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# Ring-like structures in magnetoactive elastomers based on magnetic hard powder

G V Stepanov<sup>1</sup> , D Yu Borin<sup>2</sup> , A V Bakhtiarov<sup>1</sup>, D A Lobanov<sup>1</sup> and P A Storozhenko<sup>1</sup>

<sup>1</sup> State Scientific Research Institute for Chemical Technologies of Organoelement Compounds, Moscow 105118, Russia

<sup>2</sup> Chair of Magnetofluidynamics, Measurement and Automation Technology, Technische Universität Dresden, 01062 Dresden, Germany

E-mail: [dmitry.borin@tu-dresden.de](mailto:dmitry.borin@tu-dresden.de)

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## Abstract

In this study, we report on the experimentally observed phenomenon of ring-like structures formation from chains of magnetic microparticles in magnetoactive elastomers based on magnetic hard powder. In order to find an explanation for the specific macroscopic magnetic properties of composites, microstructural observations on the elastic composite based on a thin layer of magnetic hard particles have been carried out. Particles embedded in such magnetic composites can move inside the matrix with some restrictions and form chain-like structures on being subjected to magnetizing and turn over as first the strength of the external magnetic field decreases and then the polarity of the field switches to opposite. The quantitative parameters of these processes depend on a number of factors including coercivity and remanence of particles, polymer matrix elasticity, and external field intensity. In zero-field, the magnetized particles can assemble into ring-like structures, thus tending to bring the free energy of the overall system to a minimum. The study is primarily aimed to shed a light on understanding the specific magnetic properties of magnetic polymers, such as magnetoactive or magnetorheological elastomers with a magnetic hard filler. Furthermore, the phenomenon of ring-like structures formation can be used for controlled remote patterning of particles in magnetic elastic composite thin films, which is attractive for various applications.

Supplementary material for this article is available [online](#)

Keywords: magnetoactive elastomers, magnetic microparticles, controlled structuring

(Some figures may appear in colour only in the online journal)

## 1. Introduction

Interest towards magnetorheological (MR) elastomers has been continuously growing over time. There have been obtained a number of novel elastomeric compositions based on various fillers and polymer matrices. Different names given to this kind of material, be that magnetoactive, magnetocontrollable or MR elastomers, magnetic gels or polymers, are reflections of a vast variety of properties demonstrated by this type of composites [1–6]. Lately, magnetic elastomers containing



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magnetically hard particles have attracted increased attention [7–13]. Such materials are expected to demonstrate the capability of functioning as both conventional magnetocontrollable elastomers and good dampers requiring no bulky system of magnets for setting their conditions. Despite the fact that such materials assume rather limited external controllability, as a result of which they will exhibit a lower efficiency, their compositional simplicity will be a compensation to determine their extensive implementation.

Composites of this type were initially expected to exhibit a remnant magnetization and possess an interior magnetic field conditioning their damping features. As has been discovered, primary exposition to a magnetic field results in a memorized field-influence effect manifesting itself as an asymmetrical dependence of their elastic properties on external magnetic fields [7–10]. These results have found confirmation in [11]. It should be noted, however, that according to [11], this is only true if the magnetizing field was higher than the applied magnetic field. It can be assumed that this condition depends on the elasticity of the polymer matrix.

Another distinctive feature of MR elastomers based on a magnetic hard filler is that the loss-factor values they demonstrate have been noticed to be significantly higher than those of multiple members of the MRE-family [12]. At the same time, these magnetic elastomers can be expected to show a major positive or negative magnetostriction depending on the degree of co-directionality of primary magnetization and external field. This hypothesis is not yet addressed experimentally and further studies are necessary. Experimental measurements of the magnetic properties of these composites showed a significant reduction in the coercive force measured for the composite compared to the original magnetic filler. When a magnetic filler is introduced into a polymer matrix, i.e. diluted, one can expect an increase in the coercive force of the material due to a decrease in the demagnetizing factor, but in experiments the coercive force decreases [10]. As discussed in studies [14–20], the coercive force of the composite with an elastic polymeric matrix decreases due to changes in the mechanism of magnetization from pure magnetization of the particle to its rotation, turning over in the reverse magnetic field. As has been established, influenced by external magnetic fields, magnetized high-coercivity particles contained in the polymer tend to turn after the field as it changes direction. The softness of the matrices conditions the possibility for the particles to rotate at small magnetic fields. As a result, the mechanical changes of magnetization vector that takes place in the sample, occur at weaker fields in comparison to the coercivity of the filler. The behavior of such materials under the influence of magnetic field has been a subject for discussion in several recent experimental and theoretical studies [14–20]. Microstructural changes depending on the magnetic field used to magnetize the bulk sample as well as the magnetic field additionally applied to the sample with a certain residual magnetization were investigated using computed microtomography in [21]. The two main processes associated with microstructural changes and responsible for changing the macroscopic properties of the material during its magnetization are the structuring of particles into chain aggregates and the rotation of

particles within an elastic matrix. Some effects are also related to the processes of re-magnetization within magnetic particles and under certain circumstances to their under-magnetization [18, 20]. The computed tomography method is used to study the microstructure of a composite in a static state and has some limitations regarding the ability to vary the intensity and direction of the magnetic field applied during the measurements. Therefore, additional methods of microstructural examination are required to explain the experimental results of magnetic measurements and to confirm the proposed theoretical models.

From the practical point of view, specimens based on magnetic hard powder and elastic matrices with medium rigidity have been meanwhile tested as the working bulk element in acceleration sensors development [22, 23]. It is obvious that correct explanations of the processes of magnetization and remagnetization of magnetic hard particles introduced into an elastic matrix are important for further development of such practical applications.

