

Magnetorheological Elastomers with Efficient Electromagnetic Shielding

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ABSTRACT

This study is concerned not only with an investigation of rheological performance of magnetorheological elastomers (MREs) with random distribution based on modified magnetic particles, but also discusses their electromagnetic shielding properties for the first time. The current work further highlights the necessity of magnetic particles protection against oxidation species affecting them in real application conditions. Two kinds of MREs differing in matrix type represented by silicone elastomer and thermoplastic elastomer are prepared, and pristine carbonyl iron (CI) particles and siloxane-modified CI particles were used for each matrix. The difference in magnetic properties after the modification of particles is negligible while the modification significantly improves the anti-acid-corrosion properties. Moreover, modification of particles surface enhances their compatibility with silicone elastomer matrix which is evidenced by the measurements of viscoelastic properties under the external magnetic field applied. It is also shown that all the developed MREs can be successfully applied for the absorption of electromagnetic radiation in ultra-high frequency band, namely the frequency range from 700 MHz to 1.6 GHz can be readily covered by these materials.

Keywords

Electromagnetic shielding; Magnetorheology; Elastomer; Coating; Smart material

1. Introduction

Magnetorheological elastomers (MREs) are multi-phase composite materials frequently described as a solid analogue to MR suspensions. Generally, MREs consist

of an elastomeric matrix interspersed with micron sized ferromagnetic particles and hence combine the functionality of a highly elastic polymer matrix with the magnetic field-responsive properties [1–3]. The particles of choice for MREs are multi-domain iron particles satisfying requirements for soft magnetic material with high induced magnetic dipoles due to high magnetic permeability and high magnetization saturation while the residual magnetization is minimal. The magnetic particles can be incorporated into the elastic body either in random distribution or in ordered structures [4,5]. If a uniform magnetic field is applied to the reactive mixture during the cross-linking process, formed particle chains become locked in the elastomer. The resulting composites are called transversely isotropic MREs [6]. However, from a manufacturer's point of view it would be convenient if the use of magnetic field during cross-linking process could be avoided.

When the external magnetic field is applied to the MRE, the magnetic particles interact with the surrounding polymer chains and alter their positions. This results in an overall shape change of the composite. Thus, the coupling of magnetic field with the elastic response of reversible extend from 5 – 700 % depending on the type of elastomeric matrix can result in powerful actuators such as artificial muscles and sensors [7,8]. Although the MREs are quite new materials, the giant deformational effects, high elasticity, and quick response to magnetic fields, *i.e.* field-dependent control of modulus, open new opportunities for using such materials for various applications [9,10]. However, there are still some limitations for their utilization such as corrosion of magnetic particles inside the systems due to the diffusion of corrosion agents through the polymeric matrix or insufficient wetting of magnetic particles with the matrix. Thus, the modification of particles surface is an effective tool for the

improvement of MR performance in time due to the improved compatibility between magnetic particles and matrix [11], and improved oxidation and chemical stability.

Furthermore, the objective of the design of electromagnetic shielding materials (ESMs) widely used in civil and military fields due to their ability to absorb undesirable electromagnetic signals and wave pollution is to obtain the material having minimal thickness and the lowest possible reflectance within the widest possible operating bandwidth [12]. One of the possible ways providing these properties is the use of multi-component core-shell structured magnetic particles with magnetic core and electrically conducting shell dispersed in polymer matrix since the absorption of electromagnetic waves in such materials is governed by various loss mechanisms related to the magnetization and electric polarization processes [13].

Nanostructured tetraethylorthosilicate (TEOS) grafted onto the carbonyl iron (CI) magnetic particles was used in this study to evaluate the influence of siloxane-based coating of particles on their compatibility with two different elastomeric matrixes, namely thermoplastic and silicone elastomer matrix. Furthermore, this study will indicate a broadening in application spectrum of current MREs via the examination of their electromagnetic shielding properties.

2. Material and methods

2.1. Reagents

Carbonyl iron (ES grade, BASF, Germany) spherical particles with iron purity > 97.7 % and size 2–3 μm were used as the magnetic agent in MREs under investigation. For the modification of CI particles, namely tetraethylorthosilicate (TEOS, purity = 98%) produced by Sigma-Aldrich (St Louis, USA), hydrochloric acid

(HCl, ACS reagent, 37%) and toluene (anhydrous, 99.8%) both produced by Penta Chemicals (Czech Republic), were used. All the chemicals were used without further purification.

