Carbonyl iron powder surface modification of magnetorheological elastomers for vibration absorbing application

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Carbonyl iron powder surface modification of magnetorheological elastomers for vibration absorbing application

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Abstract
With excellent characteristic of magnetic-control stiffness, magnetorheological elastomer (MRE) is well suited as a spring element of vibration absorber. To improve the vibration attenuation performance of MRE vibration absorbers, this paper expects to improve the mechanical strength and reduce the loss factor of MRE by interface modification. The surface of carbonyl iron powder (CIP) was modified with silica coating by a simple and convenient approach. Several MRE samples, with different proportions of modified CIPs were fabricated under a constant magnetic field. The morphology and composition of modified CIP were characterized by scanning electron microscope and Fourier transform infrared spectra. The results indicated that the modified CIPs were coated with uniform and continuous silica, which can make a better combination between particle and matrix. The tensile strength, magnetorheological properties and the damping properties of the MRE samples were tested by material testing machine and rheometer. The experimental results demonstrated that the loss factor of the MRE which incorporated with modified CIPs decreased markedly, and the tensile strength of such material has been much improved, at the same time this kind of MRE kept high MR effect. It is expected that this MRE material will meet the requirements of vibration absorber.

Keywords: magnetorheological elastomers (MRE), surface modification, the dynamic mechanical properties, vibration absorber

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

1. Introduction
There are often some harmful vibrations in most mechanical, civil, construction systems. These vibrations may bring about damage, mechanical fatigue, and shorten the service life. To avoid these problems, many researchers have paid attention to suppress these unwanted vibrations by several vibration isolation devices [1–4]. Among them, dynamic vibration absorbers (DVA) are widely used in engineering applications to suppress undesired vibration of structures [5]. It is often used as an additional vibration subsystem to be installed on the target. By designing or adjusting the mechanical parameters of the vibration absorber itself in real time to change the vibration condition of the object, so as to realize the vibration control of the target [6]. However, traditional DVA is only effective at narrow working frequency range due to its fixed parameters. With the external excitation frequency varying, the vibration attenuation effect of the DVA decreases or even collapses, which restricts the practical applications of the DVA [7]. The adaptive tuned vibration absorber (ATVA) can adjust its natural frequency in real time to track the uncertain or time-varying excitation frequencies and this is one of the most effective way to improve the vibration attenuation performance [8]. It is known that the core of the ATVA is its stiffness which can be altered by employing the adaptive spring element. While, with applying an external magnetic field, the modulus of MRE can be controlled rapidly, continuously, and reversibly, as it is a kind of smart...
material [9, 10]. And this characteristic makes it very suitable for using as the smart springs in the ATVA. The working principle of the shear type MRE vibration absorber is tracking the change of the external excitation frequency by adjustment the natural frequency of the vibration absorber with the control current. In order to obtain a higher vibration attenuation performance of the ATVA, the damping of spring element should be as small as possible [11, 12]. Low damping leads to high vibration reduction and the better transferring capability of vibration [13]. However, the damping of conventional MRE is relatively large for application in ATVA, although it has the advantage of controlling rapidly and reversibility. In addition, ATVA works in the resonance region and the amplitude of vibration is higher, so the MRE as the spring element which requires high mechanical strength. Therefore, it is necessary to find some effective methods to optimize the preparation process of MRE to reduce the damping and improve the mechanical strength.

It is known that MRE can be regard as a kind of particle-reinforced polymer composite material. Therefore, the matrix, the particles and the interface between the matrix and particles are the three most important factors that affect the properties of MR materials [14]. Because the matrix and particles are organic and inorganic, respectively, it is difficult to achieve homogenous covering of magnetic powder by a polymer restricted by the immiscibility between the organic and inorganic parts [15, 16]. The poor interface between the particle and matrix will lead to poor mechanical properties of MRE and increase the damping of the material, when the particle is slipping, thus affecting the magneto-rheological damping characteristics. As a consequence, in order to improve the mechanical strength of MRE and reduce the damping, the interface modification is needed. In recent years, several researches on interface structure between matrix and magnetic particles of MRE have been carried out. By choosing three different kinds of surfactants, i.e. anionic, nonionic and compound surfactants to modify surface of CIP, Jiang [16] proved that the surface modification could improve the compatibility between particles and rubber matrix, as well as the special self-assembled structure of particles. Such a special structure has been proved beneficial to the improvement of the relative MR effect. The use of silane coupling agent methyltrimethoxysilane by Kashima et al [17] was intended to change the surface of the carbonyl iron from hydrophilic to hydrophobic. Qiao et al [18] reported that after the CIP being modified by the titanate coupling agent to enhance their compatibility with the SEEPS matrix, the obtained CI/SEEPS composites became much softer with a higher MR effect and showed almost the same mechanical properties as the SEEPS matrix does. Li et al [19] reported that by using core–shell structured poly methyl methacrylate coated particles as a way of surface modification, it could make loss factor smaller and more stable because of decreasing the relative motion between particles and matrix. Malecki et al [20] reached dynamic mechanical properties of magnetorheological composites containing silica-coated CIP and observed that the composites with modified CIP have some influence on hysteresis loops’ areas and MR effect.

