Smart Mater. Struct. 19 (2010) 055015 (8pp)

# Effect of maleic anhydride on the damping property of magnetorheological elastomers

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Received 5 December 2009, in final form 26 February 2010 Published 31 March 2010 Online at stacks.iop.org/SMS/19/055015

# Abstract

In this study, maleic anhydride (MA) was selected as the compatibilizer to modify the interfaces of magnetorheological elastomers (MREs) for improving the damping property. Several samples of MREs with different contents of MA were prepared. The content of bound-rubber was measured by the extraction method. The microstructures were observed by using an environmental scanning electron microscope (SEM). The dynamic performances of these samples, including shear storage modulus, loss factor and MR effect were measured with a modified dynamic mechanical analyzer (DMA). The tensile strength was tested by using an electronic tensile machine. The experimental results indicate that both the content of bound-rubber and the compatibility between the magnetic particles and rubber matrix were enhanced with the increase of MA. The enhancement of the bond between the two phases resulted in different mechanical properties: the increase of shear storage modulus; the reduction of the loss factor; the stability improvement of the loss factor; the enhancement of the tensile strength; and the reduction of the MR effect.

# 1. Introduction

Magnetorheological (MR) materials, including MR fluids, MR foams and MR elastomers, belong to a class of smart materials [1–3]. MR elastomers (MREs) mainly comprise of microsized soft magnetic particles and elastomers or rubberlike materials. The rheological or mechanical properties can be changed continuously, rapidly and reversibly by an applied magnetic field [4–7]. Recently, MREs have attracted considerable attention and obtained wider application prospects, e.g. replacing traditional devices in vehicle shock absorbers, noise reduction and other areas, such as suspension systems, engine mounts, bushings, absorber, etc [1, 8–12].

A number of studies have used MREs for designing vibration absorbers. Ginder *et al* [8, 9] constructed a tunable vibration absorber with MREs. The shear modulus

of the elastomer with applied magnetic field is effective to frequencies well above 1 kHz. Deng et al [10] used MREs to develop a tunable vibration absorber. The tuning frequency has a range of 55-82 Hz. Albanese et al [11] presented a stateswitched absorber using MREs. The natural frequency could be tuned from 45 to 183 Hz. Lerner et al [12] designed three vibration absorber configurations with MREs. The increases in natural frequency range are 183%, 473% and 507% in shear, longitudinal and squeeze modes, respectively. It is noted that the absorber effectiveness of these MRE vibration absorbers depends greatly on the damping property (loss factor) of MREs. The tunable vibration absorbers use MREs as variable stiffness springs, in which the MREs can transfer the vibration from a primary system to the vibration absorber system, and consequently reduce the vibration of the primary system. Low damping leads to a high vibration reduction for better vibration transferring capability, while high damping results

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in poor vibration suppression [13, 14]. The investigation of Sun et al [13] indicated that the vibration reduction effect increased obviously when the damping ratio reduced from 0.05 to 0.005. Hoang et al [14] also found this increased tendency of vibration reduction effect as the damping ratio reduced from 0.35 to 0.05. Thus it is important to study and reduce the damping property of MREs. Currently, very few reports can be found that study the damping property of MREs. In our previous work, the influencing factors on the MRE damping property, including the matrix, content of particles, dynamic strain, driving frequency and so on, were experimentally investigated [15, 16]. However, very little work has focused on the investigation of how to reduce the damping property of MREs. In order to make MRE vibration absorbers have a high absorber effectiveness in applications, there are still many problems that need to be solved to reduce the damping property of these novel smart composites. This is the major motivation of this study.

To obtain MREs with a low damping property, it is appropriate to enhance the compatibility between the magnetic particles and the rubber matrix to reduce the interfacial friction [15]. To this end, maleic anhydride (MA) was selected as the compatibilizer to modify the interfaces of the MREs and consequently decrease the damping property. This is because MA modified polymer can enhance the compatibility in polymer composites [17, 18].

This study includes the following major tasks. Firstly, samples with different contents of MA were prepared. Next, the bound-rubber and microstructures of these samples were observed, and the effect of MA on the damping property was analyzed. Mechanical properties of MREs, including the shear storage modulus, the tensile strength and MR effect, were also studied.