2.2. Coating of CI particles by 3APTS

Prior to the surface modification of the CI particles, their cleaning from any contamination and activation was performed via 0.1 M HCl according to the process described in the reference [14] in more details. The next step in the coating is the functionalization of the activated CI particle surface with TEOS polymer. Briefly, 100 g of activated CI particles were dispersed in 150 mL of a non-polar solvent toluene into a three-neck flask fitted with a mechanical stirrer and a reflux condenser. The entire assembly was placed into a heating mantle and stirred at 110 °C with sequential adding of TEOS (10 mL) for 20 min followed by the reaction under rigorous stirring (200 rpm) for additional 8 h at 110 °C. Afterward, the coated CI particles were separated from the toluene by sedimentation accelerated with a magnet, washed with distilled water (2 times, 200 mL each), ethanol (2 times, 200 mL each), and acetone (2 times, 200 mL each), and dried at a pressure of 200 mbar at 50 °C for 10 h.

2.3. Particles characterization

A Fourier transform infrared spectroscopy (FTIR, Nicolet 6700, Thermo Scientific, USA) was performed to verify the successful modification of the CI particles with TEOS. The FTIR spectra were recorded using the attenuated total reflectance (ATR) technique with Germanium crystal in a range of 680–4000 cm^{-1} at 64 scans per spectrum at a 2 cm^{-1} resolution. The magnetostatic properties of particles under

investigation were measured at room temperature using a vibrating sample magnetometer (VSM 7407, Lakeshore, USA) in a magnetic field of up to 1200 kA m⁻¹. The measurements were carried out at a frequency of 82 Hz, and the vibration amplitude was 1.5 mm. In order to examine the resistance to corrosion by acids, the same amounts of pristine and TEOS-modified CI particles were dispersed in a hydrochloric acid solution of concentration 0.1 mol/L, and the increasing pH values due to the reduction of the acid solution oxidizing the iron-based particles as a function of time were recorded via pH-meter (SensoDirect pH110, The Tintometer Ltd, United Kingdom).

2.4. MREs preparation

Two types of MREs differing in the matrix materials were prepared. The first matrix material was silicone elastomer represented by Silgard 184 supplied by Dow Chemical Company (USA) while the second one was thermoplastic elastomer represented by Vistamaxx 61020FL supplied by ExxonMobil (USA). Furthermore, two types of MREs with 40 vol.% particles loading for each matrix material were investigated differing in the surface modification of CI particles, *i.e.*, the first was based on pristine CI particles and the second was based on CI particles modified with TEOS. Hence, four types of MREs were prepared and investigated to evaluate the MR performance as well as electromagnetic shielding properties dependence on the matrix material and particles modification used in detail.

The mixing of the two-component silicone elastomer (10:1) with the corresponding magnetic particles was carried out by mechanical stirring at room temperature until the mixture reached a homogeneous state. Then, the suspension was degassed at 300 mbar

for 10 min. Afterwards the mixture was cast to a mould with the thickness of 0.5 mm, again degassed, and cured at 40 °C for 3.5 h.

In the case of thermoplastic elastomer, the mixing was carried out via a scientific twin screw extruder (Labtech Engineering Co. Ltd., Thailand) at 170 °C for 5 min. The compound was subsequently formed to plate of thickness 0.5 mm via compression molding at 170 °C for 5 min.