In current work we approach the question of explanation of specific magnetic behavior of composites in terms of microstructural changes observed experimentally using optical microscopy and samples based on a thin quasi-monolayer of particles. This approach allows tracking microstructural changes in the process of varying the intensity and direction of the magnetic field. On the one hand, our work is dedicated to demonstrate the basic behavioral features of high-coercivity particles dispersed in elastic polymers, providing a contribution to the physics of complex soft matter. On the other hand, we experimentally investigate a specific type of intelligent material in which magnetic microparticles are distributed in one plane. The composites under study can be the basis of a magnetically controlled and sensitive thin surface, for example, the so-called ‘smart skin’, in some areas of which it is possible to create certain patterns of particles through passive and active magnetization/remagnetization. The use of a magnetic hard filler allows to change the initial properties and microstructure of the composite, i.e. its anisotropy, after polymerization and at local areas. The paper is organized as follows. First, we describe used materials and chemicals, as well as basics of composite manufacturing with a focus on a fabrication of a quasi monolayer of particles in a polymeric matrix. In the same section used experimental technique is briefly presented. In experimental section we consider hysteresis magnetization loops of bulk samples with different rigidities focusing on certain features of material field response. These considerations are accompanied with optical microstructural observation of samples based on a quasi monolayer of microparticles under comparable magnetic conditions. Then the study is finalized with a discussion and outlook.

## 2. Samples and methods

Specimens of magnetic elastomer were fabricated using SIEL-254, a two-component silicone semi-product manufactured at Russian State Scientific Research Institute for Chemical Technologies of Organoelement Compounds (GNIChTEOS).

Consisting of two separately stored liquid ingredients featuring a vinyl- and a hydride-containing agent, it is a convenient means of obtaining polymer samples with the desired mechanical properties [9, 10, 12]. Mixed together in a certain proportion, they readily polymerize into a 3D cross-linked net on thermal treatment. For the purpose of fabricating samples with the desired rigidity, we also added silicone oil M100.

MQP-S-11-9- and YMM-Q-grade NdFeB-alloy powders with spherical particles and those having irregular geometries (figure 1), coercivities being 900 mT and 250 mT, respectively, were used as fillers. Well-capable of clinging to metal, glass, or ceramics, silicone elastomers demonstrate good adhesion to the surfaces of NdFeB- or Fe-grains especially when they have preliminarily been coated with hydride-containing silicones. Such surface-modification is aimed at conditioning a regularly spaced distribution of the particles inside the silicone resin. The treatment was carried out using a mixture of oligo-methyl hydrogen siloxanes  $[\text{CH}_3\text{SiHO}]_n$  and silicone oil M100 dissolved in toluene.

Owing to the fact that iron-based particles practically always contain multiple hydroxyl-groups on their surfaces occurring from interaction with adsorbed water, the hydrophobization mechanism of the filler relies on the chemical interaction of the OH-groups with the Si-H-bonds of the modifying agent. The modification of metal particles with silicone-groups bonded to their surfaces is accompanied by the formation of water. At the same time, silicone oil added serves as an agent preventing aggregation of the particles.

To estimate the macroscopic properties, bulk samples with a filler concentration of  $\sim 25$  vol.% and various elasticity were manufactured. The magnetization hysteresis loops were obtained using a Lake Shore 7407 vibrating-sample magnetometer and cylindrical-shaped specimens having 2 mm in thickness and 2 mm in diameter. Shear moduli determinations were carried out using an Anton Paar Physica MCR301 rheometer and disc shaped specimens having 2 mm in thickness and 14 mm in diameter. To avoid a slip problem [24] in the rheometry, the specimens were glued to the plates of the measuring geometry during the oscillating tests.

A sample with a quasi-monolayer of magnetic filler was fabricated using the following method. The prepared solution for polymerization of silicone rubbers SIEL-254 by GNIChT-EOS consists of two components in the ratio of 1:10, with the addition of silicone oil M-100 in the amount of 50 wt%. The solution was divided into two parts. One part was spread on the glass surface with a layer of about 1 mm in thickness, which was partially polymerized at 100 °C for 30 min. Then particles of the modified NdFeB powder were sprayed on the surface. The particles adhered to the rubber surface due to adhesion, as their modified surface had high affinity to the silicone rubber surface. The particles, which did not adhere to the rubber surface, were deflated by air flow. Thus, a quasi-monolayer of NdFeB particles was formed. The obtained surface was then covered with the other part of silicone rubber by forming a second particle free layer of about 1 mm in thickness with farther polymerization of the composite carried out at 120 °C during 1 h. Thus, a quasi-monolayer of particles located between two layers of silicone rubber

was formed on the glass plate. Samples with spherical MQP-S-11-9-grade particles and samples with irregular YMM-Q-grade particles were produced using this technology. Images of the real particle layer are presented in the experimental part. According to a rough estimate by calculating the number of particles, their volume concentration in the layers of studied samples is about 20%.

The principal microscopic observations were done using a setup, whose structure scheme is shown in figure 2. A film of composite with a quasi-monolayer of magnetic particles is placed between the two poles of the electromagnet so that the magnetic field is directed in the specimen plane. Illuminated with a light source from one side, the sample was pointed at with a CCD-camera installed on the other side. As the intensity of magnetic field created between the poles is varied by means of a computer controlling the DCI power supply, the image captured in the CCD Camera (20 MP) is digitally recorded.