# 2. Experimental details

## 2.1. Preparation of MREs

MREs comprise mainly magnetic particles and elastic matrix. The cis-polybutadiene rubber (BR) was used as the matrix, manufactured by ShangHai Gao-Qiao Petrochemical Corporation, China. The magnetic particles were carbonyl iron particles, provided by BASF in Germany (type CM) with an average diameter of 7  $\mu$ m. MA was used as the compatibilizer, purchased from Enterprise Group Chemical Reagent Co. Ltd. In this experiment, four different contents of MA, 0 phr, 1 phr, 2 phr, 4 phr, were used to mix with the rubber for fabrication of the four MRE samples, which were numbered 1, 2, 3, and 4, respectively. The fabrication of the MREs consists of mixing, pre-forming configuration and curing. Firstly, using the method of mechanochemical techniques [19], 100 phr rubber and MA were mixed using a two-roll mill (Taihu Rubber Machinery Inc. China, Model XK-160) to obtain a modified BR. Then iron particles with a weight fraction of 60%, 100 phr plasticizer and 14 phr of other additives were put into the mixture and to form a gross rubber. The gross rubber was put into a mold for pre-forming under an external magnetic field of 1300 mT, generated by our developed magnet heat couple device [20], at 130 °C for 10 min. Finally, the samples were vulcanized and formed elastomers on a flat vulcanizer (Bolon Precision Testing Machines Co. China, Model BL-6170-B). The vulcanization process was operated at 160 °C for 15 min under a pressure of approximately 13 MPa.

### 2.2. Analysis of Fourier transform infrared spectra (FT-IR)

The infrared spectra measurements of the modified BR with different contents of MA were analyzed by using a FT-IR Spectrometer (Bruker, model EQUIVOX55). The spectra were collected from 4000 to 400 cm<sup>-1</sup>. The MA, which did not participate in modification in the rubber, was firstly removed. A certain amount of gross rubber without carbonyl iron particles was taken and immersed in toluene solvent to dissolve for 24 h, then filtered to remove the impurities. Next, a certain amount of acetone was added to the solution; sediments appeared, which were obtained by filtration, and dried in a vacuum drying oven at 50 °C. Finally, the sediments were dissolved in toluene and smeared on a KBr crystal plate prior to infrared spectra measurements.

## 2.3. Measurement of the bound-rubber of the MREs

During the rubber mixing process, a kind of network by absorption and entanglement between fillers and polymer was formed, which has been named bound-rubber and leads to insoluble rubber [21]. The bound-rubber exists both before and after the vulcanization, and consequently the same kind of bound-rubber is obtained. Zhang et al [22] found the bound-rubber phenomenon in MREs, which is the foundation of measuring the bound-rubber, and investigated the influence of it on MRE performance. In this study, the content of the bound-rubber of the MRE samples was measured by adopting the extraction method. The specific process was as follows: the gross rubber was placed at room temperature for two weeks, taking about 1 g and weighing the mass  $m_1$  accurately, which was packaged with a piece of quantitative filter paper and immersed in the toluene solvent of 100 ml for 72 h. The toluene solvent was changed every 24 h. Then the gross rubber was immersed in an acetone solvent of 100 ml for 24 h to remove the toluene. It was dried to constant mass in a vacuum drying oven at 50 °C and weighed, the mass being  $m_2$ . The weight fraction of bound-rubber in the gross rubber is  $W = (m_1 - m_3 - m_4)/m_1$ , in which  $m_1$  is the mass of the gross rubber before immersion,  $m_3$  is the mass of filler before immersion, and  $m_4$  is the mass of rubber dissolved in toluene, that is  $m_4 = m_1 - m_2$ .

#### 2.4. Observation of microstructure

The microstructures of samples were observed by using an environmental scanning electron microscope (SEM, Philips of Holland, model XT30 ESEM-MP). The accelerating voltage was 15 kV. These samples were cut into flakes and coated with a thin layer of gold on the surface before the SEM observation. Through the observation of the microstructure, information about the interfacial adhesion between rubber and magnetic particles was obtained.

