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Effect of Cyclic Deformation on Magnetorheological Elastomers

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Fatigue properties of magnetorheological elastomer (MRE) samples were investigated based on cis-polybutadiene rubber by using a fatigue test machine. Three MRE samples with iron particles mass fraction of 60%, 70%, and 80% were fabricated, and their properties dependence of three strain amplitudes (50%, 75%, and 100%) were measured. The absolute magnetorheological (MR) effect, storage modulus, and loss modulus of MRE samples after fatigue were evaluated by a modified dynamic mechanical analyzer. The results revealed that MR effect, storage modulus, and loss modulus of MREs containing 80% iron particles depended strongly on the strain amplitudes and the number of cycles, while storage modulus and loss modulus of MREs containing 70% iron particles also depended on the strain amplitudes and the number of cycles but not as strongly as sample which contains 80% iron particles, but the properties of MREs containing 60% iron particles after cyclic deformation were almost independent of the fatigued conditions. In order to investigate the fatigue mechanism of MREs, the sample was carried out with a quasi-static tensile testing and its surface morphology during testing was observed \textit{in situ} by scanning electron microscopy.

Key words: Magnetorheological elastomer, Fatigue, cis-polybutadiene rubber, \textit{In situ} observation

I. INTRODUCTION

Magnetorheological (MR) materials are a kind of smart materials whose rheological properties can be controlled by an applied magnetic field. The most common MR materials are MR fluid (MRF) whose yield stress and apparent viscosity can be changed from Newtonian liquid to non-Newtonian liquid when a magnetic field applied on MRF [1, 2]. Magnetorheological elastomers (MREs) are a new branch of MR materials. Their mechanical properties, including modulus and damping capability, can be controlled by an external magnetic field. MREs are composed of magnetizable particles (iron particles) and soft rubber-like matrix [3–6]. MREs can be categorized as isotropic MREs and anisotropic (structured) MREs depending on different curing conditions. The structured MREs are fabricated under an external field during the curing process. The MR effect of the structured MREs is bigger than that of isotropic MREs under the same density of magnetic field [7–9].

MREs are promising for many applications, including adaptive tuned vibration absorbers, stiffness tun-

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were 60%, 70%, and 80%, respectively. The fabrication process of MREs was as follows: the iron particles and other additives were firstly mixed into the rubber in a double-roll mill. The mixture was pulled into a mold and sent for pre-structured under a magnetic field of 150 mT at 160 °C for 30 min, then the pre-structured sample was cured at 160 °C for 20 min under the pressure (152 kPa). The length of MREs was approximately 6 cm along the direction of magnetic field. The magnetic field of the pre-structured process was provided by a permanent magnet.

Flexing fatigue tester (JC-1008, Jiangdu Jingcheng test instruments factory) was applied to measure the fatigue properties of MREs after cyclic deformation. The samples were tested by cycle-tensile loading with three different kinds of strain amplitudes (50%, 75%, and 100% of length of the samples) at a frequency of 300 r/min which is carried out according to Chinese national standard (GB 1688-86), respectively. All the samples were prepared as dumbbell test pieces according to Chinese national standard (GB 528-82). Their size is 6 mm wide and 5.5 mm thick in the central section. Every sample was fatigued for three times at the same condition. The results were then averaged and reported here. The MR effect of MREs was evaluated by a modified dynamic mechanic analyzer [5]. The samples were analyzed at the room temperature with a frequency of 10 Hz and a strain of 0.6% under a range of applied field from 0 to 800 mT. The size of sample is about 5 mm in length, 5 mm in width, and 3 mm in thickness.

To observe MRE’s surface morphology at dynamic conditions, the sample was fixed in a quasi-static tensile setting which is placed in scanning electron microscopy (SEM). Then the sample was applied a tensile loading while its surface morphology was observed in situ by SEM (XL30 ESEM-TMP). The speed of tensile was 1 mm/min and the strain amplitude was 0−400%. The morphology of samples after fatigue was also viewed by the SEM.

**III. RESULTS AND DISCUSSION**

The properties of samples after cyclic deformation depend on the number of cycles and the applied strain amplitude. The strain amplitude dependency of storage and loss modulus for MREs with 60%, 70%, and 80% iron particles is shown in Fig.1, respectively. For MREs with 60% iron particles as shown in Fig.1(a), the storage modulus ($G'$) and loss modulus ($G''$) are almost independent of the strain amplitudes and the number of cycles, except for a slight decrease of $G'$ with the increase of time at the high strain amplitude (100%). In contrast, $G'$ and $G''$ for MREs with 80% iron particles, as shown in Fig.1(c), are strongly dependent on the strain amplitudes and the number of cycles. The results show that $G'$ and $G''$ will first dramatically decrease with the increment of the strain amplitudes and fatigue time, and then the values reach a plateau when number of cycles further increase. It should be noted that under the applied strain amplitude of 100% the samples with 80% iron particles will fracture when the number of cycles is above $10^5$. The properties of MREs containing 70% iron particles are between the two samples which
Weaker than that between pure polymer materials. Intermolecular forces between particles and polymer is the weak intermolecular forces of MREs, because the propagation path is parallel to the direction of stretching [14]. This results from polymer materials whose propagation path is also vertical to the direction of particles’ chain-like structure. The result is different from conventional materials. A large number of cracks exist on the surface of samples with 80% iron particles after fatigue while only a small number of cracks present on the surface of MREs with 60% iron particles. It is worth mentioning that the crack propagation path orient vertical to the direction of stretching. And the crack propagation path is also vertical to the direction of particles’ chain-like structure. The result is different from conventional polymer materials whose propagation path is parallel to the direction of stretching [14]. This results from the weak intermolecular forces of MREs, because the intermolecular forces between particles and polymer is weaker than that between pure polymer materials.

