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High-Performance Hybrid Magnetorheological Materials: Preparation and Mechanical Properties

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To improve mechanical performance of magnetorheological elastomers (MREs), two novel hybrid magnetorheological elastomers that were embedded with magnetorheological fluids (MRFs) and magnetorheological gels (MRGs) were fabricated. In this work, MRFs and MRGs were injected into different numbers of holes that were punched on MRE specimens regularly. The nonlinear mechanical properties of the as-prepared magnetorheological fluid-elastomers (MRFEs) and magnetorheological gel-elastomers (MRGEs) were investigated in the presence of external homogeneous magnetic fields. The modulus, loss factor, and hysteresis were evaluated with a modified dynamic mechanical analyzer (DMA) and vibrating sample magnetometer (VSM). Dependence of the rheological response on the volume fraction was also investigated. As the experimental results show, not only were the initial moduli of the two novel hybrid magnetorheological materials higher than those of both the MRFs and MRGs, but also their magnetorheological (MR) effects were better than those of MREs. The loss factors of the two new hybrids were different from those of traditional MREs. Moreover, their dynamic properties changed according to the different volume ratios of MRFs and MRGs injected into the MRE specimens. These results suggest that the two novel hybrid magnetorheological elastomers are improved systems with volume-fraction-dependent rheological responses and the mechanical properties of MREs can be improved by embedding them with MRFs and MRGs.

1. Introduction

Magnetorheological (MR) materials are a class of smart materials whose mechanical and magnetic properties can be varied by applying an external magnetic field.[1] Since the MR effect was discovered by Rabinow in 1948,[2,3] MR materials have become a large family including MR fluids, MR foams, MR gels, and MR elastomers (MREs).[4] The most common MR materials are MR fluids (MRFs), whose yield stress and apparent viscosity can be changed from those characteristic of a Newtonian liquid to those characteristic of a non-Newtonian liquid when a magnetic field is applied.[5,6] MR elastomers (MREs) are a new branch of MR materials whose mechanical properties, including modulus and damping capability, can be controlled by an external magnetic field. Such materials are composed of magnetizable particles (iron particles) and a soft rubberlike matrix[7−11] and they can be divided into isotropic MREs and anisotropic (chainlike structure) MREs based on different curing conditions. The MR effects of MREs with chainlike structures is larger than that of isotropic MREs under a magnetic field of the same strength.[12−14] In recent years, the electrical resistivity and piezoresistivity of magnetorheological elastomers were researched by Kchit and Bossis.[15,16] Isotropic magnetic gels, which are gels containing randomly dispersed magnetic particles, are able to react to magnetic field gradients, as introduced by Zrínyi et al.'s pioneering work[17,18] in the 1990s. In 2003, ferrofluid-based anisotropic magnetic gels with frozen-in magnetic order that can respond to homogeneous fields were reported.[19] Magnetorheological gels (MRGs), which are composite fluids of magnetic particles suspended in polymer gels, are known as a new generation of MRFs and are used in vibration control and damping devices.[22] These fluids have the advantages of being able to control off-state viscosity and reduce the settling rate of the magnetic particles in the fluid.[23]

Because the mechanical properties of MREs can be controlled by an external magnetic field, MREs are promising for many applications, including adaptive-tuned vibration absorbers,[20] stiffness-tunable mounts and suspensions,[21] and variable-impedance surfaces.[22−24] It is important to improve the mechanical performance of magnetorheological elastomers, because MRE devices often work in dynamic modes. In this new emerging field, only a few studies have been reported during the past few years on improving the performance of MR elastomers by injecting MRFs into traditional MREs.[25] Despite its great potential as a strongly actuating system, no attempts have been made to obtain MREs embedded with MRGs.

In this work, two novel hybrid magnetorheological elastomers that were embed with MRFs and MRGs were fabricated. The mechanical properties of MREs containing MRFs or MRGs and the relationship between the rheological response and the different volume fractions of the matrix were intensively investigated. It is shown that a relative MR effect of 399% can be obtained at a small strain (0.5%), depending on the MRG volume fraction. The maximum storage modulus of the samples increases with increasing volume fraction of MRFs and MRGs. Conversely, the initial modulus ($H = 0$) decreases with volume fraction. A marked increase in the loss factor under magnetic field at a high frequency is reported. Hysteresis of both MRFE and MRGE samples shows characteristics that are different from those of traditional MREs.