Table 1. The weight fraction of bound-rubber of each sample.

Sample	Weight fraction of bound-rubber (%)
1	3.2
2	4.5
3	7.7
4	9.2

## 2.5. Measurement of dynamic properties

The dynamic mechanical performance of these samples was measured by using a modified dynamic mechanical analyzer (DMA) [20] (Triton Technology Ltd, UK, model Tritec 2000B). In this system, a self-made electromagnet was introduced to generate a variable magnetic field from 0 to 1000 mT.

MR materials can be tested under several modes. Farshad *et al* [2] investigated the MRE properties under the compression mode. Brigley *et al* [23] evaluated the MR isolator using multiple fluid modes, including shear, flow and squeeze. In this study, the shear storage modulus, loss factor and MR effect were measured in the shear mode. The MRE samples had dimensions of 10 mm  $\times$  10 mm  $\times$  3 mm. At room temperature, the following three testing types were used:

- (a) Testing of the magnetic field dependency: the frequency was 10 Hz, the shear strain amplitude was 0.5% and the magnetic field was swept from 0 to 1000 mT.
- (b) Testing of the shear strain amplitude dependency: the frequency was 10 Hz, the magnetic field was 0 mT and the shear strain amplitude was swept from 0.1% to 1.5%.
- (c) Testing of the frequency dependency: the shear strain amplitude was 0.5%, the magnetic field was 0 mT and the frequency was swept from 5 to 30 Hz.

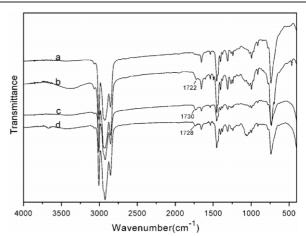
#### 2.6. Testing of the tensile strength

An electronic tensile machine (Jiangdu Jingcheng Test Instruments Factory, China, model JPL-2500) was used to test the tensile strength of these samples. Each sample was cut into a dumbbell, with a thickness of 2 mm and a middle width of 6 mm. The stretching rate was 500 mm min<sup>-1</sup>.

# 3. Results and discussion

# 3.1. Bound-rubber

Bound-rubber is the insoluble part of gross rubber after the extraction, belonging to the transition layer. This experiment adopted an extraction method to test the content of bound-rubber in the samples, as shown in table 1. As can be seen from this table, the weight fraction of bound-rubber increases monotonically with the increase of MA. The reason may be found in figure 1, which presents FT-IR spectra of modified BR with different contents of MA. Compared to the pure BR, the other samples have a new peak located at around 1730 cm<sup>-1</sup>, which is the characteristic peak of the -C=O group. It indicates that the polar MA has modified the nonpolar BR molecules chains. Moreover, a thin oxide film layer of Fe<sub>3</sub>O<sub>4</sub>



**Figure 1.** FT-IR spectra of modified BR with different contents of MA: (a) pure BR; (b) BR with 1 phr MA; (c) BR with 2 phr MA; (d) BR with 4 phr MA.

on the surface of carbonyl iron particles can be formed in air, which can absorb a small amount of water molecules and then form hydroxyl groups. When the carbonyl iron particles were added into the modified matrix during mixing, the BR molecule chains have a certain polarity and may easily interact with the hydroxyl groups which are on the surface of the carbonyl iron particles. Thus the tangled BR molecule chains on the surface of particles will grow in number and consequently increase the content of bound-rubber as the content of MA is increased.

The micrographs of the extractive gross rubber of four samples are shown in figure 2. It can be seen directly that with no added MA, many voids can be found between the particles and the bond between them is poor with little bound-rubber existence, as shown in figures 2(a) and (e). When the amount of MA is increased, the bond is improved and the space becomes gradually smaller, as shown in figures 2(b)–(d) and (f)–(h). Also, it can be seen that the adhesive particles begin to lump increasingly for the increased bound-rubber. It further confirms that the bound-rubber increases with the addition of MA.