2.5. MREs characterization

Complex magnetic permeability and dielectric permittivity of MREs with randomly distributed particles (40 vol.%) in both types of matrix have been measured at the room temperature in the frequency range of 1×10^6 – 3×10^9 Hz by the impedance method with an impedance/material analyzer Agilent E4991A (Agilent Technologies, USA). The investigation of dielectric properties was carried out on circular samples with diameter of 15 mm, whereas the measurements of complex magnetic permeability were performed on toroidal samples with outer diameter of 8 mm and inner diameter of 3.1 mm. Specimens for both characterizations were cut out of previously prepared plates (thickness 0.5 mm) by a manual press. In order to estimate the absorbing properties of single-layer metal-backed ESMs based on investigated MREs, the frequency dependence of the reflection coefficient, R , representing the absorbing ability of ESMs in decibels, has been calculated. When the level of R is equal to – 10 dB and the absence of transmitted energy is presumed, then 90 % of absorption of incident energy by ESM is obtained. Given that an electromagnetic wave is incident on the ESM surface along the normal, R from the surface of such a material can be calculated according to [15]:

$$R_L(dB) = 20 \log \left| \frac{Z_{in} - 1}{Z_{in} + 1} \right| \quad (1)$$

, where

$$Z_{in} = \sqrt{\frac{\mu^*}{\varepsilon^*}} \tanh \left(j \frac{\omega}{c} \sqrt{\mu^* \varepsilon^*} d \right) \quad (2)$$

is the input impedance of the ESM, c is the velocity of light, $\omega (= 2 \pi f)$ is the angular frequency, $\mu^* = \mu' - j\mu''$ and $\varepsilon^* = \varepsilon' - j\varepsilon''$ are the complex permeability and permittivity of the material, respectively, and d is the thickness of the sample. The reflection from ESM is absent in case $Z_{in}=1$ condition is met. However, the absence of reflection from the ESM in real materials is reached only approximately, and the frequency, f_0 , and thickness, d_0 , for which the above condition is satisfied with highest degree of accuracy, are called matching frequency and matching thickness, respectively. In practical calculations, the minimum of R is obtained only for complex values of thickness:

$$d = d' + jd'' = \frac{c}{2\pi f \sqrt{\mu^* \varepsilon^*}} \arctan \left(-j \sqrt{\frac{\varepsilon^*}{\mu^*}} \right) \quad (3)$$

Once the dependence of complex parameter d (Eq. 3) on frequency is calculated, the minima satisfying the inequality $|d''/d'| \leq 0.01$ are taken and the thickness $d_0 = d'$ is substituted into Eqs. 1 and 2 [16] yielding the frequency dependence of R .

The MR properties of MREs under investigation were measured using a rotational rheometer Physica MCR502 (Anton Paar GmbH, Austria) with a Physica MRD 170/1T magneto-cell at 25 °C. True magnetic field intensity (0 – 691 kA m⁻¹) was measured using a Hall probe. A parallel-plate measuring system with a diameter of 20 mm and gap of 0.5 mm was used. The small-strain oscillatory tests were carried out through dynamic strain sweeps and frequency sweeps. The strain sweeps were performed in the

applied strain range 0.01 – 1 % at a fixed frequency of 1 Hz in order to get the position of the linear viscoelastic region (LVR). Afterwards, the viscoelastic moduli were obtained from frequency sweep tests (0.1 – 10 Hz) at a fixed strain amplitude in the LVR ($\gamma = 0.1$ % in our experiments).

3. Results and Discussion

In order to prove the coating of magnetic particles with polysiloxane-based polymer, FTIR spectra were examined as shown in Figure 1. The typical FTIR spectrum of pristine CI particles (Fig. 1a) illustrates, that no characteristic bands are observed because of their composition consisting of more than 97.7 % of iron. After the reaction with TEOS, the characteristic peaks of Si–O bonds were observed, which indicate that the siloxane was fabricated on the surface of CI. The band at 1105 cm^{-1} attributed to the (Si–O–C/Si–O–Si) asymmetric stretching appeared. Additionally, the peaks at 796 cm^{-1} and 937 cm^{-1} are assigned to the stretching vibration of Si–OH as matching with the structure of TEOS [14].

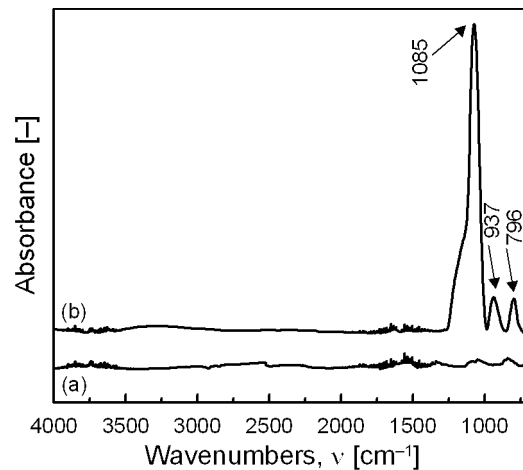


Figure 1. FTIR spectra of pristine CI particles (a), their TEOS-modified analogues (b).

