Dynamic magneto-mechanical properties of magneto-sensitive elastomers determined using a new experimental test involving forced longitudinal vibration

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ABSTRACT
A new experimental method involving forced longitudinal vibration is presented for experimentally determining the dynamic magnetomechanical properties of a magneto-sensitive elastomer in a magnetic field. A cylindrical sample is attached to a vibration platform and placed in a longitudinal magnetic field generated by a solenoid electromagnet, and a resonant technique is used to obtain the dynamic magnetomechanical properties of the tested sample. The results indicate that the resonant frequency (i) increases with the intensity of the applied magnetic field and the content of the magnetic-particle filler in the matrix but (ii) decreases with the sample length. The dynamic properties of the storage and loss moduli depend significantly on the excitation frequency and the magnetic-particle content. The testing process shows that it is simple and easy to evaluate the dynamic properties using forced longitudinal vibration, with the additional advantage of it being a nondestructive technique. This method could be extended to characterize the coupled magnetomechanical behavior of magneto-sensitive functional elastomers such as Terfenol-D/epoxy composites.

I. INTRODUCTION
Combining the elasticity of polymers and the magnetic properties of ferromagnetic particles in a single composite material has recently promoted the emergence and development of a new type of smart material that can respond to an external magnetic field by switching its physical properties rapidly. In particular, significant changes in the mechanical, dielectric, magnetic, and other characteristics of these composites have been reported since they were first discovered at the end of the 20th century. Because of their predominant magnetically controlled mechanical properties, these materials are often referred to as magneto-sensitive elastomers (MSEs). Their outstanding characteristics give MSEs attractive developmental potential and broad application prospect in many fields, such as intelligent actuation and sensing, active shock absorption and noise reduction, bionic structures, and other modern technology. Because of the inherent characteristics of the polymeric materials that are used as supporting materials, including natural rubber, silicone rubber, polybutadiene, polyisobutylene, and thermoplastic elastomers, this type of smart material often exhibits a certain degree of viscoelasticity. In addition, the optical, electrical, magnetic, and mechanical properties of these polymer-based magnetic smart materials can be tuned by means of temperature, frequency, and prestress, among others. Therefore, knowledge about the magnetomechanical coupling characteristics and behavior under different deformation modes, as functions of the magnetic field and excitation frequency, is essential for modeling the material,
predicting its performance, and using such elastomers rationally and properly.

Over the past few years, there have been many different approaches to measuring MSEs in conventional setups with the addition of an external magnetic field. For example, by using a universal testing machine with a special magnetic coil system, the quasistatic compressive, tensile, and shear performances (and their combinations) of MSEs were assessed in different magnetic fields. In addition, by using a commercial plate–plate magnetorheometer with equipment to produce a magnetic field, many researchers have measured the oscillatory shear properties of MSEs and how they react to a magnetic field. However, oscillatory shear rheometry with disc-shaped elastomer specimens is susceptible to the boundary conditions of clamping, the normal pressure, and the sample dimensions. As such, the aforementioned evaluation methods can only be used for MSEs with small strain or at relatively low frequencies. Relying on a modified split Hopkinson pressure bar, Liao et al. characterized the dynamic stress–strain curves as a function of strain rate in the range of $10^2$–$10^4$ s$^{-1}$. It can be seen that the above-mentioned techniques can achieve the performance in certain aspects of the tested material.

Meanwhile, researchers have explored some innovative characterization methods to study such materials comprehensively and in detail. Based on the photoacoustic technique, Macias et al. proposed a reliable method for determining the mechanical properties of magnetoelastic membranes; however, it is usually more complicated to identify mechanical parameters from photoacoustic signals. Using a commercial nanoindenter, which is a novel and effective experimental technique, Wang et al. investigated the quasistatic and dynamic properties of magnetic-particle-reinforced soft composites in the absence of a magnetic field.

As an extension of the existing research methods, the use of forced longitudinal vibration can be considered to access a wider range of material performance (e.g., Young’s modulus and resonant frequency) at higher-frequency excitation. In recent years, the technique of forced longitudinal vibration introduced by Norris and Young has been widely used to measure dynamically the Young’s modulus of viscoelastic materials. This involves exciting a bar-shaped sample harmonically at one end and measuring the ratio of the end amplitudes, and the response of the sample at resonance can be used to obtain the complex modulus. This approach has been used extensively to characterize numerous materials. For example, by improving the experimental apparatus, Madigosky et al. characterized the dynamic viscoelastic properties of various materials as a function of temperature. Furthermore, Willis et al. and Guillot and Trivett used the technique extensively to examine a number of materials as a function of both temperature and hydrostatic pressure; their research showed that the resonant bar technique is relatively easy to perform and has the advantage of high signal-to-noise ratio (SNR). The error analysis performed in Ref. shows that when using this method, the total measurement uncertainty is $1.6\%$ for both the storage modulus and the loss modulus.