## 3.2. Microstructure

Figure 3 shows the SEM micrographs of MRE samples, from which the interfacial adhesion between the rubber matrix and the magnetic particles can be observed. In figure 3(a), the sample without MA shows a poor bond between the rubber matrix and the magnetic particles, and many magnetic particles are exposed in the matrix. When the MA is added, the magnetic particles are embedded into the rubber matrix and the bond is improved, as shown in figure 3(b). When more MA is added, the magnetic particles are embedded more deeply and combine well with the rubber matrix, showing that the compatibility between the two phases is enhanced, as shown in figures 3(c) and (d). The bound-rubber consists of tightlybound-rubber, which is likely to aggregate in a flow field, and loosely-bound-rubber, which is attached to the particles through the tightly-bound-rubber and can experience very large deformations during flow [24]. The loosely-bound-rubber region can form a netlike structure between the rubber matrix

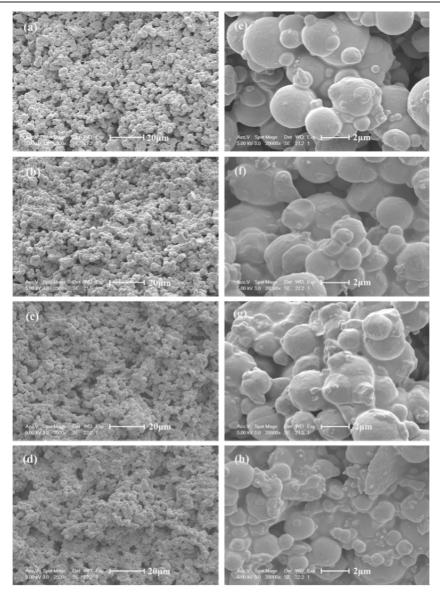


Figure 2. SEM images of four extractive gross rubber samples: (a)–(d)  $2500 \times$  respectively correspond to samples 1–4, and (e)–(h)  $20000 \times$  respectively correspond to samples 1–4.

and filler particles, and hence the bound-rubber can reflect the interaction between the two phases [24]. As the content of bound-rubber is increased with the increase of MA, as can be seen from table 1 and figure 2, the thick bound-rubber layer contains more loosely-bound-rubber and can combine the two phases better than a thin layer. So the microstructures of the samples show the compatibility between the two phases, which is strengthened by the incremental bound-rubber.

# 3.3. Shear storage modulus

The shear storage modulus of samples under various shear strain amplitudes and frequencies were experimentally characterized with the modified DMA, as shown in figures 4 and 5, respectively. Obviously, the shear storage modulus of samples is increased with the increasing MA. From the above results, the interfacial adhesion between the particles and matrix has been improved, which can reinforce the matrix. On the other hand, the polar MA can also modify the interaction between BR molecule chains by the hydrogen bonds, which can increase the stiffness of the matrix.

The shear strain amplitude dependency of the shear storage modulus is shown in figure 4. It can be seen from this figure that the shear storage modulus of samples has a reducing trend with increasing shear strain amplitude. When the shear strain amplitude increases, the motion of the rubber molecule chains and the sliding between the particles and the matrix increase, and consequently bonds among molecule chains as well as between the particles and matrix may be broken. Hence the shear storage modulus will decrease. However, the strain-induced decreases in shear storage modulus of the four samples are different, which increase gradually with the additional MA, as can be seen from the table 2. In this table, the strain-induced decrease in shear storage modulus is defined as  $A_{\Delta modulus}(\%) = \Delta modulus/modulus_{0.1}$ , where modulus<sub>0.1</sub> is the initial shear storage modulus and  $\Delta modulus$ 

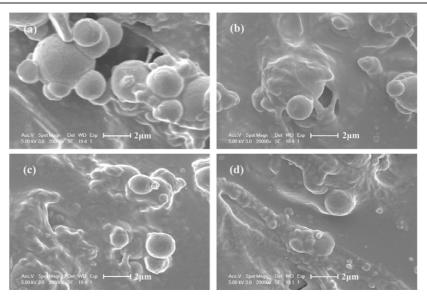


Figure 3. SEM images of four samples: (a)-(d) 20000× correspond to samples 1-4, respectively.