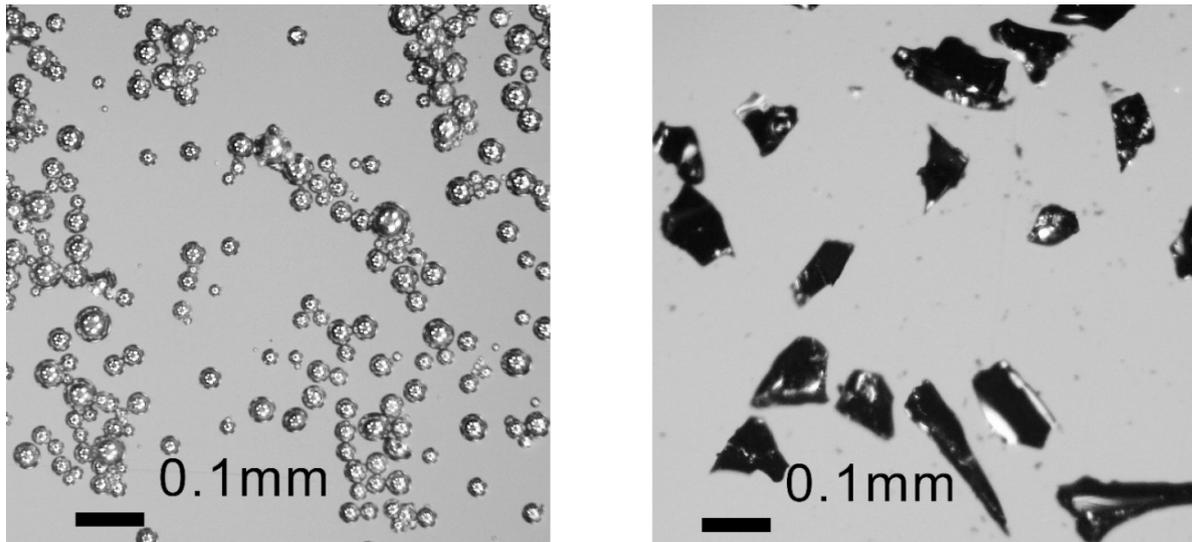
### 3. Results and discussion

#### 3.1. Magnetization

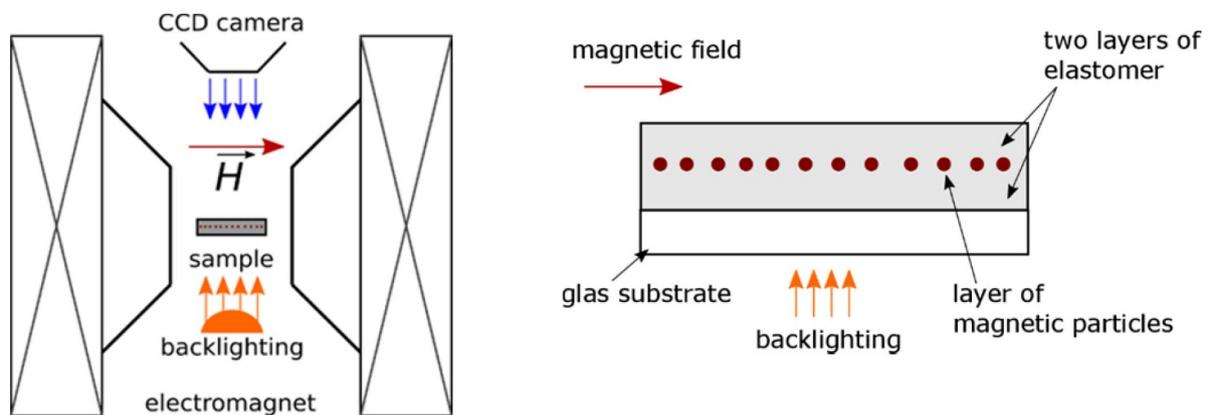
The magnetization of bulk samples of magnetic elastomer specimens prepared on the basis of polymers with different rigidities is demonstrated in figure 3. Equally filled with a powder of the same type, being MQP-S-11-9 containing spherical particles, at a concentration of 75 wt% ( $\sim 25$  vol. %), one of them has a shear modulus of 1 MPa, whereas that of the other is 50 kPa. As can be seen the rigid and soft specimens demonstrate radically different magnetic properties. Despite identical chemical compositions, their magnetization hysteresis loops are obviously dissimilar.

It has been reported previously [14–20] that such differences may be explained by interior structuring and rotation of particles occurring inside elastic polymer matrices, whereas rigid specimens demonstrate magnetization curves similar to those of pure filler. Indeed, rigidly embedded in the polymer, magnetic particles have practically no possibility to move. On the contrary, soft polymer matrices allow such motion, as a result of which they assemble into chain-like structures. On removing the sample from the area of influence of magnetic field, the elastic forces caused by distortions resulting from grains displacements tend to return them to their initial positions. However, characterized with strong remnant magnetization, magnetically hard particles retain the structures they have gathered into even after the external field is off.

At the same time, influenced by a reverse magnetic field, magnetized particles may either change their polarity by internal re-arrangement or turn after the field tending to make their magnetization vectors codirectional with it. Meanwhile, the rotation of particles may be observed at fields noticeably weaker as compared to the coercivity of the pure filler. As a result, the coercive force demonstrated by a soft sample will be lower than that exhibited by rigid specimens. It should be noted, however, that remnant magnetizations observed in elastic samples may also be significantly weaker exhibiting a strong dependence on the primary magnetizing vector.



**Figure 1.** Microscopic images of the used magnetic powder particles MQP-S-11-9 (left) and YMM-Q (right).



**Figure 2.** Scheme of the setup used for the microscopic observations (left) and structure of the sample (right).

As shown in figures 3 and 4, starting from a point P1 corresponding to a primary magnetization condition, the hysteresis loop recorded for a soft sample exhibits a certain asymmetry between the positions of its descending and ascending branches with respect to the origin. The fact that the ascending curve goes very close to the origin is indicative of the possibility for the coercivity to take practically the zero value. However, samples based on rigid polymer matrices demonstrate symmetric hysteresis loops characterized by equal coercivity and remanence values for both branches.