In the present study, a new experimental test with the simple method was conducted to determine the complex Young’s modulus of MSEs in different magnetic-field environments. This test could be extended to measure the magnetomechanical coupling performance of other magnetoelastic functional materials. This paper is organized as follows. In Sec. II, the theoretical basis and principle of the testing method are introduced. Based on the main idea of the longitudinal vibration technique, the testing device and the operating method for testing the vibration properties of MSEs are presented systematically in Sec. III. In Sec. IV, the feasibility of the experimental apparatus is verified, followed by a detailed discussion of how the intensity of the applied magnetic field influences the magnetomechanical coupling performance. Finally, conclusions are drawn in Sec. V.

II. THEORETICAL FRAMEWORK

The theoretical background of the longitudinal vibration method used in the present study can be found in several articles. However, because none of those references describes the situation with a magnetic field, we present below the most important theoretical points of the measurement.

First, many research results have indicated that a composite filled randomly with ferromagnetic particles is equivalent to a magnetic material with continuous and isotropic characteristics. The macroscopic mechanical properties are related only to the external applied magnetic field and the particle volume fraction. As such, we consider a cylindrical sample of density $\rho$, length $L$, and constant cross section, which is attached to a vibration exciter at one end and is free at the other end. The sample is then exposed to a magnetic field applied parallel to the cylinder axis denoted by $z$, as sketched in Fig. 1.

Assuming that the $z$-axis excitation displacement is sinusoidal, the relationship between time and displacement can be written as $w_0 = W_0 e^{i\omega t}$ and the resulting axial displacement (relative to the driven end) at a distance $z$ from the driven end is $w(z, t) = W(z) e^{i\omega t}$.
Neglecting the effects of lateral inertia, the equation of motion is obtained as follows:\(^\text{(1)}\):

\[
\frac{\partial \sigma_z}{\partial z} = \rho \frac{\partial^2 w}{\partial t^2} (w + w_0),
\]

where \(\sigma_z\) is the uniaxial stress in the bar in the longitudinal direction (i.e., the z axis). Here, we assume that the mechanical behavior of the polymer matrix composite material satisfies Hooke’s law \(\sigma_z = E' \varepsilon_z\), where \(E'\) is the complex Young’s modulus of the MSE measured in the presence of a magnetic field and \(\varepsilon_z\) is the axial strain experienced by the material, defined by \(\varepsilon_z \equiv \partial w/\partial z\). Note that the Young’s modulus of the sample is associated with the applied magnetic field and by the material, defined by \(\sigma = \rho \varepsilon\).

The boundary conditions for the problem are zero relative displacement at \(z = 0\) [i.e., \(w(0, t) = 0\)] and zero stress at \(z = L\) [i.e., \(\sigma_z (L, t) = 0\)], which lead to the following solution for Eq. (2):

\[
W(z) = W_0 [\cos(kz) + \tan(kL) \sin(kz) - 1],
\]

where \(k\) is the complex wave number defined as \(k \equiv \omega(\rho/E')^{1/2}\). Having solved Eq. (2), the sample is subjected to resonant analysis. From Eq. (3), the complex ratio \(Q^*\) of the free-end displacement to the driven-end displacement is

\[
Q^* = \frac{W(L)}{W_0} = \frac{1}{\cos(kL)}. \tag{4}
\]

Meanwhile, using complex notation, the Young’s modulus of a viscoelastic material can be written as \(E' = E' + iE'' = E[\cos \delta + i \sin \delta]\), where \(E'\) is the magnetic-field-dependent storage modulus, \(E''\) is the corresponding loss modulus, \(E\) is the magnitude of the Young’s modulus, and \(\tan \delta\) is the loss factor. Then, Eq. (4) can be rewritten as

\[
\frac{1}{Q^*} = \cos \left[ \omega L \left( \frac{\rho E}{E'} \right)^{1/2} \cos \left( \frac{\delta}{2} \right) \right] \cos \left[ \omega L \left( \frac{\rho E}{E'} \right)^{1/2} \sin \left( \frac{\delta}{2} \right) \right] \tag{5}
\]

\[
+ i \sin \left[ \omega L \left( \frac{\rho E}{E'} \right)^{1/2} \cos \left( \frac{\delta}{2} \right) \right] \sin \left[ \omega L \left( \frac{\rho E}{E'} \right)^{1/2} \sin \left( \frac{\delta}{2} \right) \right].
\]