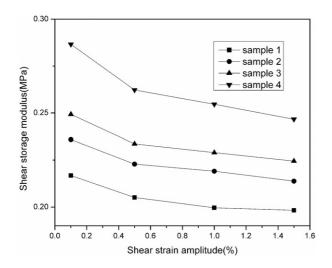


Figure 4. The shear storage modulus of four samples under different shear strain amplitudes.

 Table 2.
 The strain-induced changes of four samples in shear storage modulus and loss factor.

Sample	Strain-induced decrease in shear storage modulus (%)	Strain-induced increase in loss factor (%)
1	8.5	4.8
2	9.4	4.7
3	9.9	4.4
4	13.9	3.6

is modulus<sub>1.5</sub> – modulus<sub>0.1</sub>. The number of hydrogen bonds is gradually increased from sample 1 to sample 4 for the added MA. When little MA is added, the energy of the hydrogen bonds will be weak and can be easily broken under the shear force. When the strain amplitude increases, sample 4 can supply the most hydrogen bonds to be broken. Thus, the strain-induced decrease in shear storage modulus is increased with increasing added MA.

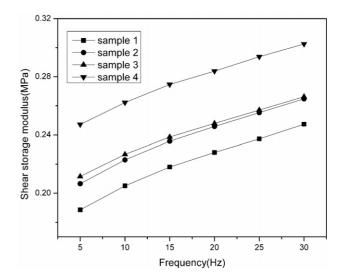


Figure 5. The shear storage modulus of four samples under different frequencies.

The frequency dependency of the shear storage modulus is shown in figure 5. We can see that the shear storage modulus presents a gradually increasing tendency with increasing frequency. It is known that when the frequency rises, the tangles in the matrix will increase as deformation of the molecule chains cannot keep up with the shear force. So the shear storage modulus increases with increasing frequency. Furthermore, at a certain frequency, the improved interaction among the BR molecule chains for the polar MA leads to increased tangles in the samples, and the enhanced interfacial adhesion between particles and matrix can also restrain the motion of the molecule chains. Hence, the shear storage modulus increases with increasing added MA.

# 3.4. Loss factor

As shown in figures 6 and 7, the loss factor of these samples under various magnetic fields and strain amplitudes were

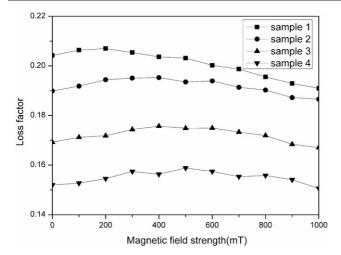


Figure 6. The loss factor of four samples under different magnetic field strength.

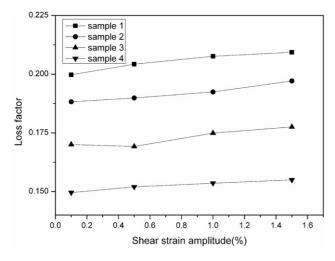
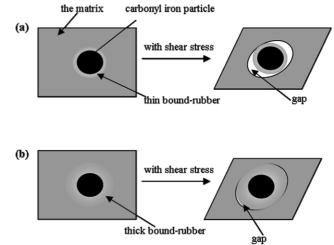


Figure 7. The loss factor of four samples under different shear strain amplitudes.

experimentally characterized with the modified DMA. It is known that the damping properties of composites reflect the ability of energy dissipation of materials [25]. The energy dissipation of MREs mainly comes from the matrix and the interfaces between the matrix and the particles.

The magnetic field dependency of the loss factor is shown in figure 6. As can be seen from this figure, the loss factors of samples show a tendency of increase plus decrease with increasing magnetic fields. This phenomenon has been reported in our previous work [26]. The loss factor of MRE samples decreases steadily when the content of MA increases. For example, at zero field, the loss factor of sample 4 decreases 25.6% compared with sample 1.

