Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Test Method

A new magneto-dynamic compression technique for magnetorheological elastomers at high frequencies

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A R T I C L E I N F O

Keywords: Magnetorheological elastomers Dynamic compression test Linear viscoelastic region Magnetic field

ABSTRACT

A new magneto-dynamic compression technique was designed and manufactured to measure the magneto-viscoelastic properties of magnetorheological elastomers (MREs) at high frequencies. Isotropic MREs filled with carbonyl iron powder were synthesised, and three volumetric particle contents were studied—0%, 15% and 30%. Viscoelastic properties were calculated using stress–strain diagrams for each frequency and strain amplitude condition. The linear viscoelastic (LVE) region in compression mode was defined for MREs, and the influence of synthesis and characterisation variables in this region was analysed. Moreover, the compression magneto-viscoelastic properties were measured up to 200 Hz in the LVE region. Two types of tests were performed to characterise isotropic MREs in compression mode, a strain-sweep test to define the LVE region and a frequency-sweep test to study the magneto-viscoelastic properties in the LVE and at frequencies. The LVE region was determined at 0.3% by the higher particle content sample and at the lower frequency. Within the LVE region, the storage modulus and the loss factor increased with frequency and particle content. The compression magnetorheological (MR) effect increases with particle content and magnetic field density.

1. Introduction

During the last two decades interest in smart materials has grown. Magnetorheological elastomers (MREs) are classified as smart materials due to the variability of their properties with the application of an external magnetic field. This characteristic is the result of ferromagnetic particles embedded in the polymeric matrix.

MREs can be clustered based on filler particle distribution. When the particles are randomly distributed MREs are isotropic, and when the particles are aligned in a certain direction the MREs are anisotropic [1,2].

MREs normally operate in the pre-yield regime and are characterised by the field-dependent modulus [3]—the variation of storage modulus owing to a magnetic field is called magnetorheological (MR) effect. That effect is larger at low strain levels or within the linear viscoelastic (LVE) region [4–6], where viscoelastic properties are independent of strain. In literature, MRE properties were measured in shear mode, and consequently the LVE region was analysed in that mode, which was determined at the point when viscoelastic properties deviates 10% [7–10]. The LVE region decreases with particle content and becomes larger with frequency [11], while magnetic field does not restrict the LVE region [9,11,12].

MREs are used in many applications and some of them are working

https://doi.org/10.1016/j.polymertesting.2018.01.011 Received 23 November 2017; Accepted 15 January 2018 0142-9418/ © 2018 Elsevier Ltd. All rights reserved. in compression mode, such in isolators [13–18]. Therefore, the viscoelastic properties of these materials have to be characterised in compression mode.

In compression tests, Varga et al. [19] concluded that the variation of storage modulus is larger when the mechanical stress and applied magnetic field are parallel. Therefore, to characterise the MREs in compression mode, the equipment was modified to apply an external magnetic field. For that purpose, two solutions have been developed, one using permanent magnets (500 mT) [15,20,21] and the other using electromagnets (1T) [1,22,23].

According to compression characterisation devices, universal testing machines [22], fatigue machines [23,24] and dynamic mechanical analysers [1] have been adapted to characterise MREs. However, the maximum characterisation frequency was 25 Hz. To characterise MRE materials at higher frequencies, an electrodynamic shaker were used. Yang et al. [15] characterised MREs in compression mode up to 30 Hz. Nevertheless, larger frequencies up to 1 kHz can be characterised using an electrodynamic shaker [25], up to 1 kHz, although these characterisations were performed in shear mode.

In this work, we developed a new magneto-dynamic compression technique to characterise MREs at high frequencies. The novelty of this work lies in the definition of the LVE region for MREs in compression mode and its subsequent analysis as a function of synthesis and





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Fig. 1. Sketch of the vulcanisation of isotropic MREs.

Table 1

Theoretical and experimental densities of the studied samples.