However, the current researches mainly focused on improving the MR effect of MRE, the existing researches on reducing the damping as well as enhancing the mechanical strength by improving the interface are less, and the adjustment range of stiffness of MRE is not enough for the application in ATVA. Thus, more studies on the interface modification of MRE are needed. Surface modification is one of the various methods to improve the compatibility between the hydrophobic polymer and the hydrophilic fillers [21, 22]. Using silane coupling agent as a surface modification method is widely adopted. This mainly because amorphous silica layers on the surface of CIP have advantages of providing thermal and mechanical stability of iron particles which prevent the CIP from further oxidation, in addition it could improve the adhesion to the polymer and develop the dispersion of the iron in the matrix [23, 24]. After these processes, the connection between iron and matrix will be more closely, due to the silica playing a covalent bond just as a bridge, between the iron particles and the matrix. Using silane coupling agent as a surface modification method not only enhances the bonding strength between iron and matrix but also improves dispersibility of the iron powder and reduces agglomeration.

With the hope of solving the problem of immiscibility between the particle and matrix, thereby reducing the damping of MRE and improving mechanical strength. In this investigation, silica coated the CIP was prepared by sol-gel method as a way of surface modification. At the same time, the morphology and composition of modified CIP were characterized by SEM and the Fourier transform infrared (FTIR) spectra. In addition, in order to verify the enhancement of the properties of MRE by the surface modification, two kinds of MRE samples were prepared with different weight percentage of modified CIP and non-modified CIP. Besides, the tensile strengths, the magnetorheological properties and the damping properties of the two groups MREs were also studied.

2. Experiments

2.1. Materials

Modified CIP: the soft magnetic particles made up of carbonyl iron particles (CIP: type CN, $d_{50} = 6.5\mu m$) were provided by BASF Corporation, Germany. Tetraethoxysilane (TEOS, 99%, boiling point = 169°C) was used as silica precursors of modification (Jining Baichuan Chemical Co. Ltd, China). Ethanol (boiling point = 78°C) was used as a reaction medium. Hydrochloric acid (36.5%–38%) was employed as a catalyst of reaction (Chongqing Chuandong Chemical (Group) Co.,Ltd, China).

D-MRE: diphenylmethane diisocyanate (MDI: 4, 4 ≈ 50%, 2, 4 ≈ 50%, Yantai Wanhua Polyurethanes Co. Ltd, China) and castor oil (CO: Sinopharm Chemical Reagent Co. Ltd,China) were selected as the main materials for preparation of polyurethane (PU) which was the matrix of MRE. Dibutyl phthalate (DBP: Tianjin Bodi Chemical
Holding Co., Ltd, China) was used as the plasticizer. CIP and modified CIP were used as the filler particles.

2.2. Synthesis of modified CIP

First of all, each component of the raw materials was accurately calculated and weighed, respectively. Secondly, in order to obtain the products of hydrolysis reaction, TEOS was mixed with ethanol in an appropriate molar ratio (TEOS: ethanol = 1:7.6). Then two drops hydrochloric acid was added to provide the acidic pH environment of reaction and served as a catalyst. The mixture was stirred for about 2 h, during which a silica overlay network was created through hydrolysis and condensation reaction. As an important reactant, ethanol was used for the hydrolysis reaction and the preparation of the hydrolyzate based on the hydrolysis of TEOS. According to these conditions, more homogenous materials in a molecular scale could be obtained. In order to extend the network structure on the CIP surface, the residual reaction solution was kept for sol-aging for 24 h. After completion of the aging process, the mixture was placed in a vacuum oven in order to remove the air bubbles, and then filled the mixture into an aluminum mold. After curing under a constant magnetic field (0.6 T) for about 24 h, two kinds of MRE samples (M-MRE and N-MRE) were prepared with modified CIP and non-modified CIP.