The shear strain amplitude dependency of the loss factor is shown in figure 7. It can be seen from this figure that the loss factor of samples shows an increasing trend with the strain amplitude. It is known that the deformation of the matrix becomes larger with increased shear strain amplitude. The slipping friction between two interfaces as well as among the molecular chains will increase. Thus the energy dissipation will increase. However, the strain-induced increases in loss



**Figure 8.** The sketch of the interaction between a magnetic particle and the matrix under a shear stress: (a) the magnetic particle with thin bound-rubber; (b) the magnetic particle with thick bound-rubber.

factor of the four samples are different, and decrease gradually with additional MA, as can be seen from table 2. In this table, the strain-induced increase in loss factor is also defined as  $A_{\Delta loss}(\%) = \Delta loss/loss_{0.1}$ , where  $loss_{0.1}$  is the initial loss factor and  $\Delta loss$  is  $loss_{1.5} - loss_{0.1}$ .

In figure 8, the simple sketch shows the interaction between the two phases with the various content of boundrubber. When the bound-rubber layer is thin on the surface of particles, which is mainly tightly-bound-rubber, the interfacial adhesion between the two phases should be very weak, as shown in figure 3(a). Under the shear strain amplitude, the rubber matrix has a certain deformation but the bound-rubber cannot follow, and the weak interfacial adhesion is damaged easily, causing a big gap, as shown in figure 8(a). As the sample is exposed to magnetic fields, interparticle magnetic forces appear and sliding is generated, the slipping friction among the two interfaces will be great due to the big gap, and the energy dissipation means that the damping will be large. When the bound-rubber layer is thickened gradually, as shown in figure 8(b), the loosely-bound-rubber increases and the interfacial adhesion between the two phases is enhanced, as can be seen from figures 3(b)-(d). The loosely-bound-rubber can follow deformation with the matrix and the gap appearing will be small between the two phases under the shear strain amplitude, and thus the slipping friction will decrease. Thus, the damping is decreased by the enhanced interfacial adhesion between the two phases arising from added MA, as shown in figure 6. Moreover, the polar MA can modify the interaction between the BR molecule chains, also increasing the stiffness of matrix and reducing the slipping friction between the BR molecule chains. So the increase in stiffness of the matrix is another reason for the decrease of loss factor.

The strain-induced increase in loss factor also closely relates to the bond degree between the two phases, which can reflect the difficult degree of sliding between the matrix and particles. The better the bond between two phases is, the more difficult the sliding is, and the energy dissipation will be decreased. Also the enhanced interaction between BR

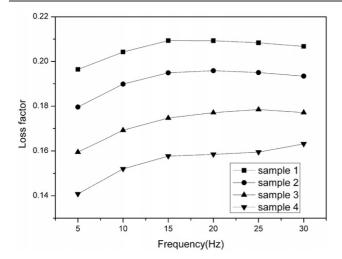


Figure 9. The loss factor of four samples under different frequencies.

 Table 3. The tensile strength of each sample.

Sample	Tensile strength (MPa)
1	0.7
2	0.89
3	0.9
4	0.93

molecule chains can reduce the sliding of molecule chains. Consequently the loss factor will change slightly with an increase of strain. In other words, the stability of the damping of MREs with various strains has been enhanced for the added MA. The effect of strain should be considered, for the strain amplitude does not remain constant in practical applications. If the strain amplitude changes obviously, the damping property of the MREs is not only a function of the magnetic field but also greatly influenced by the strain, which is not what we want. Hence the stability of the damping of MREs with various strains is significant.

The frequency dependency of loss factor is shown in figure 9, in which a tendency of increase plus decrease with the increasing frequency can be seen. This result is consistent with the report of Sun *et al* [16]. It can be seen that the frequency of the largest loss factor of these samples gradually increases when more MA is added. The frequencies of the largest loss factor are 15 Hz, 20 Hz, 25 Hz, 30 Hz, respectively from sample 1 to sample 4. This may relate to the storage modulus. The larger the storage modulus is, the higher the natural frequency will be.