2. Experimental Section

2.1. Sample Preparation. To prepare MRGs, poly(propylene glycol) (PPG-1000, $M_n = 1000$; PPG-2000, $M_n = 2000$) (The Third Petrochemical Factory, Tianjin Petrochemical Inc.) was distilled at 110 °C under a vacuum for 1 h prior to use. Toluene diisocyanate (TDI-80; Bayer Co., Ltd.), 1,4-butanediol (BDO; Sinopharm Chemical Reagent Co., Ltd.), tin(II) octoate (Sinopharm Chemical Reagent Co., Ltd.), and acetone (Sinopharm

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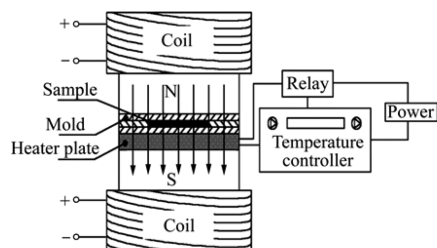
Figure 1. Principle diagram of the PS device.

Chemical Reagent Co., Ltd.) were used as received. 1-Methyl-2-pyrrolidone (NMP; Sinopharm Chemical Reagent Co., Ltd.) was used as an additive. The magnetic particles were carbonyl iron purchased from BASF (model CN) with the following size distribution: $d_{10} = 3.5 \ \mu$m, $d_{50} = 6 \ \mu$m, $d_{90} = 21 \ \mu$m.

Poly(propylene glycol) and toluene diisocyanate were mixed in a 250 mL three-necked round-bottom flask fitted with a mechanical stirrer, a thermometer, and a condenser. The temperature of the reaction system was increased to 80 °C and kept at this temperature for about 2 h under agitation. 1,4-Butanediol, used as a chain extender, was then added to the reactor, and chain extension was performed at 70 °C for the next 2 h. During the reaction, a suitable amount of acetone was added to reduce the system viscosity. Then, tin(II) octoate (0.015 g) was added dropwise into the flask, and the reaction temperature was kept at 70 °C for about 1 h under agitation. 1-Methyl-2-pyrrolidone was added as an additive under agitation. Then, the iron particles were added to polyurethane under stirring. In this study, the weight fraction of particle in the MRGs was 60 wt %. Then, the sample was placed in an oven at 50 °C for 72 h to vaporize the acetone.

In addition, iron particles that were the same for the MRGs and MREs were dispersed in silicone oil, separately, to prepare MRFs; the weight fraction of particle in the MRFs was also 60 wt %.

The MRE components include natural rubber (NR); carbonyl iron particles (average radius of 7 μm); and other additives containing carbon black, ZnO, stearin, MDA (4,4′-methylene-dianiline), sulfur, cz (N-cyclohexyl-2-benzothiazolesulphena-mide), plasticizer, and carbon black.[26] In samples, the mass fraction of the carbonyl iron particles was 60%. The fabrication procedure of MREs was as follows: The iron particles and other additives were first mixed into the rubber in a double-roll mill. The resulting material was then compression-molded into a mold in the self-developed magnet-heat coupled device, composed of a magnetic field generator, a mold, and a controllable heating system, as shown in Figure 1.[10]

The mixture was then exposed to a magnetic field and tightly fixed with a heating plate. The magnetic field generator was capable of applying an external magnetic flux density of 0−1 T over the samples. The heating plate was conterminal with a temperature controller whose temperature could be set in the range from 50 to 200 °C. In our experiments, the mixture was exposed to a magnetic field of 800 mT at 160 °C for 30 min, after which the sample was cured at 160 °C for 20 min under a pressure of 1.5 atm. MRE specimens were prepared with a thickness of 5 mm. Then, beamlike samples [10 mm (L) × 10 mm (W) × 5 mm (T)] were cut from the MRE specimens. Different numbers of holes were punched regularly in the MRE specimens, with the holes positioned parallel to the direction of the chainlike structure in the MREs by using the punch with a suitable drill. Then, MRFs and MRGs were separately injected into holes that were 1.5 mm in diameter using a syringe. Finally, both sides of the holes were sealed with a film that was 0.1
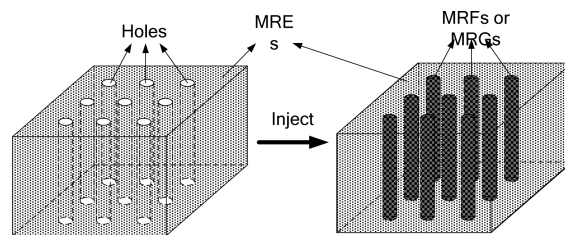

