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Research articles

In-situ observation of the particle microstructure of magnetorheological elastomers in presence of mechanical strain and magnetic fields

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ABSTRACT

Magnetorheological elastomers are a type of smart hybrid material which combines elastic properties of a soft elastomer matrix with magnetic properties of magnetic micro particles. This composition creates a complex interplay of magnetic and elastic phenomena. The best known is the magnetorheological effect which describes the alteration of the Young's modulus with external magnetic fields. The particle properties and their arrangement in the material play a major role in determining the resulting effects. In this paper X-ray microtomography has been utilized to analyze the particle microstructure. Besides a characterization of the particle microstructure in presence of magnetic fields, here the in-situ observation was combined with an application of mechanical strain. Thus, the situation of the sample during mechanical testing was recreated during tomography to observe the particle microstructure under the exact circumstances of occurring magnetorheological effects. A significant impact of the magnetic field and the strain on the rotation of the particles and their radial distribution was verified.

1. Introduction

The idea of magnetorheological elastomers was developed to overcome some disadvantages occurring with the well-established magnetorheological fluids, which are sedimentation and leakage in particular. A great amount of scientific work was conducted to obtain specific magnetic and mechanical properties with this new material and to gain an understanding on the present physical correlations leading to the observed macroscopic magnetorheological effects [1–6]. Most of this complex interplay of mechanic, magnetic and structural properties is not understood yet, which motivates a lot of recent fundamental research connected to magnetorheological elastomers.

By now it is well known that the arrangement of the particles in the matrix has an important influence on the macroscopic properties and is a key parameter in tailoring magnetorheological elastomers [1,7]. X-ray computed microtomography proved to be a reliable method to analyze the arrangement of the particles in those materials [8–10]. Besides the approaches to track particles during varying external magnetic fields [11,12], recent work utilizes statistical evaluation methods to characterize the particle microstructure as well [13]. This way seems to be a promising approach to gain a deeper understanding in the reactions of particle arrangements on external stimuli and may provide important indications for theoretical studies of magnetic hybrid materials [14].

As said, previous studies analyzed the interplay of the particle microstructure of magnetorheological elastomers with magnetic fields. These results were connected to occurring macroscopic magnetorheological effects. This approach often neglects the fact that during mechanical testing and thus during occurring magnetorheological effects mechanical strain is present. In this work the analysis of particle microstructures was expanded to structure characterization in presence of magnetic fields *and* mechanical strain. To accomplish this measurement X-ray computed microtomography of a magnetorheological elastomer showing strong magnetorheological effects was performed in presence of magnetic fields and mechanical compression for the first time.

2. Materials and methods

2.1. Sample preparation

The investigated samples were synthesized using silicone polymers by Gelest Inc. The composition of the polymers used is given in Table 1.

Höganäs® ASC200 carbonyl iron particles with a median diameter of $d_{50} = 40\ \mu m$ were added to obtain magnetic properties. The irregular shape of this type of particles enables an optical analysis

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Table 1
Used polymer composition for preparing the samples.

Component	Amount
Polydimethylsiloxane DMS-V25	100 parts
Copolymer HMS-151	2.8 parts
Catalyst: Alfa Aesar Platinum (0)-1,3-divinyl-1,1,3-tetramethyldisiloxane, 1:20	0.12 parts

of their rotation [12]. An amount of 40 wt% was chosen to get strong magnetorheological effects as well as a statistically generous number of evaluable particles. Due to the separate catalyst, the polymerisation time could be adjusted to a manageable minimum. Thereby samples with a mostly isotropic particle distribution were accomplished. Large cylindrically shaped samples with a diameter of 12 mm were produced for mechanical testing; smaller samples with a diameter of 4 mm were used for tomography. All samples were synthesizes simultaneously from the same batch and mix of material to ensure consistent material properties for all samples.

2.2. Mechanical testing

For the mechanical characterization a Dyna-Mess universal testing device was used to perform stress-strain tests. A setup containing two permanent magnets was used to provide a homogeneous magnetic field with $B = 250$ mT. To observe possible nonreversible effects the tests were performed without, in presence and again without the magnetic field consecutively. As visualized in Fig. 1, the direction of the mechanical testing and the direction of the magnetic field are parallel to the cylinder axis of the samples.