The magnetic properties of the pristine CI particles as well as the TEOS-modified ones are crucially influencing the controllable stiffness of the MREs and also their shielding properties. The dependence of the magnetization saturation on the magnetic field strength can be seen in Fig. 2. The pristine CI particles exhibited magnetization saturation of 196 emu g^{-1} . After the coating of the particles with thin layer of TEOS, the magnetization saturation was negligibly affected and decrease to 194 emu g^{-1} . Such decrease is very promising for the potential application since the magnetic performance of the particles sustain on the same level after the coating procedure.

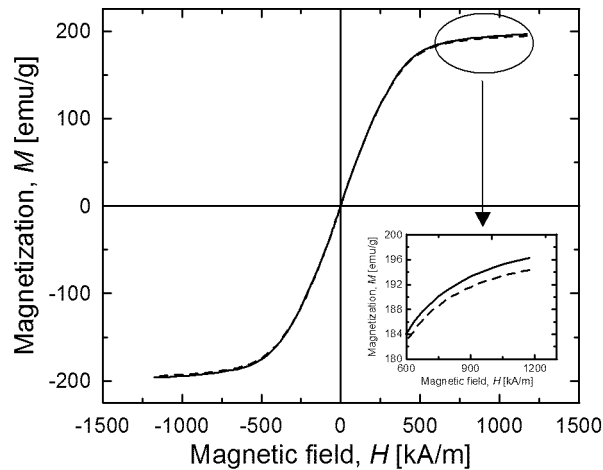


Figure 2. VSM of pristine CI particles (solid) and TEOS-modified analogues (dashed).

For practical applications of MREs it is very important to improve their durability by suppressing the chemical reaction of iron particles due to the diffusion of acidic reactive species coming from *e.g.* acid rains or sea humidity through the matrix material resulting in chemical reduction of iron followed by decreased magnetic parameters such as saturation magnetization or magnetic permeability. The trends of pH values of acid (HCl) dispersions of pristine and TEOS-modified CI particles over time were used to compare the anti-acid-corrosion properties (Fig. 3). Evidently, the surface of pristine CI

particles reacts strongly with HCl which was also visibly accompanied by intensive H₂ bubble generation in the first stage of experiment and a linear increase in pH [17]. On the other hand, only moderate pH increase was observed for TEOS-modified CI particles resulting in the improved anti-acid-corrosion properties for real applications.

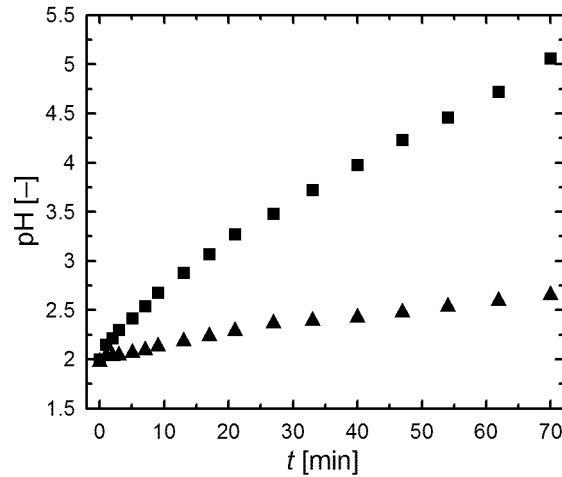


Figure 3. Resistance to corrosion by acids of pristine CI particles (■) and their TEOS-modified CI particles (▲).

Frequency dependences of complex magnetic permeability and dielectric permittivity of MREs are depicted in Fig. 4 and Fig. 5, respectively.