2.3. Preparation of MRE samples

Firstly, MDI was added into CO stirred well in a beaker while the temperature remained at 80 °C for 10 min to obtain the polyurethane (PU) matrix. Next, DBP and iron particles were mixed with the mixture successively with vigorous stirring for 30 min and the temperature was remained to 80 °C. Finally, when the viscosity of reactant increased obviously approximately after 2 h, the mixture was placed in a vacuum oven in order to remove the air bubbles, and then filled the mixture into an aluminum mold. After curing under a constant magnetic field (0.6 T) for about 24 h, two kinds of MRE samples (M-MRE and N-MRE) were prepared with modified CIP and non-modified CIP.

Figure 1. Scheme of the preparation of modified CIP based on TMOS.
respectively. The detailed compositions of the MRE samples are shown in Table 1.

### 2.4. Characterization

The microstructures of modified CIP and MRE samples were observed by a scanning electron microscope (SEM, TESCAN VEGA). FTIR spectroscopy was used to identify the existence of silica shell on CIP. FTIR spectra was obtained on a NicoletiN10 FTIR spectrophotometer (Thermo Fisher Scientific, USA) scanning from 400 to 4000 cm\(^{-1}\).

The tensile strength values were obtained by a material testing machine (CTM2100, Shanghai, People’s Republic of China) with the crosshead speed of 5 mm min\(^{-1}\). Each sample was cut into a dumbbell shape with a thickness of 2 mm and a middle width of 4 mm. All tensile strength values were the average of the five measurements.

The dynamic mechanical properties of the MRE samples were measured by using an advanced commercial rheometer (Modal: MCR301, Anton Paar) with the magnetic flux density ranging from 0 to 1 T. The strain amplitude and the frequency were set to 0.1% and 10 Hz, respectively. The schematic diagram of the testing part of the rheometer can be acquired from our previous work [26].

### 3. Results and discussion

#### 3.1. Morphology of modified CIP

The SEM images of non-modified CIP and modified CIP are shown in Figure 2. The microstructure of non-modified CIP, seen in Figure 2(a), showed a regular spherical surface, while the morphology of modified CIP, seen in Figure 2(b), coated with a thin layer of gauze-like substance on the surface of CIP.

The FTIR spectra of non-modified CIP and modified CIP are revealed in Figure 3, where the bands at around 654 cm\(^{-1}\) are related to the Fe–O groups in two curves. The main absorption bands at around 1632 cm\(^{-1}\) and 3437 cm\(^{-1}\) are observed in the two samples which can be assigned to the H–O–H stretching modes and bending vibration of the free or

### Table 1. Compositions of MRE samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Matrix (wt%)</th>
<th>CIP (wt%)</th>
<th>Sample</th>
<th>Matrix (wt%)</th>
<th>Modified-CIP (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-40</td>
<td>60</td>
<td>40</td>
<td>M-40</td>
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<tr>
<td>N-50</td>
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<td>M-60</td>
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<tr>
<td>N-70</td>
<td>30</td>
<td>70</td>
<td>M-70</td>
<td>30</td>
<td>70</td>
</tr>
</tbody>
</table>

#### Figure 2. (a) The SEM micrograph of non-modified CIP (b) the SEM micrograph of modified CIP.

#### Figure 3. FTIR spectra of (a) non-modified CIP and (b) modified CIP.
adsorbed water, respectively. Existence of OH and Fe–OH groups are so important on the core–shell structure due to their reaction with Si–OH groups in the shell sol. The new band was appeared in the FTIR spectra of the modified CIP at around 1059 and 2971 cm$^{-1}$. The band at 1059 cm$^{-1}$ is belonged to Si–O–Si groups. The existence of these groups in sample is a sign of particles coating. But as it shown the intensity of Si–O–Si band is low, which means that the shell thickness should be thin. The absorption band at 2971 cm$^{-1}$ is associated with the C–H of vinyl groups. These results confirmed the presence of an amorphous silica coatings on the surface of CIP and showed that the surface modification of CIP by TEOS was successful.