### 3.2. Microstructural observations

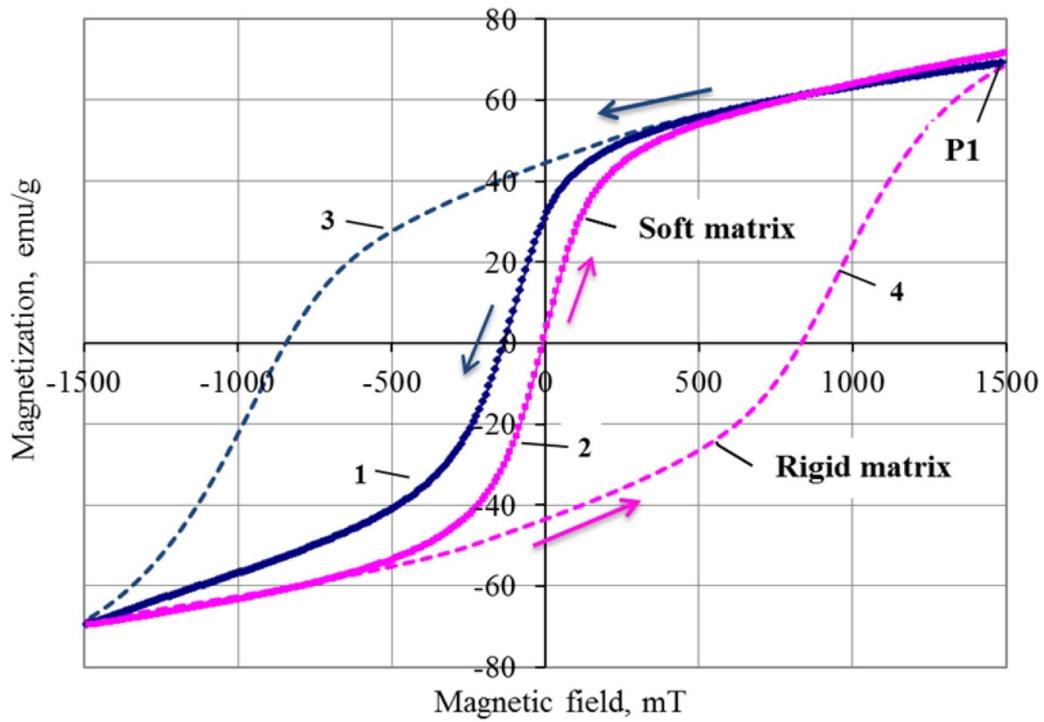
A more profound comprehension of the phenomena of interest can be reached when considering the curves brought in figure 4 complemented with microstructural observations taken for a thin layer of soft magnetic elastomer, considered as a model material. This model thin layer based sample contains the same magnetic filler and has a polymer matrix with the same elasticity as the bulk specimen. These parameters are determining for the specific properties of the composite.

For example, figure 5 presents a pattern of distribution of particles in a monolayer of initial non-magnetized composite corresponding to point 0 in figure 4. The particles are arranged randomly inside the matrix. The numbers provided on the microscopic images correspond to the points given in figure 4.

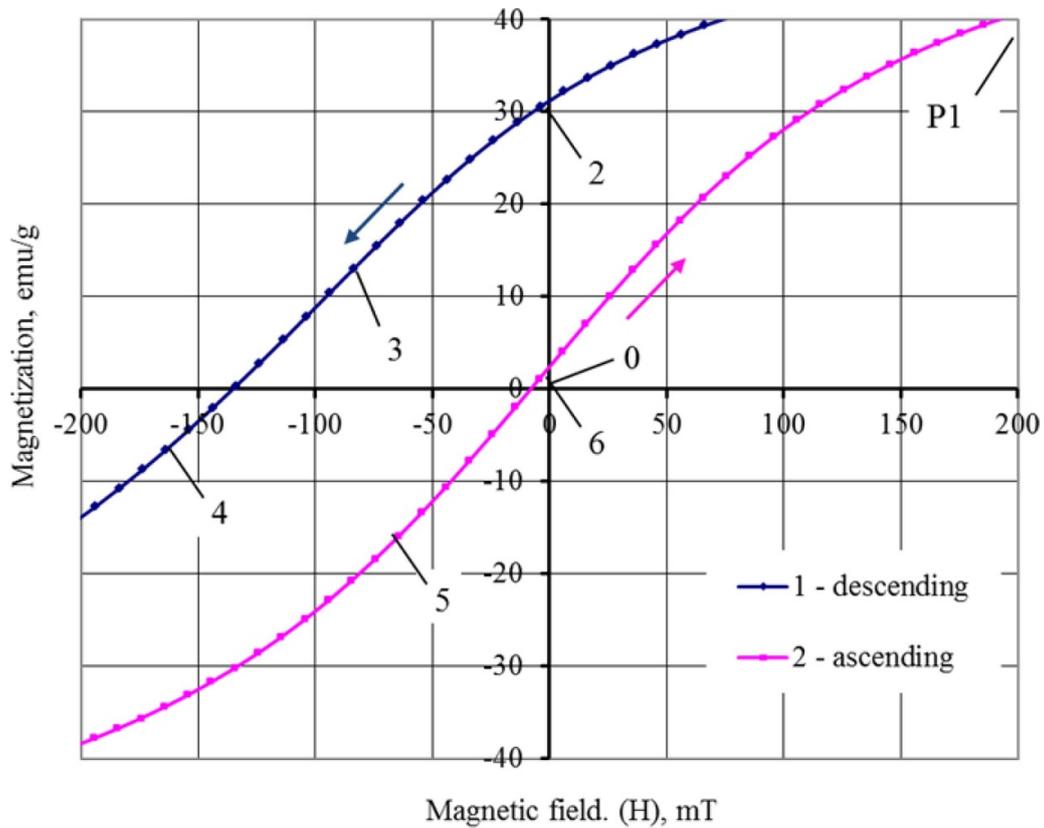
On exposure to a primary magnetic field of 1.5 T, the sample develops chain-like structures in the bulk, a zoomed-out image of which is presented in figure 6 (point 1 in figure 4). As can be noticed, influenced by the external field, the particles have overcome the elastic forces of the polymer and assembled into long chains.

