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Silica-coated carbonyl iron microsphere based magnetorheological fluid and its damping force characteristics

Y D Liu\textsuperscript{1,2}, J Lee\textsuperscript{3}, S B Choi\textsuperscript{3} and H J Choi\textsuperscript{1}

\textsuperscript{1} Department of Polymer Science and Engineering, Inha University, Incheon 402-751, Korea
\textsuperscript{2} State Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, People’s Republic of China
\textsuperscript{3} Department of Mechanical Engineering, Inha University, Incheon 402-751, Korea

E-mail: hjchoi@inha.ac.kr

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Abstract
Silica-coated soft magnetic carbonyl iron (CI) particles with a reduced density and enhanced anti-corrosion properties compared to pristine CI were synthesized and applied as magneto-responsive particles in a magnetorheological (MR) fluid in this study. The MR fluids containing both pristine CI and silica-coated CI particles were injected into a custom-designed MR damper, and their damping characteristics, such as damping force as a function of time, displacement and velocity, were investigated, since vibration attenuation using mechanical damper systems is one of the main applications of MR fluids. Under the same magnetic field strength applied, the damping characteristics of the two MR fluids were observed to be directly related to their yield stresses.

(Some figures may appear in colour only in the online journal)

1. Introduction
A magnetorheological (MR) fluid is a smart concentrated suspension of micron-sized magnetic particles dispersed in a non-magnetic liquid [1–3]. The freely moving magnetic particles in the MR fluid can be polarized magnetically and connected to adjacent particles to form particulate chains or columns parallel to the magnetic field applied. The transition in the structure of the dispersed magnetic particles alters the behavior of the MR fluid from a Newtonian fluid to a Bingham fluid with extremely different rheological properties, such as shear stress, shear viscosity, yield stress and dynamic modulus [1]. MR fluids have been extensively studied for both academia and industrial engineering applications owing to their interesting controllable mechanical and rheological properties. MR fluid-filled devices, such as shock absorbers, dampers, clutches and polishing devices [4–7], have been designed, and some have already been commercialized [8].

Carbonyl iron (CI), a soft magnetic microparticle with a high purity of Fe (>97%), is the most widely adopted magnetic source in MR fluids, on account of its high saturation magnetization and low magnetic hysteresis. On the other hand, compared to carrier liquids (e.g., insulating oils, ionic liquids), CI particles are so heavy that sedimentation occurs easily in an MR fluid. One strategy [2, 9] to solve this problem is to coat the CI particle with polymers or inorganics [10–15], which not only reduces the density of the pristine CI particles but improves the anti-corrosion properties. In a previous study, silica-coated CI (CI@SiO\textsubscript{2}) particles were synthesized using a facile Stöber (sol–gel) method [14, 16]. The CI@SiO\textsubscript{2} particles exhibited high acid corrosion resistance and heat-induced oxidation resistance, but showed a reduced saturation magnetization [17].

Concurrently, as one of the commercially available MR devices, MR dampers have been developed for vibration control in automobiles, washing machines, bridges and buildings [8]. The modeling and validation of MR dampers are also focused on improving the application of MR dampers.
Figure 1. Schematic diagram for synthesizing the CI@SiO₂ particles.

Figure 2. Configuration of the proposed MR damper: (a) components, (b) design parameters.

2. Experimental details

2.1. Materials

The raw carbonyl iron (CI) powder used in this study was CM grade (soft, Fe min.% = 99.5%) from BASF Corporation with a mean particle size of 4.5 μm and a particle density of 7.9 g cm⁻³. The process for fabricating the CI@SiO₂ core–shell particles was performed as described in our previous studies [14, 17]. The raw CI particles (10 g) were first treated with methacrylic acid (MAA, 3 g) in ethanol with sonication for 10 min, and then grafted by vinyltrimethoxysilane (V-TMOS, 5 g) in the same manner. Grafting by the V-TMOS involves catching the tetraethoxysilane (TEOS) molecules and forming a silica layer. The silica shell was then coated on the chemically grafted CI particles by the Störber (sol–gel) method using TEOS as a precursor and a 5 M ammonia solution as a catalyst. In the sol–gel process, the grafted CI particles were dispersed in an ethanol solution of TEOS (10 g) with vigorous stirring. A mixture of ammonia (5 M, 87 g) and ethanol (50 ml) was then added to the system, and stirred for 24 h. The synthesized particles were washed several times with ethanol and de-ionized water, then dried in an oven for 24 h. A schematic of the process for synthesizing the silica-coated CI particles is shown in figure 1.