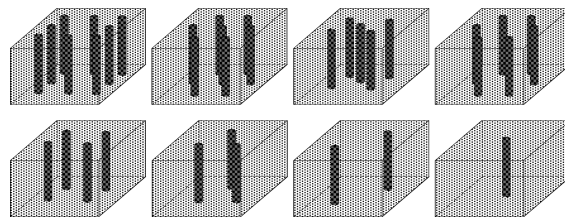**Figure 2.** Schematic diagram of sample with nine holes.

Figure 3. Schematic diagram of sample geometries with different volume ratios of MRFs and MRGs.
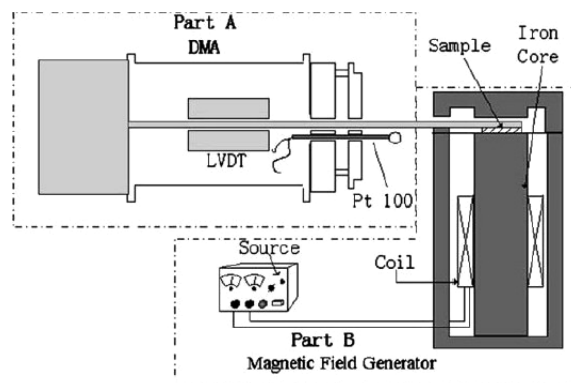

Figure 4. Sketch and principle diagram of the modified DMA.

mm thick. Then, the MRFE and MRGE samples were prepared for testing, as shown in Figures 2 and 3.

2.2. Testing. Samples were prepared according to the procedure described in the preceding section. Then, the mechanical properties of the samples, such as modulus and damping factor, were evaluated by using our modified dynamic mechanic analyzer (DMA).[9,10] The principle diagram of the modified DMA is shown in Figure 4. This system is composed of two parts: labeled part A and part B. Part A is a common DMA (Triton Technology Ltd., model Tritec 2000B), and part B is a self-developed electromagnet that can generate a variable magnetic flux density up to 1 T. The specimen was set between the driving draft of part A and the magnetic core of part B. When the draft moved, the specimen was deformed in a shear mode. This system applied a fixed oscillatory strain to the specimen and measured the amplitude and phase of the output force, from which the shear storage modulus G' and loss modulus G'' were calculated. Hysteresis behaviors of MRFE and MRGE samples were evaluated by using a HyMAC hysteresis meter measurement of the ac magnetic properties (Metis Instruments & Equipment NV, Leuven, Belgium).

