particle's volume fraction. This equation only considers the volume effect but neglects other interactions of particles.

3.2. Differential equation for the particle's movement

During the sedimentation, the forces applied to particles include gravity, buoyant force and thermal force (can be neglected). The particle motion equation is

$$f + F_D + m\ddot{y} = G \tag{7}$$

where f is the resultant of gravity and buoyancy, F_D is the matrix's resistance, and m is the particle's mass, G is the gravity. By substituting the expressions of force in this equation, the movement of the particle can be solved.

The sedimentation of particles is caused by gravity. It can be expressed as

$$\begin{aligned} &\tfrac{4}{3}\pi a_0^3 \rho \ddot{y} + \tfrac{19}{4}\pi a_0[\eta(1 + 2.5\phi + 14.1\phi^2)\dot{y} \\ &\quad + 2\tau_0 a_0] - \tfrac{4}{3}\pi a_0^3 g(\rho - \rho_0) = 0 \end{aligned} \tag{8}$$

where ρ and ρ_0 are the density of particles and media, respectively. y is the displacement of particles along the vertical direction. It can be solved as

$$\begin{aligned} y &= \frac{[\frac{4}{3}\pi a_0^3 g(\rho - \rho_0) - \frac{19}{2}\pi a_0^2 \tau_0]}{[\frac{19}{4}\pi a_0 \eta(1 + 2.5\phi + 14.1\phi^2)]^2} \\ &\quad \times \left\{ \tfrac{19}{4}\pi a_0 \eta(1 + 2.5\phi + 14.1\phi^2)t + \tfrac{4}{3}\pi a_0^3 \rho \right. \\ &\quad \left. \times \left[1 - \exp\left(-\frac{57\pi a_0 \eta(1 + 2.5\phi + 14.1\phi^2)}{16\pi a_0^3 \rho}\right)t\right]\right\}. \end{aligned} \tag{9}$$

According to equation (9), if the inequality $\frac{8}{57}a_0 g(\rho - \rho_0) \leqslant \tau_0$ is met, sedimentation will not occur. Therefore, if the original critical shear stress of a thixotropic MRSTF is over a critical value, sedimentation can be totally avoided.

Figure 8 shows the quasi-static shear result of a MRSTF based on 15% w/w STF. It can be seen from the experimental results that the solidified MRSTF at zero field is not a real Bingham fluid, because its shear stress will decrease sharply after an original shear. However, it still has an original critical shear stress value of about 45 Pa at very low first shear.

In our experiment, the diameter of iron particles is about 5 μm, the density of carbonyl iron particles is about 4×10^3 kg m^{-3}. If we substitute these parameters into equation (9), we find that the inequality is met. Thus in this case, the particles would not result in sedimentation. This is also demonstrated by the particle settling testing, as shown in figure 7 case D.

4. Conclusion

In this paper, a kind of novel MR fluid based on high concentration STF was fabricated. Its mechanical behavior and MR effect were tested. The important behaviors of MRSTF based on high concentration STF are as follows.

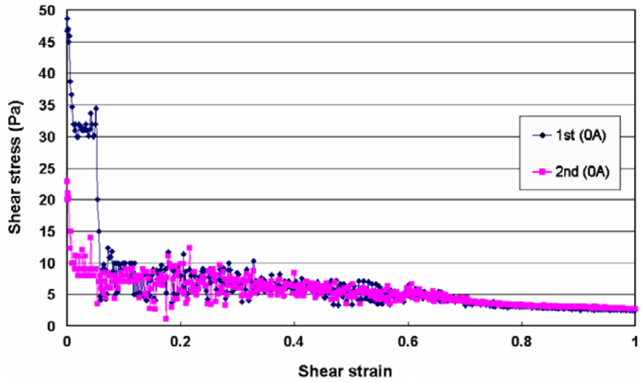

Figure 8. Quasi-static shear result of MRSTF based on 15% w/w STF.

(a) The shear-thickening phenomenon without magnetic field, and the MR effect in magnetic field.
(b) It has the thixotropy phenomenon (static solidification and re-flowable after shearing). Because there is a phenomenon of standing solidified, there is no sedimentation of ferromagnetic particles, and the resumption of flow after shearing can guarantee its ability after long-term storage, i.e. when the weight ratio of silica in the STF is about 15%, the MRSTF has an obvious thixotropic phenomenon and good MR effect.

Major differences between conventional MRF and the presented MRSTF include the following.

(a) After a long-term storage, the sedimentation of particles in MRF causes an overall decline in viscosity. It can be re-mixed after fully shearing, and then recover its original state. The MRSTF has no sedimentation, but its viscosity increases after a long-term storage. After shearing, it will be re-flowable and recover to its original state in a few seconds.
(b) For fail-safe requirements, the MRSTF is always in the state of high viscosity. Even in the absence of current input, it can also provide high viscosity in high shear rate because of its shear-thickening behavior in the zero field.

With these behaviors, MRSTF has comparative advantages to conventional MRF in some special applications, such as MR dampers for anti-seismic structures. It can guarantee that no sedimentation occurs after a long-term storage. And a high viscosity state for fail-safe can be achieved. This MR fluid has no sedimentation, high MR effect and low zero-field viscosity after shear. However, the zero-field viscosity of this sample is a little bit higher when compared with conventional MR fluids. It is good for dampers, which need their original damping force without control, but not suitable for clutches, which need very low friction while uncoupling.

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