3.2. Microstructures of MR elastomers

The SEM micrographs of the MRE samples with different kinds of CIP are shown in figure 4. As illustrated in figure 4, the white parts in the images are the iron particles while the black parts are the PU rubber. It has been expected that the modification of CIP surface could improve the compatibility of iron particles and the matrix.

The SEM analysis provided direct evidence for evaluating the compatibility of these two phases. It can be seen clearly that the distribution of the modified CIP in the sample is more uniform, and there are less bubbles or defects in the sample with modified CIP than the sample with non-modified CIP. The reason of this phenomenon is that the surface covered by silica layer can improve the dispersion of iron particles and the adhesion between iron particles and matrix [27]. It also can be seen that the chain like structures and the aligned directions of the samples are indicated by the red arrows seen from figure 4. The adulteration of modified CIP plays an important influence on the microstructure of MRE, presented in figures 4(a) and (b), when the modified CIP are used as the filler particle in the MRE sample, these particles can be more uniformly distributed in the rubber matrix while the sample with non-modified CIP has more particle agglomerations. This is mainly due to the sol-gel method as a way of CIP surface modification which has improved the compatibility between the organic and inorganic parts.

3.3. Mechanical properties of MRE

The result of tensile testing is exhibited in figure 5, the values are the average of the five measurements in which the tensile strength of MRE samples filled with modified CIP increases than the samples of non-modified CIP. In addition, the standard deviation from the average has been shown in the form of error bars in figure 5. It mainly because that the compatibility between the matrix and particles has improved by the surface modification of CIP, resulting in modified CIP distributed more uniformly in the matrix and reduced the gaps.
which exist between CIP and matrix. In addition, stress concentration is more likely to occur at the interface defects when MRE subjected to stress. Due to the interface modification reduced the bubbles and defects, the stress concentration of the MRE with modified CIP is alleviated [28, 29]. This phenomenon is also benefit to improve the tensile strength of MRE. Therefore, it is credible that the way of surface modification has a significant effect on the mechanical properties.

3.4. Magnetorheological property of MRE

When a certain magnetic field strength is imposed to the MRE samples, as the result of an induced magnetic dipole–dipole interaction, the CIP is easily magnetized and arranged along the magnetic field direction which is the main reason of the change of shear modulus. The relative MR effect is defined to evaluate the relative change of shear storage modulus, generally expressed by the formula [30]

\[ G_{\text{MRE}}(\%) = \frac{(G - G_0)}{G_0} \times 100\% \]

(1)

where \(G_0\) is the initial storage modulus, \(G\) is the maximum shear storage modulus under variable applied magnetic fields. The shear storage modulus of MRE samples with different proportions of non-modified CIP and modified CIP are shown in figure 6, and the relative MR effect under different magnetic fields are shown in table 2. The testing results revealed that the initial modulus of the samples with modified CIP is higher than the samples of non-modified CIP. As can be seen in table 2, the initial modulus of sample M-40, M-50, M-60, M-70 is 0.1949 MPa, 0.2609 MPa, 0.4969 MPa and 0.5653 MPa respectively, which is higher than 0.1706 MPa of sample N-40, 0.2316 MPa of sample N-50, 0.4101 MPa of sample N-60 and 0.5109 MPa of sample N-70. Moreover, the

<table>
<thead>
<tr>
<th>Samples</th>
<th>(G_0) (MPa)</th>
<th>Magnet-induced (\Delta G)</th>
<th>Relative MR effect (\Delta G/G_0(%))</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-40</td>
<td>0.1706</td>
<td>0.2045</td>
<td>119.87</td>
</tr>
<tr>
<td>M-40</td>
<td>0.1949</td>
<td>0.2819</td>
<td>144.64</td>
</tr>
<tr>
<td>N-50</td>
<td>0.2316</td>
<td>0.7123</td>
<td>307.56</td>
</tr>
<tr>
<td>M-50</td>
<td>0.2609</td>
<td>0.9373</td>
<td>359.26</td>
</tr>
<tr>
<td>N-60</td>
<td>0.4101</td>
<td>1.9259</td>
<td>469.61</td>
</tr>
<tr>
<td>M-60</td>
<td>0.4969</td>
<td>2.4561</td>
<td>494.28</td>
</tr>
<tr>
<td>N-70</td>
<td>0.5109</td>
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<td>500.12</td>
</tr>
<tr>
<td>M-70</td>
<td>0.5653</td>
<td>2.9957</td>
<td>529.94</td>
</tr>
</tbody>
</table>