It is worth to mention that the values of magnetic permeability and dielectric permittivity of MREs based on thermoplastic elastomer are smaller than those of MREs based on silicone elastomer in the entire frequency range though the filler concentration is the same for both systems. This can most probably be attributed to the difference in the processing conditions of composites. Compared to MREs based on silicone matrix which were processed at 40 °C, the thermoplastic MREs were manufactured at elevated temperature (170 °C) which could have led to the thermal oxidation of CI particles and to consequent deterioration of electromagnetic properties. Nevertheless the difference

may also stem from altered microstructure of composite material with one matrix having higher affinity towards the dispersed particles than the other which can lead to bigger clusters.

As it can be seen from Fig. 4a, the deposition of TEOS layer on the surface of CI particles has small influence on the magnetic spectra of MRE based on silicone elastomer. It can only be mentioned that in the whole frequency range, TEOS coating slightly decreases the magnetic permeability compared to the MRE based on pristine CI particles (Fig. 4a).

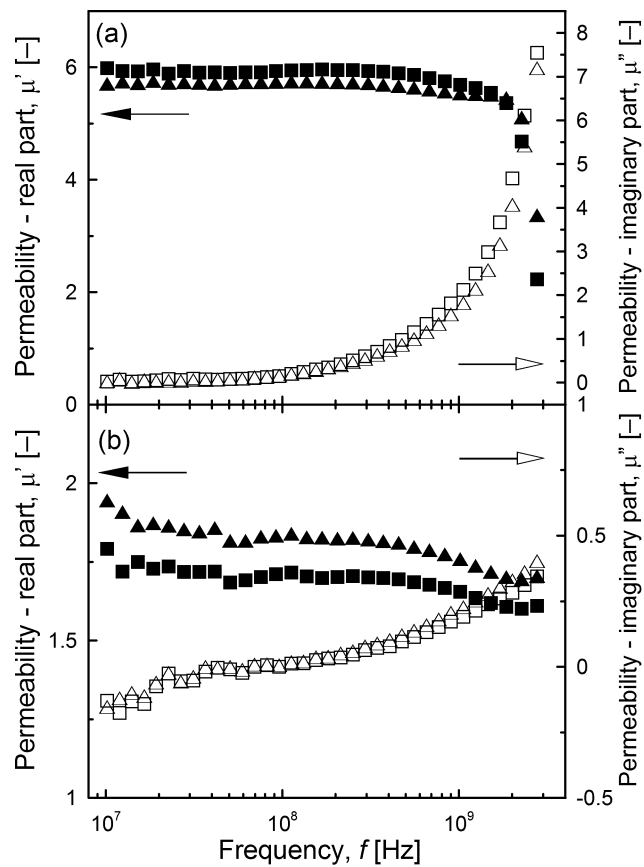


Figure 4. Frequency, f , dependence of the real, μ' , (solid) and imaginary, μ'' , (open) part of the magnetic permeability for MRE based on pristine CI particles (■, □), and TEOS-modified analogues (▲, △) dispersed in silicone (a) and thermoplastic matrix (b).

Such negligible decrease in permeability indicates that the thickness of TEOS interlayer separating CI particles is sufficiently small not to make a disorder at filler–matrix interface and, thus, to cut-off the inter-particle magnetic interaction. In contrast to the trend observed in Fig. 4a, the magnetic permeability of MRE based on thermoplastic elastomer matrix is slightly higher in case of TEOS-modified CI particles (Fig. 4b) which can be the evidence that TEOS over layer inhibits the thermal oxidation of CI at higher processing temperatures. The similar trend is also traceable in case of dielectric properties where the relative permittivity of MRE based on TEOS-modified CI particles is slightly higher than that of MRE based on pristine CI particles (Fig. 5b).