3. Results and Discussion

3.1. Modulus and MR Effect. The modulus and MR effect are important mechanical properties for MR materials, so they were evaluated in this work with a modified dynamic mechanical analyzer (DMA). The storage modulus G' and loss modulus G'' for the MRFE and MRGE samples with different numbers of
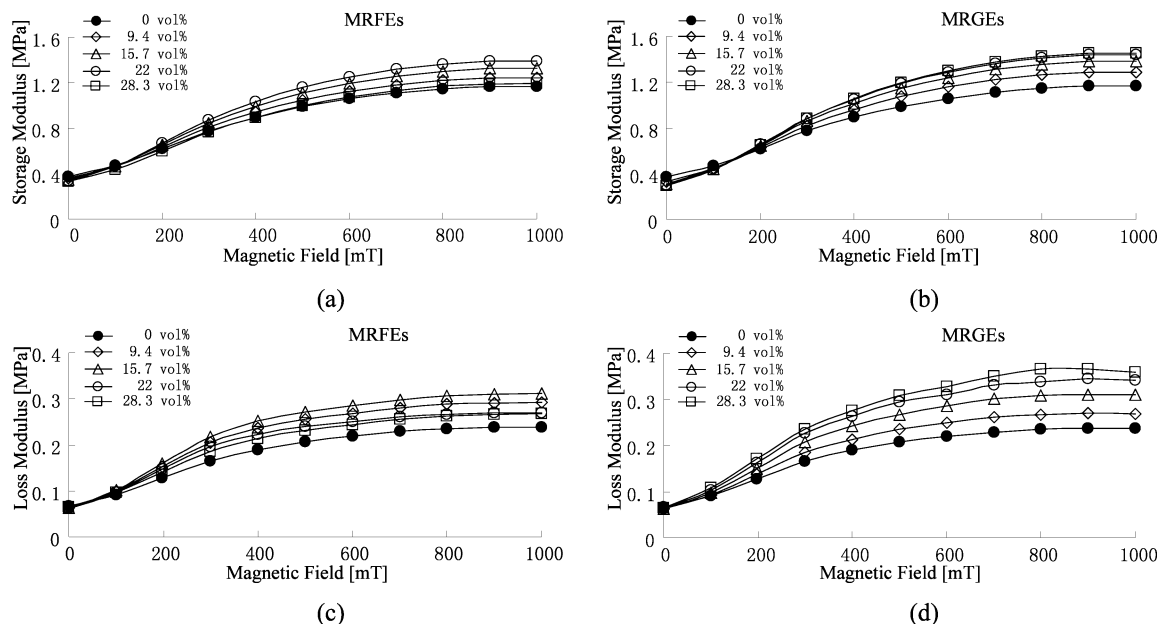
Figure 5. Dependence of storage modulus G' and loss modulus G'' on applied magnetic field for different MRF and MRG volume fractions.
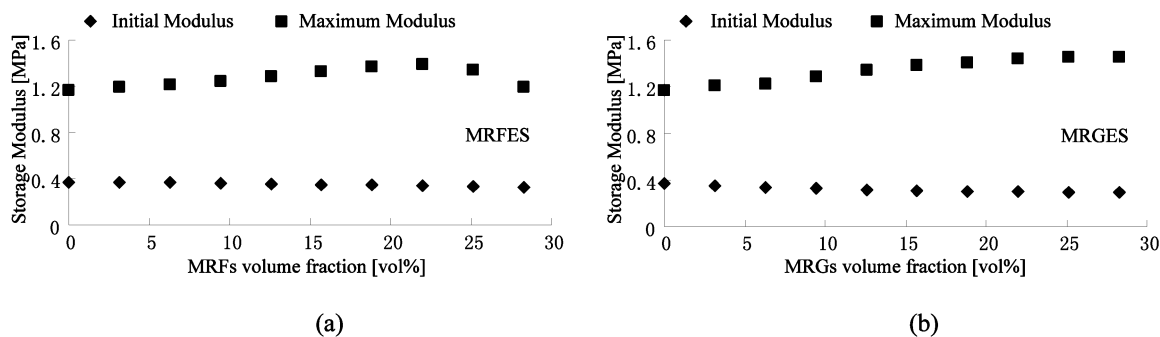

Figure 6. Increment of initial G' and maximum G' for different (a) MRF and (b) MRG volume fractions.

holes (different volume ratios of MRFs and MRGs) are shown in Figure 5. The volume fractions of MRFs and MRGs were varied from 0 to 28.3 vol %. As can be seen from these experimental data, the field-induced moduli G' and G'' of each sample exhibited an increasing trend with increasing magnetic flux density. This is because, with the increasing of the magnetic field intensity, the average particle−particle interaction increased. This will result in an increase in mechanical stiffness due to hardening of the filler strings with respect to the polymer matrix. However, the modulus of the samples that were injected with MRFs first increased with increasing MRF volume fraction up to 22 vol % and then modulus decreased with increasing MRF volume fraction as shown in Figure 5a. For example, the magneto-induced modulus of MRFEs, $G'_{H=1000\,mT}$, was 1.16 MPa when the MRF content was 0 vol %, compared to 1.39 MPa when the MRF content was 22 vol % and 1.19 MPa when the MRFs content was 28.3 vol %. It can easily be seen that the magneto-induced storage modulus reached a maximum of 1.39 MPa when the MRF content was 22 vol %. However, different from the MRFEs, moduli of the MRGE samples always increased with increasing MRG volume fraction and magnetic flux density, as shown in Figure 5b. For example, the magneto-induced storage modulus of MRGEs, $G'_{H=1000\,mT}$, was 1.16 MPa when the content was 0 vol % compared to 1.45 MPa when the MRGs content was 28.3 vol %. It can be seen in Figure 5c,d that the loss moduli of both MRFE and MRGE samples increased with increasing volume fraction and magnetic flux density. In addition, the increment for the MRGEs is greater

than that for the MRFEs, which is because the loss modulus of the fluid is smaller than that of the gel and elastomer.