2.2. Design and manufacture of the damper

A miniature, shear mode type MR damper, which is applicable to vibration control systems, was designed and manufactured. Figure 2 shows the configuration of the proposed shear mode type MR damper, consisting of a cylinder, piston, magnetic core located inside the cylinder, bearings, oil-seals and MR fluid. The MR fluid was filled between the outer piston and inside the magnetic core, then sealed with oil-seals.

Generally, MR dampers can be operated in three different fluid modes: flow mode, shear mode and squeeze mode [8, 22–25]. Shear mode occurs when one plate with a gap translates or rotates relative to another fixed wall. At this time, the MR fluid is sheared parallel to the plates and generates a yield stress. To simplify the analysis of the shear mode type damper, it was assumed that the MR fluid is incompressible, and the yield stress of the MR fluid is constant across the gap between the two plates. The flow of the MR fluid was assumed to be laminar and steady, and the centrifugal force was neglected. The MR damping force of the shear mode damper $F_{MR}$ is directly related to the shear force on the top plate, and can be calculated using the following equation [4]:

$$F_{MR} = \tau A_c$$  \hspace{1cm} (1)

where $\tau$ is the shear stress of the MR fluid and $A_c$ is the charged area of the MR fluid in the shear mode damper, which is given by $A_c = 2\pi rl_e$, where $r$ is the axial radius of piston, $l_e$ is the effective yielding length of the magnetic core, i.e. $l_e = 4l_p$, where $l_p$ is a thickness of magnetic pole. For simplicity, the Bingham plastic model was used to describe the flow behavior of the MR fluid. Bingham plastic flow is a
Table 1. Design parameters of the MR damper.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total length of the MR damper ($l_d$)</td>
<td>242 ± 30 mm</td>
</tr>
<tr>
<td>Diameter of cylinder ($d_c$)</td>
<td>42 mm</td>
</tr>
<tr>
<td>Diameter of magnetic core ($d_m$)</td>
<td>35 mm</td>
</tr>
<tr>
<td>Height of magnetic core ($h_m$)</td>
<td>58 mm</td>
</tr>
<tr>
<td>Radius of piston ($r$)</td>
<td>8 mm</td>
</tr>
<tr>
<td>Thickness of magnetic pole ($t_p$)</td>
<td>8 mm</td>
</tr>
<tr>
<td>Gap between piston and magnetic core ($d$)</td>
<td>1 mm</td>
</tr>
<tr>
<td>Number of turns of coil</td>
<td>255 turns</td>
</tr>
</tbody>
</table>

function of the yield stress, $\tau_y$. The shear stress of the MR fluid can be defined as

$$\tau = \tau_y(H) + \frac{\eta \dot{x}}{d} \quad (2)$$

where $\tau_y(H)$ is the yield stress of the MR fluid determined by the magnetic field strength $H$, $\eta$ is the fluid viscosity coefficient, $\dot{x}$ is the piston velocity of the damper and $d$ is the gap between the piston and magnetic core. The magnetic field strength $H$ can be determined by Kirchhoff’s law for the magnetic circuit design of a magnetic core, and calculated using the following equation [26, 27]:

$$H = \frac{N_c I}{2d} \quad (3)$$

where $N_c$ is the number of turns of the coil and $I$ is the input current. Therefore, the total damping force of the MR damper $F_d$ can be determined by

$$F_d = (F_{MR} + 2f_{seal})\text{sgn}(\dot{x}) \quad (4)$$

where $f_{seal}$ is the Coulomb friction force between the piston and oil-seals.

To manufacture the proposed MR damper, design parameters are determined as given in table 1. The maximum damping stroke of the MR damper was designed to be ±30 mm, and the required amount of MR fluid was approximately 5 ml. The smaller usage of MR fluids highlights the need to verify the characteristics of specially fabricated MR fluids in a range of applications, such as vibration control of washing machines. Based on the MR damper model and parameters, the shear mode type MR damper was manufactured, as shown in figure 3.

2.3. Characterization

The morphology of the CI particles before and after coating with silica was observed by scanning electron microscopy (SEM) (S-4300, Hitachi). Two MR fluid samples (20 vol.% in silicone oil, 100 cS) based on the pristine CI and silica-coated CI particles were prepared to examine the MR responses of the particles. Rheological responses of the MR fluids were measured using a commercial rheometer (MCR 300, Anton Paar, Germany) with a controlled magnetic field supported by a MR device (MRD 180). A parallel-plate geometry of the rheometer for the sample loading included non-magnetic metals to prevent introduction of radial magnetic force components on the shaft of the measuring system. The damping performance of the two MR fluids was examined using the designed shear mode damper mentioned above.