If the complex ratio is written as \(Q^* = Q_0^\theta\), then at resonance, when \(\theta = (2n - 3)(\pi/2)\) and \(n = 1, 2, 3, \ldots\) is the resonance number, we have

\[
E = \rho \left[ \frac{4f_{\text{res}}}{(2n - 1)} \cos \left( \frac{\delta}{2} \right) \right]^2, \tag{6}
\]

\[
\delta = 2 \tan^{-1} \left[ \frac{\sin^{-1} (1/Q_0)}{(2n - 1)^{1/2}} \right]. \tag{7}
\]

Note that Eqs. (6) and (7) are valid at resonance only and that \(f_{\text{res}}\) is the resonance frequency and \(Q_0\) is the amplitude of the displacement ratio. As the above analysis shows, measuring \(f_{\text{res}}\) and \(Q_0\) at resonance in different magnetic fields and incorporating Eqs. (6) and (7) provide a direct method for determining the complex Young’s modulus of an MSE. Once the modulus amplitude and the loss factor are known, the elastic and loss moduli can be computed using

\[
E' = E \cos \delta, \quad E'' = E \sin \delta. \tag{8}
\]

III. EXPERIMENTAL DETAILS

A. Sample preparation

In this work, spherical carbonyl iron particles of reactive deposition epitaxy type (Jiangsu Tianyi Ultra-fine Metal Powder Co., Ltd., China) with a mean size of 5.89 μm were chosen as the filler and commercial two-component Essil-296 silicone (Axson Technologies Shanghai Co., Ltd., China) was chosen as the host matrix material. Essil-296 is a flexible silicone organic polymer synthesized using vinyl methyl polysiloxane as the base compound (0.22% vinyl groups) and methyl hydrogen polysiloxane as the curing agent (1.02% Si–H groups). The structures of the two components are shown in Figs. 2(a) and 2(b), and the corresponding viscosities being 85,000 cp and 2500 cp, respectively. Beijing Hagibis Technology Co., Ltd., China supplied silicone oil with a viscosity of 500 cp, and its structure is shown in Fig. 2(c).

The details of how the MSEs were fabricated are as follows. First, the silicone rubber mixing components of the base compound and the curing agent (15:1 by weight), the silicone oil of appropriate proportion, and the desired ratio of filler magnetic particles were mixed in a beaker and stirred adequately at room temperature to ensure an even particle dispersion. The resulting mixture was then degassed for 30 min in a vacuum oven to remove trapped air pockets so that the composite was achieved in a dense state. The
compound was then poured into a cylindrical organic glass tube with an inner diameter of 10 mm and a length of 30 mm, 40 mm, or 50 mm and cured at 60 °C for 2 h. The prepared physical samples are shown in Fig. 3(a). In this study, composite samples were prepared containing carbonyl iron particles in different concentrations by weight, namely, 70% or 75%. The compositions of the synthesized MSEs are given in Table I. The completed samples were approximately isotropic composites with a ferromagnetic particle filler, and the cross-sectional scanning electron microscope image in Fig. 3(b) shows that these particles were dispersed randomly in the elastomer matrix.

### B. Experimental apparatus

This section describes the experimental apparatus used for the dynamic test to determine the magnetomechanical properties of magnetosensitive functional materials. Based on the test principle, the corresponding equipment shown in Fig. 4 was constructed. It has three main parts, namely, (i) the vibration excitation device, (ii) the magnetic-field generator, and (iii) the vibration detection apparatus.

In the first part, a cylindrical sample is glued at one end to the platform, which is connected to a vibration exciter using a 30-cm-long vibration-transmitting aluminum rod. The exciter is controlled by a signal generator (model DG1022U; Beijing RIGOL Technology Co., Ltd., China) and a power amplifier (model GF-500).

To investigate the dynamic mechanical properties of an MSE under different external magnetic fields, the system was equipped with a customized magnetic device (Fig. 4). The magnetic-field generator comprises an electromagnet (solenoid) and a DC regulated power supply. The size of the solenoid magnet and its position relative to the sample are shown in Fig. 1. The height of the electromagnet is 17 cm, the diameter of the central air gap is 6 cm, and the distance h from the top of the platform to the bottom of the magnet is 6 cm. By controlling the current applied to the electromagnetic coil, the strength of the magnetic field used in the test can be adjusted expediently.