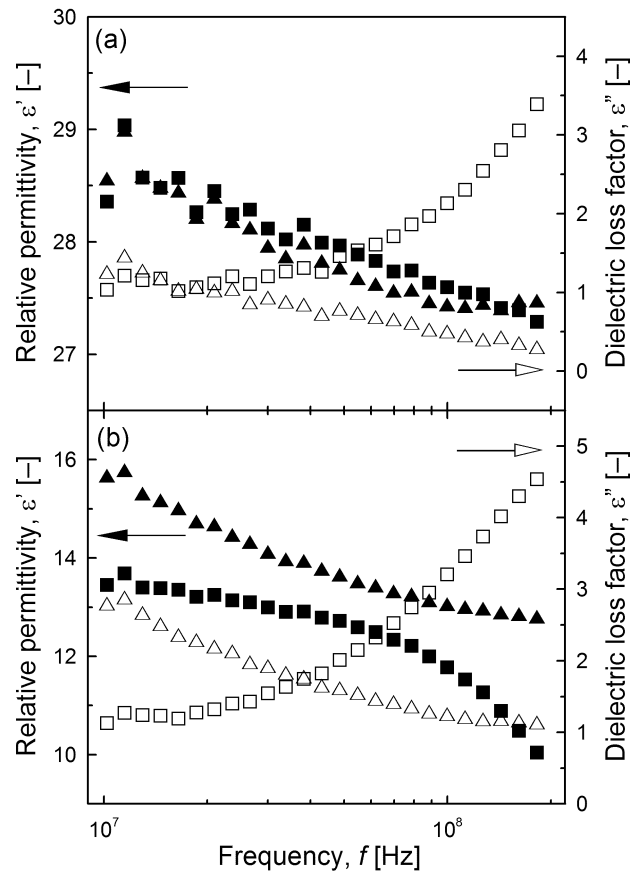


Figure 5. Frequency, f , dependence of the relative permittivity, ϵ' , (solid) and dielectric loss factor, ϵ'' , (open) for MRE based on pristine CI particles (■,□), and TEOS-

modified CI particles ($\blacktriangle, \triangle$) randomly dispersed in silicone elastomer (a) and thermoplastic elastomer (b).

The measured electromagnetic spectra of MREs were used to calculate the frequency characteristics of R utilizing the procedure described in section 2.5. The results of the calculations are presented in Fig. 6 and Table 1. As can be seen from Fig. 6, R has deep minima R_0 at matching frequencies f_0 for all ESMs. The bandwidth properties of individual ESMs are estimated from the values of the ratio f_{\max}/f_{\min} , where f_{\max} and f_{\min} correspond to the edge frequencies of the operating frequency band taken for the reflection level of -10 dB.

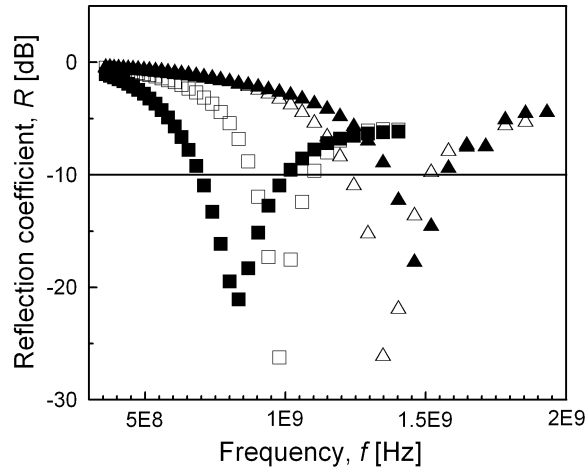


Figure 6. Frequency, f , dependence of the reflection coefficient, R , for the MRE based on pristine CI particles (\blacksquare) and TEOS-modified CI particles (\square) in silicone elastomer, and pristine CI particles (\blacktriangle) and TEOS-modified CI particles (\triangle) in thermoplastic elastomer.

As can be seen from Fig. 6 and Table 1, all ESMs exhibit good absorbing ability at ultra-high frequencies with bandwidths ranging from 1.15 to 1.44. Estimated thicknesses of individual ESMs tabulated in Table 1 represent the minimum required

thickness which will provide maximum possible absorption of incident electromagnetic wave by the respective ESM at a matching frequency. The electromagnetic shielding materials based on TEOS-modified particles have different operating frequency ranges comparing to ESMs based on pristine CI particles. Thus it is possible through the particles surface modification and thereby affecting the frequency characteristics of permeability and permittivity of MREs to tune the operating frequency range of ESMs making possible their application in the frequency range from nearly 700 MHz to 1.6 GHz in which many communication and information transmission systems operate. Hence, it is evidently presented here for the first time in the field of MREs that these systems can be used also for sufficient electromagnetic shielding.