3. Results and discussion

Figure 4 shows the morphology of the pristine CI and the CI@SiO$_2$ particles. The pristine CI (figure 4(a)) and silica-coated CI (figure 4(b)) particles were almost spherical or had a hemispherical shape with a quite wide size distribution. On the other hand, the surface of the CI@SiO$_2$ particles appeared much smoother than that of the pristine CI particles, apart from some small spherical bubbles, which were considered to be silica particles. The measured density of the particles reduced from 7.9 g cm$^{-3}$ for pristine particles to 7.3 g cm$^{-3}$ for coated particles. Thus the coating thickness could be estimated to be about 80 nm according to the density difference of the coated magnetic particles before and after coating.

Figure 5(a) shows the flow curves (shear stress versus shear rate) of the MR fluids measured at various magnetic field strengths over a wide shear rate range of 0.01–200 s$^{-1}$ using a rotational rheometer. Without a magnetic field, none of the MR fluids behaved as Newtonian fluids, due to the dense volume percentage of magnetic particles, indicating that they showed non-Newtonian fluid characteristics. When the magnetic field was applied, the shear stresses of the two MR fluids increased suddenly to a high level, increasing with increased magnetic field strength due to enhanced magnetic dipole–dipole interactions between the particles. This means that the MR fluids behave as a Bingham fluid, where a yield stress ($\tau_y$) is needed to initiate flow in the fluid. No flow occurs if the applied shear stress is below the yield stress. The shear stress ($\tau$) at each magnetic field strength can be described using the following equation:

$$\dot{\gamma} = 0, \quad \tau < \tau_y(H_0);$$
$$\tau = \tau_y(H_0) + \eta \dot{\gamma}, \quad \tau \geq \tau_y. \quad (5)$$

The shear stress at each magnetic field strength is stable over the entire shear rate range, indicating a strong interaction.
between the particles. Meanwhile, the CI@SiO₂ based MR fluid exhibits lower shear stress at each magnetic field in accordance with the coated non-magnetic silica layer. In addition, the shear stress of this MR fluid increases with increasing shear rate in the higher shear rate regions. This is also due to the reduced inter-particle forces caused by the coated silica layer. The yield stress of MR fluids can be obtained by extrapolating the shear rate to the zero shear rate limit, where the shear stress is defined as the dynamic yield stress. The yield stresses for the CI@SiO₂ MR fluid were 210, 968 and 2130 Pa at magnetic field strengths of 63.75, 127.5 and 191.25 kA m⁻¹, respectively, which are much lower than those of the pristine CI MR fluid (351, 1840 and 3030 Pa at a magnetic field strength of 63.75, 127.5 and 191.25 kA m⁻¹, respectively), due to the non-magnetic nature of the silica layer.

The shear stress can also be described by a power law of the shear rate (Ostwald–de Waele equation): \( \tau = K(\dot{\gamma})^n \). All the MR fluids behave as non-Newtonian fluids in both the off and on states (\( H = 0 \) and \( H \neq 0 \)). For \( \dot{\gamma} > 10 \) s⁻¹, \( n \) could be fitted to 0.87 and 0.13 for the CI@SiO₂ MR fluid, respectively, when the magnetic field was off and on; both indicating shear thinning behavior as \( n < 1 \). For the CI based MR fluid, \( n \) was found to be 0.64 (\( H = 0 \)) and 0.13 (\( H \neq 0 \)). The value of \( n \) was the same for both MR fluids when \( H \neq 0 \).

The dependence of the shear viscosity on the shear rate was predicted as: \( \mu \equiv \tau/\dot{\gamma} \propto \dot{\gamma}^{n-1} \), with the value of \( n - 1 \) given by 0.13 (\( H = 0 \)) and −0.87 (\( H \neq 0 \)) for the CI@SiO₂ based MR fluid, and −0.36 (\( H = 0 \)) and −0.87 (\( H \neq 0 \)) for the CI based MR fluid. Surprisingly, the actual fitting results revealed the power of the shear rate to be the same as the predicted values. As shown in figure 5(b), all the shear viscosity curves measured in the presence of a magnetic field indicated a shear thinning behavior due to deformation and breakage of the magnetic-field-induced particulate chains in the shear flow. In addition, the CI@SiO₂ based MR fluid exhibited a lower shear viscosity than that of the pristine CI based MR fluid at the same magnetic field strength applied. On the other hand, the off-state shear viscosity of the CI@SiO₂ based MR fluid was higher than that of pristine CI based MR fluid, which might be caused by the increased packing distance of the coated particles due to the repulsive force between the particles caused by the negative surface of silica [28].