The decrease in storage and loss modulus upon increasing strain amplitude and number of cycles is attributed to the filler networking broken down. The trapped or caged rubber in the filler networking is released through the large number of cycles and applied strain amplitudes [19, 20]. The phenomena of MREs containing 60% iron particles are obvious because a large number of particles exist in the matrix and the formation of agglomerates or chain-like structures is much more. As shown in Fig. 2(d), the crack propagation begins with the interface between the particles and matrix. The agglomerate is easy to be disaggregated when the sample is applied a tensile stress. However, for MREs containing 60% iron particles, there are only a few chain-like structures and agglomerates. Meanwhile, the bonding of force between particles and rubber chains is so strong that the filled networking is hardly broken down. The crack propagation begins with the break of intermolecular forces between polymer, which can be observed in Fig. 2(c). The crack propagation is all perpendicular direction of stress. Intermolecular forces of MREs containing 60% iron particles is stronger than that of of MREs containing 80% iron particles, so crack propagation of MREs containing 60% iron particles is more obvious and wider. When intermolecular forces were broken under the high strain amplitude and high number of cycles, the corresponding storage modulus will decrease. The loss modulus is determined mainly by the hydrodynamic effect of the filler so the strain amplitude dependence will be eliminated. This result also agrees well with Ref. [20].

Absolute MR effect is a key parameter for evaluating MREs performance. Here saturation MR effect (saturation magneto-induced modulus) is used to characterize the material. At this time, the applied magnetic field is 800 mT. The relationship between saturation MR effect and number of cycles for structured MREs with 60%, 70%, and 80% iron particles under various strain amplitudes (50%, 75%, and 100%) were indicated in Fig. 3(a), (b), and (c), respectively. In Fig. 3(a), the result shows that MR effect is independent of number of cycles with the increment of strain amplitudes from 50% to 100% when the content of iron particles is 60% in the matrix. However, MR effect of MREs with 70% and 80% iron particles content showed a remarkable change with the strain amplitudes and number of cycles. As shown in Fig. 3(b) and (c), MR effect can be divided into three stages (I, II, and III) with number of cycles. At stage I, MR effect decreases dramatically with the increment of number of cycles. At stage II, the values almost keep constant. At stage III, MR effect decreases further with the increment of number of cycles until the sample fractures. The change of MR effect will also be analyzed in the next section through the microstructure evolution of MREs during the processing of tensile.

**IV. MECHANISM STUDY**

Figure 4 shows that the process of rearrangement of chain-like structure for MREs with 60% iron particles is viewed by in situ observation of SEM. It is observed that the particle chain-structure is parallel to the external stretching. The particle chain-structure is not absolutely parallel to the direction of external field, especially in some local regions (Fig. 4(a)). When the sample is applied a strain amplitude, the particle chains tend to the direction of external stretching during the fatigue time. The particle aggregates will rearrange along the direction of stretching with the increment of strain amplitudes 0%, 100%, 200%, and 400%, corresponding to the picture Fig. 4(a)–(d) and form much more integrated chain-like structure. When the sample is applied a constant strain amplitude of 400%, the crack propagation is also present in another region of the sample.
FIG. 3 Relationship between MR effect and number of cycles for structured MREs with (a) 60%, (b) 70%, and (c) 80% iron particles, under various fatigue strain amplitudes.

(Fig.5). The arrow denote the direction of crack propagation. The local strain of the region may be much far away the 400% strain amplitude. Figure 5 reveals that the crack propagation is vertical to the particles chains and the direction of stretching, which consists well with the fatigue result in Fig.2.

Based on above experimental results, the evolution mechanism is analyzed to explain the fatigue process. Particles aggregates of MREs with 60% and 80% iron particles content are shown in Fig.6. As can be seen from this figure, a few particles form the aggregates while each particle is circled by the inner and upper rubber. The trapped or caged rubbers exist in the aggregates of MREs containing 80% iron particles content while the rubbers doesn’t exist for 60% iron particles content. The schematic process of aggregates is analogous to the carbon black filled rubber [20].

Figure 7 illustrates the schematics of microstructure evolution for MREs containing 80% iron particles during the processing of fatigue. Firstly, a large number of particles chains and aggregates exist in the matrix and the trapped or caged rubber is restrained in the filled networking. The filled networking will be disrupted and restrained rubber is released when the sample is loaded an external force. The interactional force between the iron particles under the external magnetic field will dramatically decrease and the magneto-induced shear modulus of MREs will also fall off, corresponding to the process of stage I. Secondly, the breakdown of the filler network will increase and the particles chains will rearrange and reform along the direction of stretching with the increment of number of cycles. At the same time, the upper rubber of particles will be tensioned along the direction of stretching. Above the factors, the value of magneto-induced modulus maintains a balance, corresponding to the process of stage II. Thirdly, when the number of cycles is further increasing, the upper rubber...
with 80% iron particles are strongly dependent on the strain amplitudes and number of cycles. The magneto-induced modulus of MREs containing 60% iron particles keeps a constant. It is worth mentioning that the magneto-induced modulus of MREs containing 80% iron particles is divided into three stages according to the fatigue time. The change of magneto-induced modulus of MREs is related to the evolution of microstructure of filled networking. The proposed fatigue mechanism could well explain the experimental phenomena.

VI. ACKNOWLEDGMENT

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