Particle content	Theoretical [g/cm3]	Experimental [g/cm3]
0% 15% 30%	1.127 2.011 3.079	$\begin{array}{rrrr} 1.122 \ \pm \ 0.001 \\ 2.077 \ \pm \ 0.001 \\ 3.013 \ \pm \ 0.001 \end{array}$

characterisation variables. The compression magneto-viscoelastic properties of isotropic MREs within the linear region are measured at high frequencies. Using an electrodynamic shaker, frequencies from 50 to 200 Hz were characterised, and a magnetic field parallel to the mechanical stress was obtained with an electromagnet. A standard procedure was defined to calculate the viscoelastic properties of MREs using stress–strain diagrams. Two tests were employed, a strain-sweep test to define the LVE region of isotropic MREs in compression mode as a function of particle content and frequency and, using that limit, a frequency-sweep test to analyse magneto-viscoelastic properties in the linear region.

2. Materials

The isotropic MREs studied in this work contain carbonyl iron powder (CIP) as the filler and natural rubber (NR) as the matrix. The CIP particle were spherical with an average size of $1.25 \pm 0.55 \,\mu$ m. The particles were supplied by BASF, The Chemical Company. To determine the curing time of the NR, a rubber process analyser (RPA) was used at a frequency of 100 cycles per min at 150 °C and a rotatory angle of 0.5° for 20 min. After 2.14 min 90% of the 0% sample was vulcanised. Therefore, the vulcanisation was done at 180 °C for 10 min. By increasing the particle content, the matrix amount and consequently the curing time were reduced.

The mixing of the particles and the matrix was done with a two-roll mixing mill; the particles were added gently to guarantee a homogeneous mix. The mixture was poured into a 10-mm-thick mould (Fig. 1) and placed in an oven at 180 °C with a hydraulic pressure of 200 bar for 10 min (indicated with a P in Fig. 1). The samples were 10 \pm 0.1 mm in diameter and 10 \pm 0.05 mm high. Due to particle distribution, two kinds of MREs can be synthesised; in this work, the



Fig. 3. Experimental set-up.

particles were randomly distributed, resulting in homogeneous and isotropic MREs. Three volumetric particle contents were studied—0%, 15% and 30%. The density of each sample was measured and compared with theoretical densities (Table 1). A Nova Nano SEM 450 scanning electron microscope (SEM) was used to observe the particle distribution (Fig. 2). The images were taken in low-vacuum conditions with a voltage acceleration of 18 kV.

3. Experimental

A new magneto-dynamic compression test device was designed and manufactured (which was fixed to an electrodynamic shaker) to characterise MREs at high frequencies. In this study, the magneto-dynamic response was obtained in the frequency range of 50-200 Hz. The test is detailed in Fig. 3. The proposed test is an open-loop test in which a sinusoidal signal is introduced and the output signal is measured. The frequency and the level of the generated signal are defined using the analyser (A). The OROS (OR763) analyser consists of two output channels and four input channels. The analyser (A) generates a sinusoidal signal at a certain frequency that is sent to the electrodynamic shaker (B). The shaker (B) applies a sinusoidal displacement to the testing device, which is measured using an accelerometer (C). The response of the system is measured with a force sensor (D), and the magnetic field density applied to the testing device is regulated with a DC power supply (E). The data acquisition for acceleration and force signals is accomplished using the analyser (A).

After the overall view of the proposed technique is defined, the details of each element, the characterisation procedure and the data processing are explained.

Fig. 4(a) shows a photo of the whole testing device and Fig. 4(b) shows a close-up of the sample. A harmonic signal was sent to a Ling V406 electrodynamic shaker (1), which applies a harmonic movement at a defined frequency and level conditions. The connection between the testing device and the shaker was made via a ball joint (2), which only transmits a linear displacement to the device. The next element is



Fig. 2. SEM images of (a) 15% and (b) 30% isotropic MRE samples in low-vacuum conditions and with a voltage acceleration of 18 kV.

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