# 3.5. Tensile strength

The tensile strength of each sample is shown in table 3. It indicates that added MA can also enhance the tensile strength of MREs. MREs belong to a class of particle reinforced composites, the particles in the matrix can obstruct the movement of rubber molecule chains and the dislocation within the MREs, and then prevent the matrix from rising stress concentration. The better the bond between the two phases is,

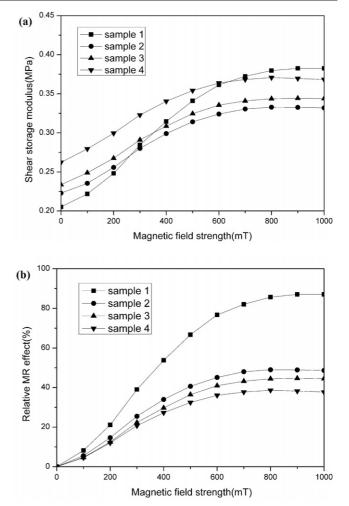


Figure 10. The shear storage modulus and the relative MR effect of four samples under different magnetic field strength.

the higher the reinforcement will be. When the bond between two phases is poor, as shown in figure 3(a), many interstices can be found, which can easily lead to stress concentration, and so the tensile strength of the MREs is reduced under a tensile force. The bond is enhanced and the interstice is reduced when the MA is added, as can be seen from figures 3(b)-(d), the effect of stress transfer is improved and the stress concentration is decreased. In addition, the stiffness of the matrix has been increased by the improved interaction between the rubber molecule chains, and the movement of BR molecule chains is reduced. Also, the enhanced compatibility reduces the voids in the composite, which can decrease the stress concentration. For the above reasons, the tensile strength is increased.

# 3.6. MR effect

The field dependence of shear storage modulus and relative MR effect for the four samples are shown in figure 10. In figure 10(a), the shear storage modulus shows an increasing tendency with magnetic field before reaching magnetic saturation at high field strength. It agrees well with our previous work [26]. In contrast, the shear storage modulus of sample 1, without MA, increased sharply with increased magnetic field. It also indicates that the MA did influence

the shear storage modulus of the samples, which relates to the relative MR effect. As shown in figure 10(b), a decreased tendency with an increase of MA is presented. The relative MR effect is defined as  $\text{RMe}(\%) = \Delta G_{\text{max}}/G_0$ , in which  $G_0$  is the initial modulus and  $\Delta G_{\text{max}} = G_{\text{max}} - G_0$ , where  $G_{\text{max}}$  is the storage modulus when the particle is in magnetic saturation. It can be seen that  $G_0$  was enhanced with increasing MA for the improved stiffness of the matrix, as shown in figure 10(a). During the pre-forming process, whether it is hard or easy for particles to move depends on the viscosity resistance of the matrix. Due to the enhanced interaction among the BR molecule chains, as well as the improved interfacial adhesion between particles and matrix, the resisting force for particles to move within the matrix becomes great, and so the particles are difficult to form into the regular chain structure. However, some studies have reported that MREs with regular chain structures have a greater MR effect [16, 20]. It indicates that MA can reduce the relative MR effect of MREs. Therefore, the MA content should not be too high.

## 4. Conclusion

In this study, four MRE samples with different contents of maleic anhydride (MA) were prepared. The boundrubber was measured and the microstructures of samples were observed. The dynamic properties were characterized under different magnetic field strengths, shear strain amplitudes and frequencies; also the tensile strength was tested. The MA additive increases the content of bound-rubber, which can affect the interaction between the two phases and improve the interfacial adhesion from the microstructures of samples. Thus, the compatibility between the magnetic particles and the rubber matrix was enhanced. Consequently, this process has resulted in a decrease of MRE damping properties, an enhancement of the stability of the MRE damping properties under various strains, an increase of shear storage modulus and an improvement of the tensile strength. Furthermore, the MA additive increased the viscosity resistance of matrix, which reduced particle movement in the matrix. This resulted in the reduction of the relative MR effect. MREs with low and stable loss factor, as well as good strength, are significant for some applications-for example, adaptive tuned vibration absorbers based on MREs, where the lower the MRE damping is, the higher the absorber effectiveness can be. Thus, the development of MA additive based MREs provides a feasible method.

# Acknowledgments

Financial support from NSFC (Grant No. 10672154) and SRFDP of China (Project No. 20093402110010) is gratefully acknowledged.

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