To clearly demonstrate the relationships between the modulus (for the MRFEs or MRGEs) and the volume fraction (of the MRFs or MRGs), the initial modulus G_0 (for a magnetic flux density of 0); the maximum modulus G_{max}, which is the storage modulus when the particles are at magnetic saturation; and the absolute modulus difference, $\Delta G = G - G_0$ were studied and are displayed in Figure 6. The volume fractions of MRFs and MRGs were varied from 0 to 28.3 vol %. As shown in Figure 6a,b, the initial modulus G_0 of both MRFE and MRGE samples slowly decreased with increasing MRF and MRG volume fractions. For example, the initial moduli G_0 of the MRFEs and MRGEs were both 0.37 MPa when the MRF and MRG contents were 0 vol % compared to 0.33 and 0.29 MPa, respectively, at MRF and MRG contents of 28.3 vol %. Moreover, the maximum modulus G_{max} of the MRFEs first increased up to an MRF volume fraction of 22 vol %, and then the trend of G_{max} decreased with continually increasing MRF volume fraction. However, G_{max} of the MRGEs increased with increasing MRG volume fraction. As can be seen in Figure 6, the absolute modulus difference (ΔG) of the MRGEs was higher than that of the MRFEs, which is because the mechanical stiffness of the polymer gel matrix is higher than that of the liquid oil matrix. In addition, in our previous work, we found that initial modulus of pure MRGs is higher than that of MRFs and that the increment in the absolute modulus of pure MRGs is higher than that of pure MRFs for the same increase of the magnetic field
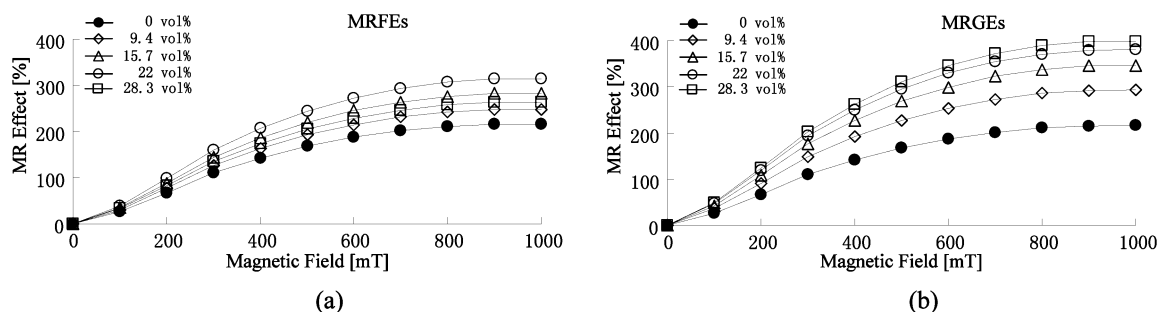
Figure 7. Dependence of the MR effect on applied magnetic field for different (a) MRF and (b) MRG volume fractions.
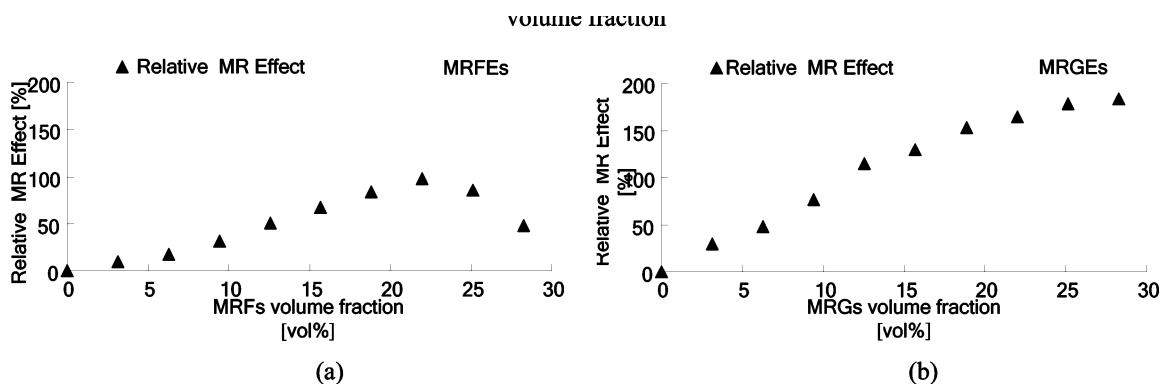