For a given carrying current, the distribution of the magnetic field in the solenoid was characterized as shown in Fig. 5 by means of an experimental test and the COMSOL finite-element software. Figure 5(a) shows the magnetic flux density in the axial direction inside the solenoid. The simulation and experimental results agree well with each other, with the magnetic field inside the solenoid increasing at first from bottom to top and then decreasing along the axis. Furthermore, the distribution of magnetic flux density in the longitudinal section of the air gap is shown in Fig. 5(b) for a carrying current of 10 A. Both the axial and transverse distributions of the field in the central region of the magnet are nearly uniform, thereby allowing the magnetic field to be considered as being homogeneous. For a carrying current of 10 A, 20 A, and 30 A, the corresponding average magnetic field is 41 mT, 83 mT, and 123 mT, respectively.

A laser Doppler vibrometer (LDV) (PSV-400-B; Polytec), which can measure out-of-plane (normal) vibrations, was used to record the amplitude and phase of the output signals. With a maximum frequency of 250 kHz and speed options of 10/100/1000 mm s$^{-1}$ V$^{-1}$, the LDV comprises a scanning head, a junction box, and control subsystems. In the test, the LDV scanning head was used to measure the vibration velocities of the platform and the free end of the sample simultaneously. The response curves of the vibration rate vs the frequency were obtained using the fast Fourier transform incorporated in the software PSV 8.7.

### C. Measurement procedure

Based on the theory described in Sec. II, the resonance measurements were made as follows. In frequency sweep mode, the signal

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**TABLE I. Composition ratios of cylindrical MSEs.**

| Sample | Silicone rubber (wt. %) | Silicone oil (wt. %) | Iron powder (wt. %) |
|--------|-------------------------|---------------------|-------------------|
| 1      | 22.5                    | 7.5                 | 70                |
| 2      | 18.75                   | 6.25                | 75                |
generator excited the vibration exciter with a continuous sine wave whose frequency ranged from a lower limit to an upper limit and the scanning head recorded the amplitude and phase of the LDV output signals. During a test, one measuring point was located at the center of the free end of the sample (\( z = L \)) and another was located at the excitation end of the sample (\( z = 0 \)). Because the sample surface was black and did not scatter enough light, the following approach was adopted. To measure the motion of the top end of the sample, a small piece of metallic tape (smaller than the cross-sectional area) was glued to the end of the sample; this small piece of tape did not constrain the longitudinal motion, and its mass was negligible compared to that of the sample. To reduce the energy leakage caused by truncating the signal, the vibration signal was processed with a Hamming window in the experiment, and the random error was reduced further by sampling each measuring point 10 times separately. A VD-04 controller was used to control the LDV (the maximum measuring frequency was 250 kHz, and the maximum measuring speed was 100 mm s\(^{-1}\) V\(^{-1}\)). From the testing process, the dynamic properties of the samples were tested using axial resonance, which is nondestructive and simple and easy to carry out.

IV. RESULTS AND DISCUSSION

First, to ensure that the established experimental apparatus was not influenced by the external magnetic field applied during the testing process, as a reference sample that was insensitive to the magnetic field, a polycarbonate rod with a diameter of 5 mm and a length of 50 mm was glued to the vibration platform with YH-840 glue and tested in the range of 0–2 kHz. Figure 6 shows the relationship between the end-displacement ratio and the excitation frequency of the reference sample in different magnetic environments. As this figure shows, the test results for the polycarbonate rod are similar to those of a previous study and are reproducible in different conditions.
magnetic environments. From this, we infer that the experimental device was free from the influence of the external magnetic field in the testing process.

Subsequently, the test sample was scanned in a wide frequency range to obtain its approximate resonant frequency. As Fig. 7 shows, the MSE with a mass fraction of 70% and a length of 50 mm was tested repeatedly in the range of 0–2 kHz and the maximum end-amplitude ratio occurred at an excitation frequency of around 106 Hz. In addition, the results of each test were reproducible. To determine the resonant frequency of the sample more accurately and reduce the swept frequency range, the latter was set to 60–400 Hz with a sampling time of 8 s and a sampling frequency of 1.024 kHz, thereby giving a sampling accuracy of 0.125 Hz. Representative curves of the vibration rate at the top of the sample, that of the vibration platform, and the end-amplitude ratio varying with the excitation frequency are shown in Fig. 8. The measurements made using this method have high SNR because of an obvious peak value at the resonant frequency.

Upon applying a magnetic field, this type of magnetosensitive material exhibits significant field-controlled performance. The mechanism responsible for the bulk effect is the induced magnetic interaction of particles within the matrix. Figure 9 shows the end-amplitude ratio vs the excitation frequency for the sample with a mass fraction of 70% and a length of 30 mm in different magnetic fields.