Within the region of linear elastic behaviour from 10 % to 20 % strain in direction of the magnetic field a Young's modulus was calculated. The magnetorheological effect *MRE* was calculated from the Young's moduli E_0 before the application of the magnetic field, and E_B in the presence of the 250 mT field:

$$MRE = \frac{E_B - E_0}{E_B} \quad (1)$$

2.3. Tomography

To analyze the particle microstructure of the sample, the TomoTU setup [15] was deployed at an acceleration voltage of U = 90 kV and an electron current of I = 170 μA. For a full rotation of the sample 1440 radiographs were taken in 0.25° steps to maximize the spatial resolution. With the cone beam geometry of the TomoTU setup a magnification of the projection of 15 was chosen to obtain a resolution better than 15 μm [16]. Slight variations in radiation intensity were corrected using individual gain and offset radiographs for all tomograms. The reconstruction process calculating a 3D model from the radiographs was performed by a software package developed in our institute based on the FDK algorithm [17].

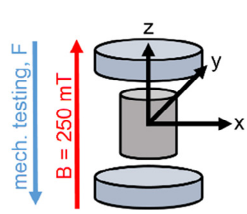

Fig. 1. Scheme of the magnetic field direction and the mechanical testing. The cylindrical sample is placed between two disc shaped permanent magnets during mechanical testing.

In analogy to the magnetic setup utilized for the mechanical testing, a setup containing two strong movable permanent magnets were used here as well. The distance was set to obtain the maximum field of $B = 250$ mT, in analogy to the situation at the mechanical testing. Similar setups were used successfully before to analyze the particle microstructure of lower concentrated magnetorheological elastomers [11–13]. Measurements of the magnetic field show a field homogeneity of over 95% for all directions at 250 mT within the sample volume. For this investigation the setup was upgraded to apply mechanical strain to the sample. To accomplish a distinct uniaxial deformation an adjustable plunger was mounted to the upper movable magnet. The applied strain was evaluated via the radiographs of the sample during strain application. Thus it was possible to adjust the strain to be within the region of linear elasticity, as determined via mechanical testing.

With this device a zero field can only be obtained by removing the magnets, whereas a strain only can be applied with the magnets in place. With magnets in place the minimum magnetic field strength at the sample position equals to 10 mT. To avoid uncertainties in data interpretation due to unknown effects induced by the minimal field of 10 mT measurements were conducted both for the removed magnets as well as for the field of 10 mT. The utilized setup is shown in Fig. 2, the parameters for all tomography measurements are concluded in Table 2.

2.4. Image processing and data evaluation

The reconstructed tomography data was processed in Matlab with the plugin DIPimage [18]. To safely separate the particles, an algorithm utilizing a seeded watershed algorithm in combination with a local threshold was applied [19]. About 50.000 particles

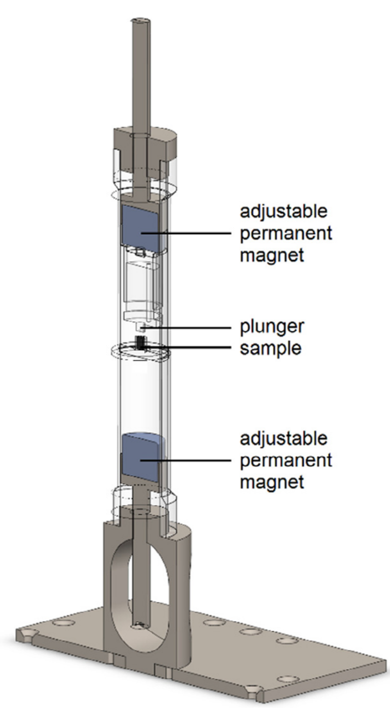

Fig. 2. A cut 3D CAD model of the utilized magnet setup which was used with the tomography site. The sample is placed in the middle between the movable permanent magnets.