A reciprocating experimental apparatus was manufactured to examine the damping performance of both CI based and CI@SiO₂ based MR fluids, as shown in figure 6. The experimental apparatus is driven by a DC motor and has different frequencies depending on the input voltage from the computer data acquisition (DAQ) system. The MR damper was fixed on both ends of the experimental apparatus and
Figure 6. Experimental apparatus for measuring the damping force of the MR damper.

excited by a sinusoidal movement. In this experiment, the damping force, displacement and velocity of the MR damper were measured using a load cell and linear variable differential transformer (LVDT). The current input of the MR damper was generated from the DAQ system. This signal was transmitted to a current amplifier and applied to the MR damper.

Figure 7 presents the field dependent damping force characteristics of the CI based MR fluid damper measured at a ±15 mm stroke and a frequency of 2.7 Hz. As shown in figure 7(a), the damping force increased with increased current (magnetic field strength). Figures 7(b) and (c) show the damping force with respect to the displacement and velocity. The values of the maximum and minimum damping force under zero field conditions were 11.1 N and −10.2 N respectively. At a magnetic field strength of 191.25 kA m$^{-1}$ (1.5 A), the values of the maximum and minimum damping force were 72.2 N and −58.9 N, respectively.

Figure 8 shows the field dependent damping force characteristics of the CI@SiO$_2$ based MR fluid damper measured at a ±15 mm stroke and a frequency of 2.5 Hz. The behavior of the damping force of the CI@SiO$_2$ based MR fluid damper was similar to that of the CI based MR fluid damper. However, the rate of increase of the damping force was much lower than that of the CI based MR fluid damper. Figures 8(b) and (c) shows that the values of the maximum and minimum damping force under zero field conditions were 10.2 N and −5.7 N, respectively. The values of the maximum and minimum damping force at 191.25 kA m$^{-1}$ were 38.1 N and −33.6 N, respectively. A comparison of the results of the two experiments suggests that the synthetic process of the silica-coated CI particles reduces their magnetic properties. Interestingly, at each magnetic field strength, the ratio between the damping forces of the pristine CI based MR fluid and the CI@SiO$_2$ based MR fluid was similar to the ratio between the yield stresses of the two MR fluids, which are all in the range 1.5–2.0. In addition, when we undertook the experiment, we chose some (limited) exciting velocities to get the results shown in figures 7 and 8. This is why the hysteresis is not clear in the figures, even though the hysteresis is in general a nonlinear phenomenon of an MR damper. Note that the hysteresis can be clearly shown using a certain...
Figure 8. Damping force characteristics of CI@SiO$_2$ based MR fluid as a function of time (a), displacement (b), and velocity (c), respectively.

Figure 9. Time response characteristics for $H = 127.5$ kA m$^{-1}$.

The time response of the damping force demonstrated by the two MR fluids was compared (figure 9). Depending on the pulsing current signal, both MR fluids showed a delayed increase and decrease in the damping force. It took 0.025 s for the pristine CI based MR fluid to reach the highest damping force and return to the stable off-state. The delay time was 0.015 s for the CI@SiO$_2$ based MR fluid, even though the on-state damping force of this MR fluid was lower than that of the pristine CI fluid. This suggests that the coated particles respond much faster than the pristine particles, even though the viscosity of the MR fluid with the coated particles was higher (figure 5(b))—attributed to the improved wettability of the silica-coated CI particles to the silicone oil. The delayed response was not observed in the shear stress and shear viscosity curves because the interval time for each point was longer than 2.0 s.

4. Conclusion

Silica-coated CI particles were synthesized to reduce the density and improve the anti-corrosion properties of the magnetic particles in an MR fluid. The MR fluids based on both pristine CI and CI@SiO$_2$ particles were applied to a newly designed shear mode damper. The damping force of the MR fluid using the coated particles, which was measured as a function of time, displacement and velocity, respectively, was lower than that of the MR fluid using the pristine CI particles. At the same applied magnetic field strength, the damping characteristics of the two MR fluids were found to be directly related to their yield stresses. The response time of the coated particles was shorter than the pristine CI particles due to the improved affinity between the silica-coated particles and silicone oil.

Acknowledgment

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