However, as may be seen from figure 7, the interior structure suffers noticeable changes when the field is off. Removal of magnetic field influence results in diminishing of the magnetic moments of high-coercivity hard particles. At the same time, their remanence remains sufficiently high making it possible to retain the chains undestroyed, distorted by the tendency of the polymer to diminish interior stress, though.

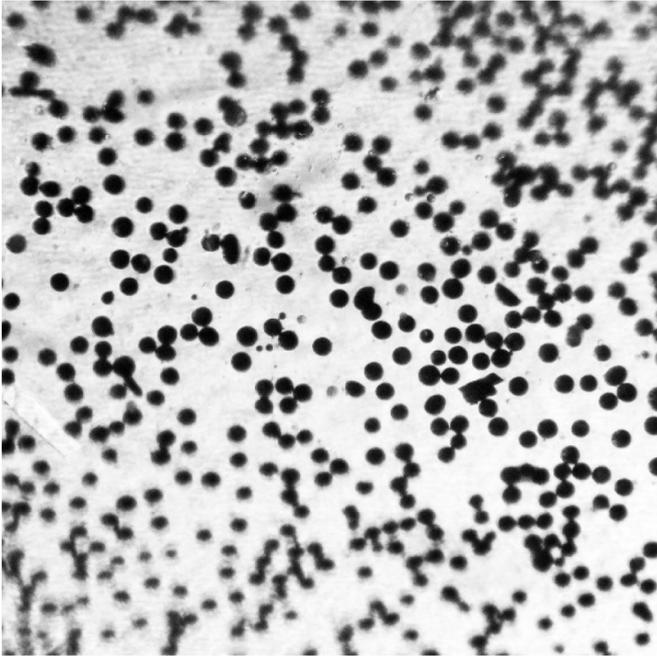
Application of an 80 mT reverse magnetic field destroys their preferred orientation resulting in a more chaotic pattern (figure 8). In these conditions, the particles are still gathered



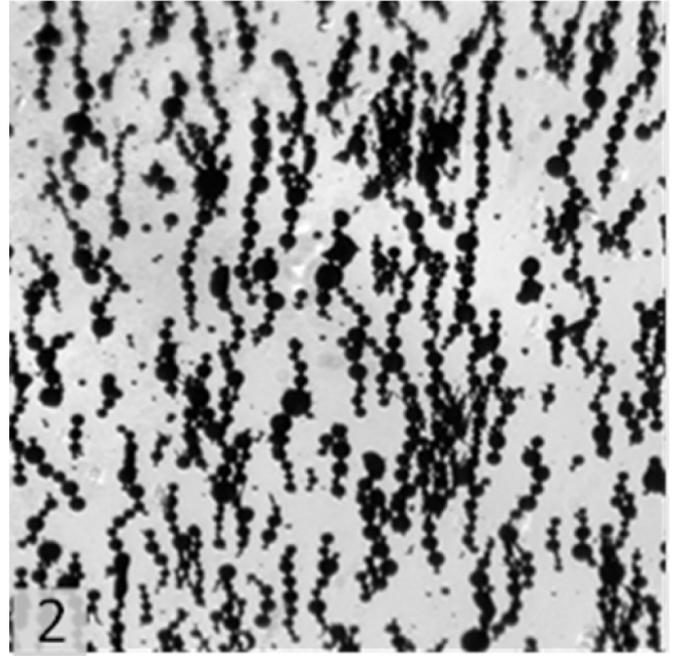
**Figure 3.** Hysteresis magnetization loops obtained specimens with a soft (lines 1 and 2,  $G' = 50$  kPa) and rigid (lines 3 and 4,  $G' = 1$  MPa) polymer matrix. The arrows show the path of variation of magnetization starting from primary exposition to the strongest magnetic field (point P1).



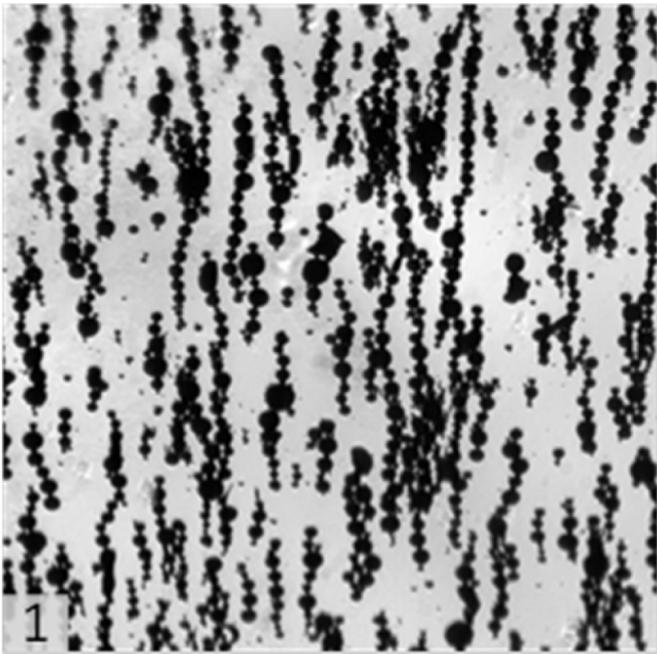
**Figure 4.** Central part (low-field range) of the magnetization hysteresis loop obtained for a specimen of magnetic elastomer with a soft polymer matrix ( $G' = 50$  kPa).