Figure 6. Shear storage modulus under different magnetic field of MRE samples with different contents of non-modified CIP and modified CIP.
magneto-induced modulus ($\Delta G$) of the MRE samples with different proportions of modified CIP is much higher than the MRE samples with different proportions of non-modified CIP. According to the formula above the relative MR effects of sample M-40, M-50, M-60, M-70 are higher than the sample N-40, N-50, N-60, N-70, respectively. The main reason is that the tiny structures composed of particles constrain a fraction of rubber matrixes, and these matrixes become a part of chain like structures, which appear as rigidity. Because of the better combination with matrix, the modified CIP will be more firmly connected with the matrix, and the strength of the whole chain structure becomes greater, which causes a higher magneto-induced modulus.

### 3.5. Damping property of MRE

The loss factor of composite material which mainly caused by the interaction among different inner phases at the interface is the representative of the materials to dissipate energy. In the MRE, the overall damping capacity can be evaluated by the formula.

$$D_{MRE} = D_C + D_I + D_M,$$

where $D_C$, $D_I$ and $D_M$ are the intrinsic damping, interface damping, and the magnetomechanical damping, respectively. The magnetomechanical damping is much smaller compared to the others, and it does not cause significant changes in the overall damping [31]. The intrinsic damping is determined by the content and the constitutive damping capacity of each individual component, but as the loss factor of CIP is far lower than that of the polymer matrix, $D_C$ is mainly determined by the constitutive damping capacity of the polymer matrix of the MRE [32].

For the MRE samples with different contents of modified CIP, the curves of loss factors under different magnetic fields range from 0 to 900 mT are shown in figure 7. It can be seen that as the CIPs trended to form a columnar parallel under the magnetic field, the loss factor of MRE samples shows a general decreasing trend. With the increasing magnetic flux density, more CIPs began to agglomerate and more columnar parallel structures were formed. When the magnetic flux density reached the saturation magnetization of the iron particles, the number of columnar parallel structures did not increase and the loss factor of all MRE samples tended to level off [26]. In addition, the loss factor of the samples with modified CIP is lower than that of the samples with non-modified CIP. This can be attributed to the following reasons: due to the bubbles and defects which categorized into the matrix are reduced by interface modification, the samples with modified CIP have a lower $D_C$ than samples with non-modified CIP. Besides, by using the surface modification, the distribution of CIP in the

![Figure 7. Loss factor under different magnetic field of MRE samples with different contents of non-modified CIP and modified CIP.](image)
MRE is more uniform and the combination between modified CIP and the matrix is better, so this kind of MRE has a lower $D_i$. In addition, the relative movement between the matrix and the CIPs created more dissipation of energy. The slippage occurred more easily between the non-modified CIP and the matrix, there has a friction phenomenon occurred at different inner phases of the interface, which resulted in more energy consumption. Due to functionalization of CIP surface, there is a better combination between modified CIP and the matrix so the relative slippages of molecular chains decreased, thereby causing a lower energy dissipation. This also explained why the loss factors of samples of non-modified CIP were higher than those of modified CIP. The above results also indicated that the silica coated CIP as a way of surface modification can reduce the MRE sample’s loss factor. This conclusion is significant for some devices because it is known that the effect of absorber is mainly affected by the damping property, while low damping leads to high vibration reduction while high damping results in poor vibration suppression. In such a case, using modified CIP as the filler particle to add into the matrix would provide an effective way to develop the desired MRE with high MR effect and low loss factor.

4. Conclusion

In this study, magnetic CIP modified by TEOS was synthesized via sol-gel method, and a novel type of MRE with modified CIP was prepared. The dynamic mechanical performances of these MREs were also measured and analyzed. The results revealed that the tensile strength, the MR effect were increased and the loss factor was also markedly reduced by modification of CIP. The analyses indicated that surface modification by coating silica on the surface of CIP could improve the immiscibility between the organic and inorganic parts. These influences are very helpful in avoiding the magnetic energy loss originating from the particle agglomeration or gaps. In addition, these study results indicated that sol-gel method as a way of CIP surface modification is an efficient measure to develop the desired MRE with excellent mechanical performance, high MR effect and low loss factor. The fabricated MREs studied here can be used as an elastic element in adaptive tuned vibration absorbers for its excellent comprehensive performances.

Acknowledgments

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