The resonant frequencies of column-shaped MSEs dependence on length and applied magnetic field are shown in Fig. 10. The open symbol denotes the sample with a particle mass fraction of 70%, while the solid symbol represents a particle mass fraction of 75%.

The complex Young’s modulus of MSEs with different length dependence on excitation frequency and applied magnetic field is shown in Fig. 11. The open symbol denotes the sample with a particle mass fraction of 70% and a length of 30 mm in different magnetic fields.
The vibration response of the sample is similar for each magnetic field: the end-amplitude ratio first increases with the excitation frequency and then decreases, and the maximum value occurs at the resonant frequency. In addition, the resonant frequency changes significantly as the magnetic field strength is increased, whereas no variation of the end-amplitude ratio is obvious.

From how the end-amplitude ratio depends on the excitation frequency for different MSEs in different environments, the corresponding resonant frequency can be obtained. Figure 10 plots the resonant frequency of each material varying with the sample length and the applied magnetic field. The resonance frequency is related to not only the inherent properties of the test sample (e.g., composition and geometric size) but also the external applied magnetic field. For sample rods with the same length, the more the content of iron powder, the higher the resonant frequency of the material. This is because the quality and modulus of the composite material increase with the amount of inclusion. Next, the resonant frequency of the column-shaped samples decreases with their length. Therefore, the intrinsic properties of the material (e.g., internal components and geometry) play a decisive role in determining its natural frequency. Figure 10 also shows that the natural frequency of the column-shaped specimens increases obviously with the strength of the applied magnetic field. The reason for this reinforcing effect is mainly the increased elastic modulus of the MSEs, namely, the magnetoinduced modulus, which is attributed to the interaction among the micrometer-sized magnetically permeable particles within the magnetic field. 27,29

After obtaining the resonant frequency of the sample and the corresponding end-amplitude ratio, the complex Young’s modulus and the loss factor of the measured material are obtained by combining the geometric properties with Eqs. (6) and (7). Figure 11 shows that the complex Young’s modulus of MSEs with different lengths but the same amount of inclusion changes with the magnetic field and the vibration frequency. Clearly, the complex modulus increases significantly with the magnetic field intensity and the excitation frequency, showing excellent correlation.

Furthermore, mechanical properties such as the storage and loss moduli of MSEs with different particle mass fractions can be evaluated using Eq. (8). The storage and loss moduli of the prepared samples vs the excitation frequency under different magnetic field strengths are shown in Fig. 12. The dynamic mechanical performance of the composites increases with the excitation frequency at a fixed magnetic field strength, and the trend is consistent with results obtained using other characterization methods at low frequencies. 25,26 This may be attributed to the speed mismatch between the slower movement of particle chain and the rapid excitation force applied to the cylinder. 30 With increasing frequency, the dynamic response time decreases because of the freezing of the internal structure of the elastomer, thereby improving the dynamic mechanical performance. In addition, the storage and loss moduli of the composites increase with the applied magnetic field intensity at a fixed oscillation frequency. This behavior is well known and is attributed to the particle–particle and particle–rubber interactions. 2,31 When the sample is exposed to a magnetic field, an additional force arises among the magnetized particles inside the matrix, which causes them to close together to form a more regular structure with the lubrication help of the silicone oil. In turn, this results in better mechanical properties. Besides, the improvement in dynamic mechanical performance is more obvious at higher particle concentration, which is because there is more force on the load cell when the magnetic field is applied. Another factor could be the increased energy absorbed by interfacial friction caused by the increased interfacial area due to the increased particle content. The values of the magnetomechanical properties determined from the resonant frequencies of each sample agree well among them and are consistent with those reported in Refs. 21 and 22 within a deviation of approximately 8% or smaller.

V. CONCLUSIONS

This paper presents the results of an experimental study to determine the dynamic mechanical properties of MSEs using a newly developed nondestructive evaluation system, namely, the method of forced longitudinal vibration. Cylindrical specimens of MSEs with different concentrations of ferromagnetic particles were fabricated and tested in a magnetic field. The experimental results show that the resonant frequency decreases with the sample length but increases with the intensity of the applied magnetic field and the
mass fraction of the magnetic filler. The storage and loss moduli of the MSEs increase significantly with the resonant frequency and the intensity of the applied magnetic field. As indicated, the proposed method is a simple and effective way to characterize the magnetomechanical coupling properties of this type of magnetosensitive functional elastomer.

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