Figure 8. Increment of relative variation of MR effect for different (a) MRF and (b) MRG volume fractions.
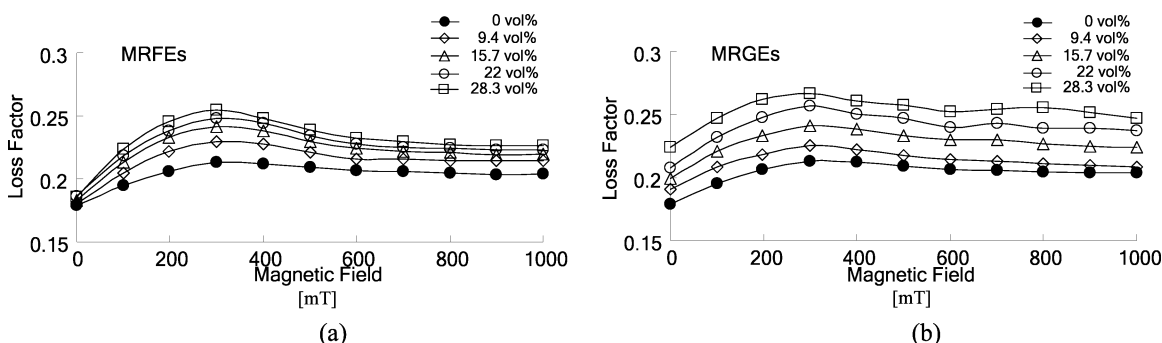

Figure 9. Dependence of loss factor on applied magnetic field for different (a) MRF and (b) MRG volume fractions at 1 Hz.

intensity. Moreover, the field-induced modulus is a key to the modulus increment. Therefore, the modulus of the MRGEs increased more than that of the MRFEs in the same magnetic field. The maximum absolute modulus differences (ΔG) of the MRFEs and MRGEs were 0.23 and 0.29 MPa, respectively, and the maximum absolute initial moduli were 42 and 77 kPa, respectively. From the above analysis, it is very clear that the mechanical properties of MREs can be improved by injecting MRFs or MRGs into the matrix.

The absolute MR effect is a key parameter for evaluating MRE performance. The MR effect is commonly used to characterize the MR material and is defined as MR_effect = $\Delta G_{max}/G_0$, in which G_0 is the initial modulus and $\Delta G_{max} = G_{max} - G_0$. G_{max} is the storage modulus when the particle is at magnetic saturation (900 mT). In this study, the applied magnetic field ranged from 0 to 1000 mT. Figure 7 shows the relation between the MR effect and magnetic field for different MRF and MRG volume fractions. Obviously, with increasing magnetic flux density, the MR effects of all kinds of samples show marked changes, and the maximum MR effects of samples without MRFs or MRGs was 216% (1000 mT), as shown in Figure 7a,b. However, the trends of the difference in the absolute maximum MR effects (ΔMR_effect$_{max}$ = MR_effect$_{max}$ −

MR_effect$_{0 vol \%}$) with different MRF and MRG volume fractions were dissimilar. As shown in Figure 8a, the MR effects for the MRFE samples reached a maximum when the MRF volume fraction of the MRFE samples was 22 vol %, and the maximum MR effect was 314%. In the meantime, the MR effects of MRGE samples always increased with increasing MRF volume fraction and magnetic flux density (Figure 7b, and the maximum MR effect was 399%. As a result, it is obvious that MR effects can be markedly improved by injecting MRFs or MRGs into MREs, and MRGE samples show higher MR effects than MRFE samples at the same volume fraction.