Table 1. Electrodynamic characteristics of ESMs based on the different MREs.

Polymer matrix	Filler modification	f_{\min} [MHz]	f_0 [MHz]	f_{\max} [MHz]	R_0 [dB]	d_0 [mm]	f_{\max}/f_{\min}
Silicone elastomer	Pristine CI	691	825	998	-21	7.0	1.44
	TEOS-modified CI	881	977	1100	-26	5.0	1.25
Thermoplastic elastomer	Pristine CI	1370	1460	1570	-26	9.8	1.15
	TEOS-modified CI	1220	1350	1510	-18	9.6	1.24

A novel possible application of MREs with improved oxidation resistance as an electromagnetic shielding material has been discussed up to now. The external magnetic field-responsive properties represented by controllable stiffness development as a typical phenomenon in MREs will be studied further. In general, the mechanical properties of materials depend on frequency of deformation. A good understanding of the influence of frequency on a material is therefore very important for its practical use. An oscillatory test for MREs should be performed within the LVR that the magnetized structure of the system is being maintained.

To ensure validity of the oscillatory test, the amplitude sweep test was always carried out first in our case. After the LVR has been defined by a strain sweep for each sample separately, its structure was further characterized using a frequency sweep at a strain below the critical strain of LVR. This provides more information about the interactions among particles and matrix.

The rheological response of MREs under investigation under frequency sweep test is shown in Figures 7 and 8 for silicone and thermoplastic elastomer matrix, respectively. Evidently, the storage modulus, G' , characterizing the elastic component of MRE, is nearly independent of frequency, as would be expected from a structured or cured gels. The, G' , is almost identical for pristine CI particles randomly dispersed in both types of matrix. However, the modification of magnetic particles via TEOS has an impact on their improved compatibility with both matrixes. The extreme improvement is in the case of silicone matrix where the, G' , increased almost in one order of magnitude probably due to the chemical bonding of TEOS coating with siloxane group of the matrix (Fig. 7). The loss modulus, G'' , characterizing the viscous component of MRE, changes comparatively with, G' , in almost all conditions applied. This trend is different

only for TEOS-modified CI particles dispersed in silicone matrix and under the application of high external magnetic fields (\circ and \triangleright symbols in Fig. 7b). The interpretation of, G'' , is complicated here since it linearly decreases in log-log coordinates with increasing frequency. This can be caused due to the internal movement of magnetized particles bonded to the matrix and such relative movement of particles can create some friction between neighboring particles [17].

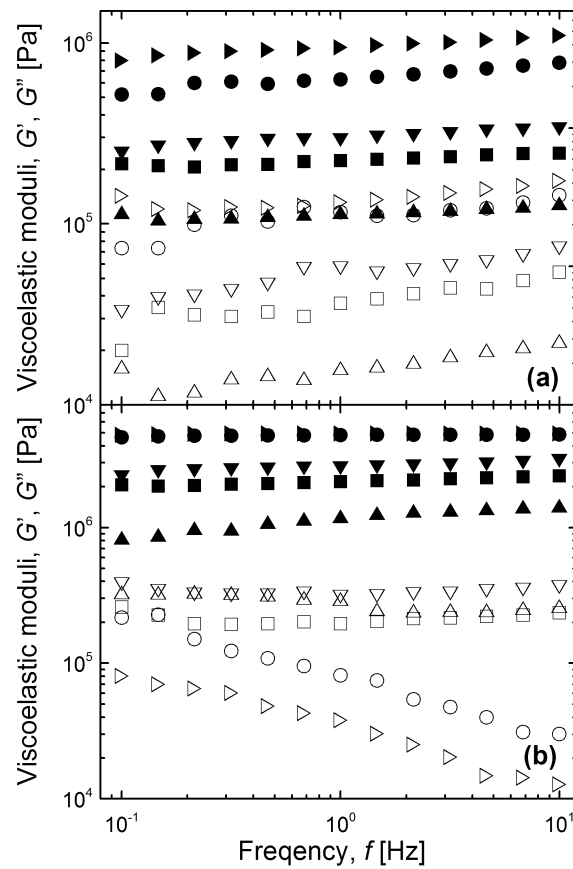


Figure 7. Storage, G' , (solid) and loss, G'' , (open) moduli as a function of frequency, f , for MRE based on pristine CI particles (a), and TEOS-modified CI particles (b) randomly dispersed in silicone elastomer matrix at various magnetic field intensity, H , (kA/m): 0 ($\blacktriangle, \triangle$), 171 (\blacksquare, \square), 343 ($\blacktriangledown, \triangledown$), 464 (\bullet, \circ), 691 ($\blacktriangleright, \triangleright$).