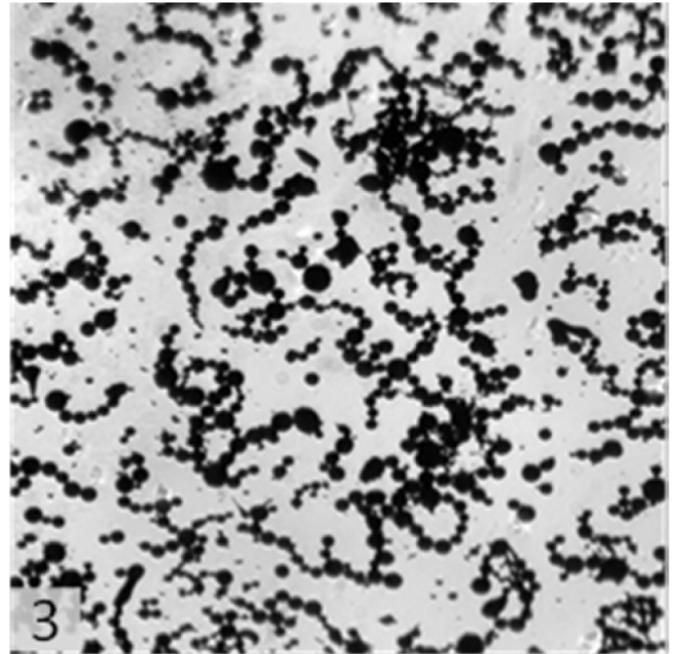
**Figure 5.** Magnetic particles of the MQP-S-11-9-grade NdFeB-alloy embedded in the polymer matrix before being subjected to primary magnetization (point 0 in figure 4).



**Figure 7.** Chains of magnetic particles distorted by the elastic forces of the polymer after diminishing the external field to zero (point 2 in figure 4).



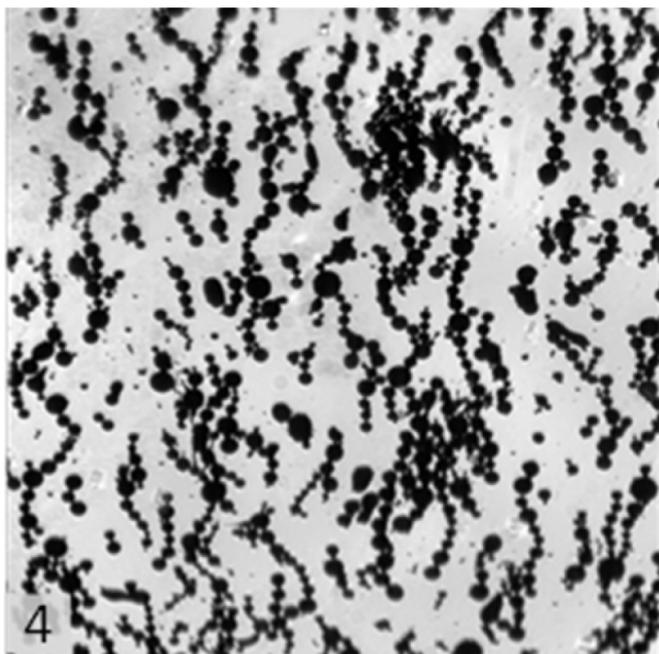
**Figure 6.** Magnetic particles of the MQP-S-11-9-grade NdFeB-alloy embedded in the polymer matrix influenced by a 1.5 T external field (point P1 in figure 4).



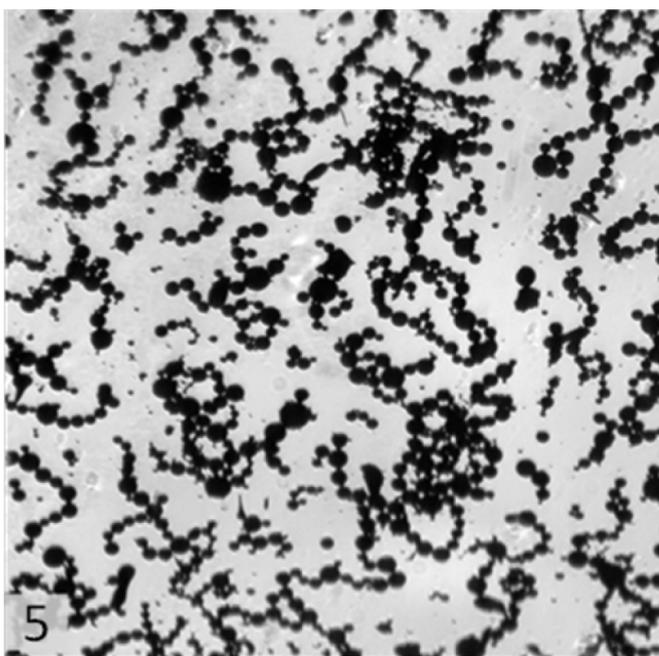
**Figure 8.** Chain-like structures 'perpendicular' to the external magnetic field vector. The sample is being under the influence of a reverse field of 80 mT (point 3 in figure 4).

into chains; however, they now demonstrate a tendency for significantly more pronounced warping, which even creates an impression that the chains are inclined to turn perpendicular to the exterior field vector. On further strengthening of the reverse field, magnetic particles start turning and restructuring into new chains along the magnetic force lines.

Figure 9 depicts a pattern observed in a 155 mT reverse magnetic field. As the field becomes yet stronger (1 T), the chains demonstrate only a slight straightening without fundamental changes. If, however, the reverse external magnetic field weakens to 80 mT, the overall structure, influenced by the elastic forces of the polymer, begins to suffer changes again



**Figure 9.** Formation of a new set of chains of straightened-up configuration in a reverse magnetic field of 155 mT (point 4 in figure 4).



**Figure 10.** Initiation of formation of rings by magnetized particles in an external reverse magnetic field reduced to 80 mT from a higher value (point 5 in figure 4).