3.2. Loss Factor. The loss factor (tan $\delta = G''/G'$) is an important factor of damping capability, so the loss factors of all samples were also evaluated by a modified dynamic mechanical analyzer (DMA). The loss factors of samples with different MRF and MRG volume fractions were also measured at a dynamic strain of 3%; driving frequencies of 1, 10, 20, and 30 Hz; and various magnetic fields from 0 to 1000 mT. It can be seen from Figure 9 that the loss factors of all samples increased markedly with an increment in MRF and MRG volume fraction at the low frequency of 1 Hz. The loss factor increased with volume fraction of MRFs and MRGs. The loss
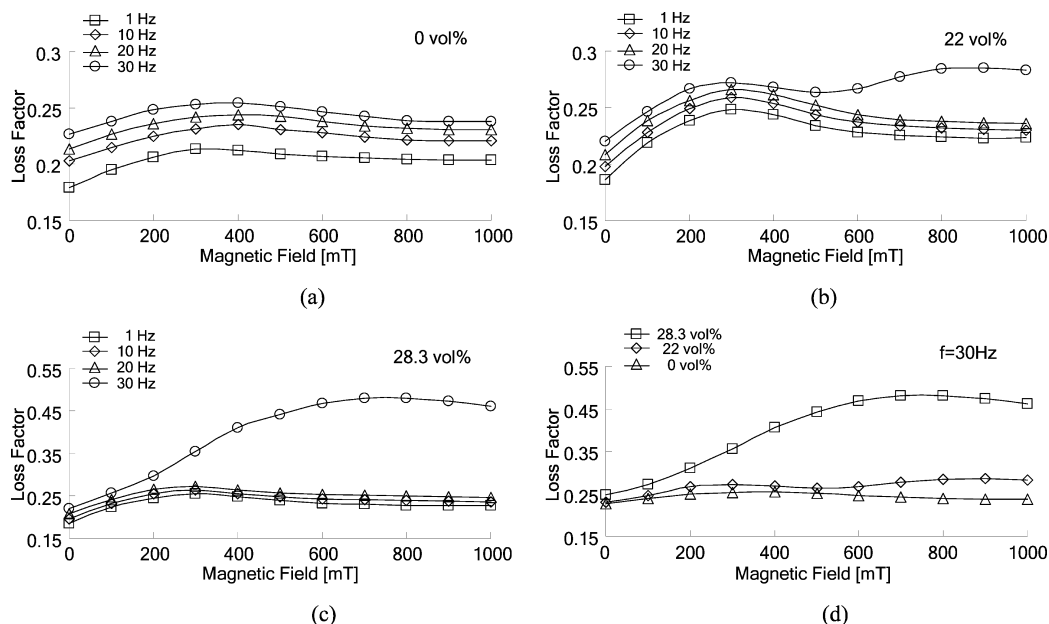
Figure 10. Dependence of damping factor on applied magnetic field for different MRF volume fractions at different frequencies.

factor of all samples exhibits a maximum at an intermediate value (300 mT) of the field shown in Figure 9. The loss factor first increased with the magnetic field, whereas it saturated at larger field values. The clear maximum point of loss tangent with increasing magnetic field in MREs was recently reported by Chen and Böse.[26,27] It is more natural to be understood on the basis of the competition of loss modulus and storage modulus. Obviously, MRFEs (or MRGEs) with different loss factors can be carefully controlled by varying the volume fraction of the injected MRFs and MRGs.

The results show that the loss factors of the MRGE samples increased more obviously than those of the MRFE samples. Moreover, it also can be seen that the off-state loss factor ($B = 0$) of the MRGE samples was higher than that of the MRFEs with the same volume fraction. There are mainly two kinds of damping sources for MREs. One is from the matrix, and the other is from the friction between iron particles and matrix.[26] The friction between iron particles and polymer gels is stronger than that between particles and liquid matrix, which has lower viscosity than polymer gels, and the energy loss of particle movement in the polymer matrix is also lower than that in the liquid matrix, so the loss factors of MRGEs are higher than those of MRFEs when they are under the same applied magnetic fields and volume fractions of MRFs and MFGs. Moreover, the loss factor of the liquid matrix is also lower than that of gels. Therefore, the loss factors of MRGEs increased more markedly with the increment of the MRG volume fraction than those of the MRFE samples, as shown in Figure 9.

The frequency-induced loss factor with different volume factors is shown in Figure 10. It can be seen that the loss factor measured at 1 Hz is lower than those measured at other frequencies. In the figure, there is an interesting phenomenon: the loss factor of MRFE samples with high MRF volume fractions increased significantly with increasing applied magnetic flux density at a high frequency (30 Hz). For example, the maximum loss factor of MRFE samples, at $H = 800$ mT, was 4.8 when the MRF content was 28.3 vol % compared to 2.85 at an MRF content of 22 vol % and 2.37 at 0 vol %. The inherent resonance frequency of liquid is lower than that of solid and gels, so MRFEs contains more MRFs has a lower inherent resonance frequency than other samples based on MRGs. Then,
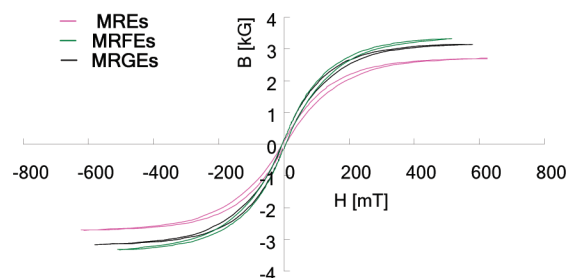