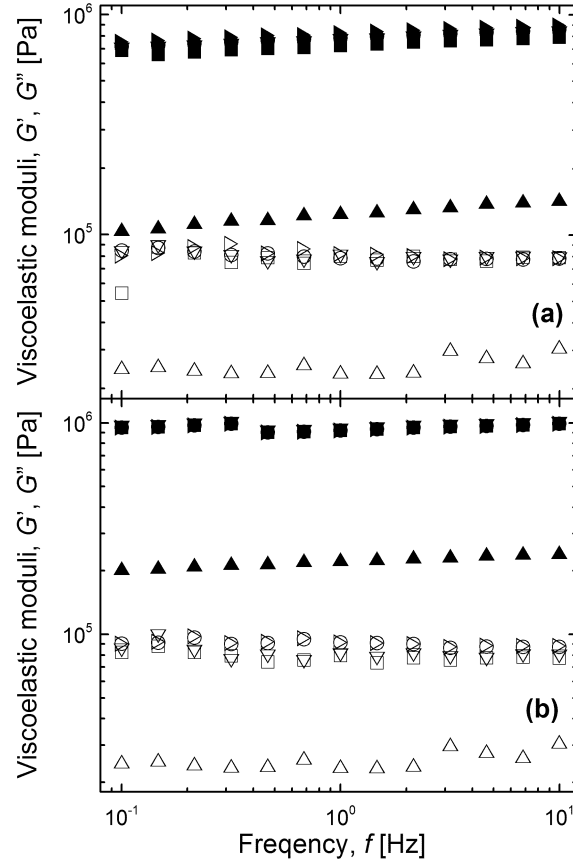


Figure 8. Storage, G' , (solid) and loss, G'' , (open) moduli as a function of frequency, f , for MRE based on pristine CI particles (a), and TEOS-modified CI particles (b) randomly dispersed in thermoplastic elastomer matrix at various magnetic field intensity, H , (kA/m): 0 ($\blacktriangle, \triangle$), 171 (\blacksquare, \square), 343 ($\blacktriangledown, \triangledown$), 464 (\bullet, \circ), 691 ($\blacktriangleright, \triangleright$).

4. Conclusion

In this study, a detailed experimental study on the oxidation stability, electromagnetic shielding, and viscoelastic properties under an external magnetic field of MREs based on randomly dispersed pristine CI or TEOS-modified CI particles was presented. Magnetorheological elastomers have been further prepared using two different kinds of elastomeric matrix, *i.e.* silicone and thermoplastic. The volume fraction of MREs samples has been fixed for all samples, which can help to study the

influence of different magnetic particles surface layer and matrix type on above mentioned properties. From the magnetization measurements, it has been clearly observed that the basic magnetic parameters of magnetic particles are not changed significantly after their modification via TEOS while their anti-acid-corrosion properties necessary for real applications are definitely improved. The TEOS modification of CI particles further brings undeniable benefits in their improved compatibility resulting in better distribution of modified particles with both matrixes, namely silicone one in which even bonding of particles with matrix molecules could occurred. From application point of view, the better distribution of TEOS-modified particles resulted in increased viscoelastic performance under the external magnetic field applied again namely for silicone matrix based system. Moreover, the modification of particles led to slightly tuning of operating frequency range of ESMs, thereby allowing to extend their application at ultra-high frequencies.

Acknowledgments

The author M. S. would like to thank the Grant Agency of the Czech Republic (14-32114P) for financial support. This article was written with support of the Operational Program Research and Development for Innovations co-funded by the European Regional Development Fund (ERDF) and the national budget of the Czech Republic, within the framework of the project Centre of Polymer Systems (reg. number: CZ.1.05/2.1.00/03.0111).

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