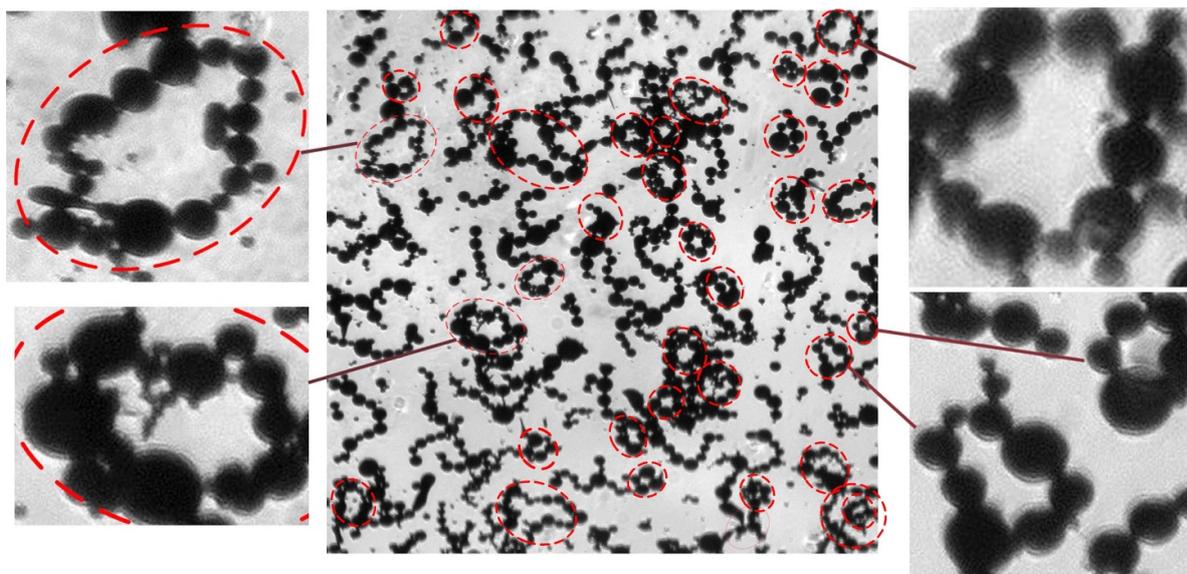
(figure 10). A result of the re-arrangement is the beginning of ring-like complexes formation.

It should be noted that while the reverse magnetic field becomes weaker approaching the point of polarity reversal, the magnetized particles start demonstrating the tendency for turning before it reaches the zero value. At the same time,

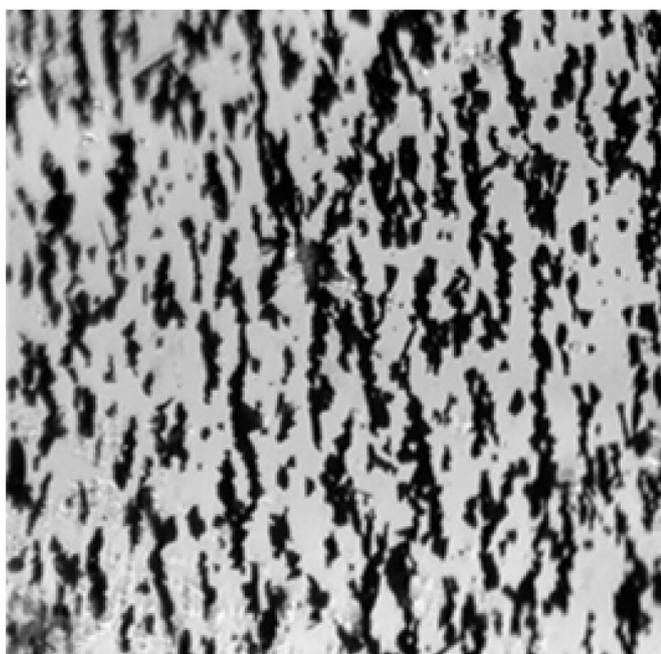
interactions among the grains are not interrupted and they continue to affect each other by means of their magnetized poles. Meanwhile, in the area of zero-field, the formation of rings progresses and finally results in fundamental structural changes: tending to diminish interior tension, the elastic matrix influences the particles forcing them into their initial positions. In figure 11, the ring-like formations are circled with dash-lines. Additionally, these structural rearrangements of spherical particles occurring in a magnetized sample being placed in a reverse magnetic field are demonstrated in video 1 in supplementary materials (available online at <https://stacks.iop.org/SMS/30/015023/mmedia>).

Thus, the particles filling the composite material may structure according to the following scenarios. The first one is regarded to the primary magnetization of the sample in a magnetic field. The chain-like structures forming as a result of the initial exposure of the sample to a magnetic field occur lined up with the vector of the influence. Owing to the fact that the formation of chains causes tension in the bulk of the polymer, the structures either suffer distortions or partly fall apart on turning the field off. However, high coercivity and strong remnant magnetization work against their destruction preserving them in the main. As may be seen, the specifics of this process are determined by the coercivity and remnant magnetization complemented with the elastic properties of the matrix. The second scenario is related to the subjecting the sample to the reverse magnetic field. If the external magnetic field changes polarity abruptly, particles start turning inside the soft matrix in order to make their magnetic moments co-directional with the new field vector. As the negative magnetic field becomes stronger, the particles form chains arranged in a weaker order as compared to a direct-field case. This phenomenon may be explained by the fact that the particle having to turn over before becoming a building unit for a chain forming under the influence of a reverse magnetic field experiences the necessity to overcome a more intensive resistance of the polymer matrix. At the same time, confined in a rigid matrix, it would rather demonstrate the conventional mechanism of demagnetizing followed by changing its polarity to opposite (curves 3 and 4 in figure 3). The most interesting case is observed when the external magnetic field is gradually reduced to zero. The magnetized particles start forming structures only because of their internal field in accordance with the magnitude of the elastic features of the polymer matrix. As the forces caused by tension inside the polymer tend to return the particles into their initial disordered state, magnetic interactions among them tend to diminish the overall system energy, as a result of which there occur ring-like structures confining magnetic flux. Meanwhile, the overall tendency to form chains significantly deviating from the external field direction becomes more pronounced. As may be noticed, the ascending branch of the magnetization hysteresis loop lies close to the origin, which assumes low coercivity and remnant magnetization magnitudes.