Figure 11. Hysteresis loop of different samples at applied magnetic field (MREs, MRFEs, and MRGEs).

resonance of MRFEs samples occurs earlier than MRGEs samples when excitation frequency is increased. Moreover, friction between iron particles and the matrix increases strongly when resonance occurs with an increment of frequency, and more mechanical energy turns to heat energy. In this case, the loss factors of MREF samples with high MRF volume fractions increased significantly when the testing excitation frequency increased, approaching the resonance frequency of the samples, as shown in Figure 10.

3.3. Hysteresis. The hysteresis properties of the samples were also evaluated by using the HyMAC hysteresis meter measurement of the ac magnetic properties in this work. As shown in Figure 11, the coercive fields of the MRFEs were smaller than those of the MREs and MRGEs with same applied magnetic field. However, the magnetic induction intensities of the MRFEs were greater than those of the MREs and MRGEs with the same applied magnetic field. For example, the maximum magnetic induction intensity of the MRFEs was 3.2 kG when the magnetization of the samples approached a saturation value. It is clear that the maximum magnetic induction intensity of the MRGEs and MREs reached maximum values of 3.0 and 2.5 kG, respectively, when the magnetization of the samples approached a saturation value. The results show that MRE samples with injected MRFs have higher magnetization than both MRGEs and traditional MRE samples. This is because the iron particles can move more easily in the liquid matrix of the MRFs under the applied magnetic field than in the MREs and MRGEs, so the chainlike structure can be formed more easily
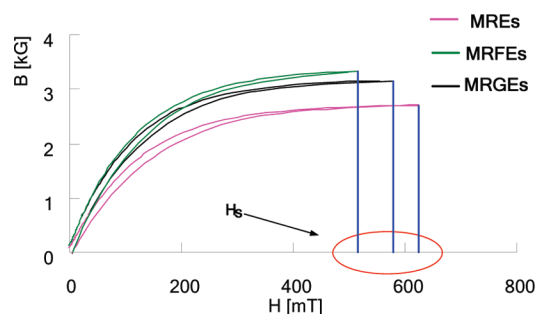
Figure 12. Magnetic saturations of different samples (MREs, MRFEs, and MRGEs).

and the magnetic induction intensity in the samples can reach a higher value.

From Figure 12, it is obvious that the magnetic saturation of MRFEs is the minimum one. For example, the MRFEs can be saturated when the applied magnetic field is 520 mT. However, for MRGEs and MREs, the applied magnetic fields for saturation were found to be 580 and 620 mT, respectively. The disposability of particles is better when it is based on a liquid matrix with lower viscosity, so fewer agglomerates form in samples based on MRFs and iron particles can be magnetized evenly, which makes MRFEs samples to achieve magnetic saturation more easily. Therefore, the saturation magnetization can be achieved with a low magnetic field, as also demonstrated by the experimental results.

4. Conclusions

Magnetorheological materials with improved mechanical properties can be successfully achieved by injecting MRFs or MRGs into different numbers of holes which punched in the MRE specimens regularly. The mechanical properties, such as storage modulus G', loss modulus G'', and MR effect, for all MRFE and MRGE samples exhibit an increasing trend with increasing magnetic flux density. The magneto-induced modulus and MR effect of MRG samples always increased with increasing MRF volume fraction, whereas the modulus and MR effect of samples that were injected with MRFs first increased with increasing MRF volume fraction up to 22 vol %, and then the modulus decreased with continuing increase in the MRF volume fraction. All of the mechanical properties, especially the MR effect, exhibited significant improvement compared with those of traditional MREs. Moreover, the loss factors of all MRFE and MRGE samples were markedly increased with increasing driving frequency and the increment of MRF and MRG volume fractions. A dramatic increase, namely, the damping factor increased significantly with increasing magnetic field intensity at a high driving frequency, was obtained when the MRF volume fraction was 28.3%. As shown in the experimental results, the MRFE samples achieved magnetic saturation more easily than traditional MRE and MRGE samples because of the higher magnetic flux density inside than in traditional MREs. In view of the above results, we can conclude that magnetorheological gels are an intermediate system between magnetorheological elastomers and magnetorheological fluids, both with directional-dependent responses and partial rearrangement of the particle network.

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