To confirm the general nature of the phenomenon, a similar experiment with a polymer matrix containing particles of irregular shape (YMM-Q-grade NdFeB-alloy powder) with lower coercivity was carried out. As in the experiments



**Figure 11.** Ring-like structures formed by magnetized NdFeB-alloy particles on diminishing the reversal field to zero (point 6 in figure 4).



**Figure 12.** Magnetic particles of the YMM-Q-grade NdFeB-alloy powder arranged into chains at 1.5 T.

conducted just previously, a monolayer of magnetic elastomer was first subjected to magnetizing at 1.5 T, by which its internal structure was formed (figure 12).

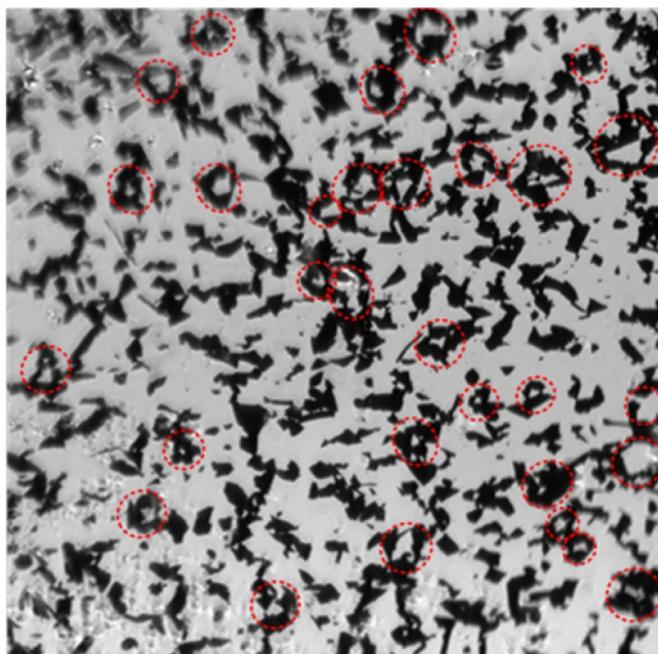
On switching magnetic field polarity to opposite followed by turning the field off, the particles of irregular shape also exhibit the tendency to arrange into rings (figure 13). Additionally, the structural re-arrangements of particles of irregular shape occurring in a magnetized sample being placed in a reverse magnetic field are demonstrated in video 2 in supplementary materials. Thus, the generality of the phenomena of structuring for the considered types of magnetic filler

is confirmed. After removing the negative field, the particles start to trim the chains dynamically. In the absence of an external magnetic field some particle chains are rolled up into rings. Obviously, this structure minimizes the magnetic energy. Meanwhile, the chain-like structures, which are more stable at stronger reverse fields, become significantly warped noticeably losing co-directionality with the external field vector and partly fall apart. The fact that the chains retain in the polymer to a certain degree is probably indicative of some steric factors preventing rings formation. Yet, the curvature of the chains may suggest that in zero-field the polymer remains stressed, as a result of which some particles remain ‘frozen’ in positions close to being perpendicular to the direction of the previously applied influence. For a strict quantitative evaluation of physical parameters of composites and external conditions under which ring-shaped structures appear, further systematic study combined with both computer modeling and analytical theoretical calculations is required. In fact, the possibility of magnetic particles to form structures differ to chains has already been a subject of theoretical [25–28] as well as experimental [29] considerations for the case of magnetically soft particles dispersed in a liquid matrix.

In addition, we will point out that the processes of structuring are fully reversible, and the obtained magnetization values as well as visualized structures are reproducible. The only situation which could not be replicated is the initial state at which particles are homogeneously distributed. It corresponds to the point 0 in figure 4 and microstructure given in figure 5.

#### 4. Conclusion and outlook

Analysis of the performance of elastic polymeric composites based on a magnetic hard filler has led us to the conclusion that along with assembling into chains and rotation, there is an interval of magnetic field strengths applied to the composite, in which particles form ring-like structures. Moved by the



**Figure 13.** Magnetized YMM-Q-grade NdFeB-particles at zero-field after application of the reverse field (0.9 T). Ring-like structures are denoted by dash-lines.

elastic forces directed at the minimization of internal tension in the polymer and held connected with each other by their magnetic moments, the particles assemble into complexes confining magnetic flux, which manifests the tendency of the overall system to diminish its free energy. This work experimentally demonstrates the fundamental possibility of creating ring structures in magnetoactive elastomers. Identification of key parameters responsible for this process was not part of the study, but should be a priority in subsequent work. For further systematic studies it will be necessary to involve computer simulation and physical analytical models intended for the target identification of physical quantities that are determining the conditions under which the formation of rings can take place.

This work not only contributes to an understanding of the physics of magnetic phenomena in elastic magnetic polymer composites, but also serve as a basis for the further development of intelligent composites with adjustable anisotropy. Here we are referring to the change in anisotropy of magnetic elastomer after its polymerization and demonstrate a possibility to obtain magnetic particle structures differ to conventional chains. Obviously, the thin layer of composite with ring structures will have, in particular, other properties (e.g. optical) than the layer of composite with linear chain aggregates. Using different situations of composite magnetization, it is possible to influence the appearance of certain types of microstructural elements. The results of the study provide the basis to develop of a magnetically controlled and sensitive thin surface, for example, for the so-called ‘smart skin’, in some areas of which it is possible to create certain patterns of particles through passive and active magnetization and re-magnetization.

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## ORCID iDs

G V Stepanov  <https://orcid.org/0000-0003-0053-1883>

D Yu Borin  <https://orcid.org/0000-0003-3842-1487>

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