Research Article

Magnetoresistive and magnetocapacitive effects in magnetic elastomers

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Abstract
Creation of and the following research on systems featuring elastomer filled with a magnetic disperse material with good electroconductive properties have been a continuation of the development of magnetorheological fluids with the goal of finding compositions exhibiting a stronger magnetorheological effect. More profound investigations have revealed that composite materials of the given type also exhibit other significant features such as magnetodeformation, magnetostriction, field-induced shape memory, and piezomagnetoresistance, for which reason they are frequently classified as 'magnetoactive elastomers'. Within the frames of this work, investigations of relationships between the electroconductive and dielectric properties of the polymer composite and external magnetic fields have been done. As has been shown by the experiments, changing the external magnetic field from zero to 330 mT causes the best samples to improve their conductive properties by six orders of magnitude. At the same time, the capacitance measured along with the resistance increases by a factor of 30. Reproducible and less subjected to the emergence of runouts at frequencies of 1 kHz and higher, the capacitance-based data offer hope that such elastomers may be good candidates for being employed as sensors. In order to make the sample-dependent results comparable, the capacitances are interpreted as the dielectric permeabilities. It should be noted though that this approach is strictly formal and the mechanism of the phenomenon observed still awaits its scrupulous study.

Article Highlights

- High-conductive magnetic powders to fill magnetoactive elastomer have been synthesized.
- Magnetoactive elastomer shows a strong relationship between its resistivity and magnetic field.
- At 330 mT, a sample of magnetic elastomer shows a capacitance higher by more than 30 times.

Keywords Magnetic elastomers · Magnetorheological elastomers · Magnetoactive elastomers · Magnetodielectric · Magnetoresistance · Magnetic capacitance · Electrical conductivity

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1 Introduction

Magnetic gels and magnetoactive elastomers (MAE) are solidified equivalents of magnetorheological liquids. Research on them is a prolongation of the development of magnetorheological materials. Such composite systems contain magnetic microparticles dispersed inside the polymer matrix [1, 2]. These materials became of specific interest about a decade ago when they were found to be able to vary their LCR-parameters under the influence of magnetic field. When the fact that the character of many field-dependences is determined by the interior structural re-arrangements suffered by the material [3] was realized, it became evident that introduction of an electroconducting filling powder will result in an elastomer exhibiting magnetoresistance. Significant work was carried out by I.Bika [4, 5]. Later, there were publications describing results on the field-dependences of the LCR-properties of magnetic elastomers [6–10].

Interest expressed towards the synthesis of materials with the magnetodielectric properties is explained by their potential for being employed in such novel devices as tunable filters, magnetic sensors, memory units with four modes, and spin-charge converters [11, 12]. The capability to control the dielectric response gives the advantage of the no-touch tuning of the dielectric permeability variation by means of an applied magnetic field [12].

The work is dedicated to the search for and creation of filling materials to be used in the fabrication of elastomeric composite systems with electrical conductivity, which may be varied by means of the application of external magnetic fields. In light of the fact that these composites feature an elastic polymer impregnated with a disperse magnetic and electroconductive powder, they in fact represent a collection of high-conductivity particles separated by a thick layer of isolator, which results in a high resistivity of the overall material when it is not influenced by magnetic field. At the same time, application of a field induces magnetic moments in the particles, by which it causes attraction among them leading to the formation of chain-like structures. Moving closer to each other, the magnetized particles strain the polymer matrix; meanwhile the degree of the stress caused depends on the geometry of the grains. Thus, the capability of the elastomer to conduct electric current is determined not only by the chemical composition of the filling material, but also by its dispersity and shapes of its particles, and the interior structure of the composite. Introduction of electroconductive powers with various chemical formulae in silicone matrices makes it possible to compare the specific features and intensities of the responses produced by the material to magnetic fields with strengths of up to 330 mT. The establishing of the conditions, under which the abovementioned factors determine the chance to fabricate an elastomer with the desired electroconductive features and the intensities of these factors, is described in the following parts. In particular, the experimental part (2) contains information about the initial ingredients and the sample synthesis technique accompanied by the description of the specimen preparation method for taking measurements (2.1); the schematic of the experimental setup followed by the presentation of the measurement method is brought in 2.2. Part 3 discloses the experimental results on resistivity determination followed by a discussion regarding the structural aspects and the mechanism of magnetoresistance (3.1). The data on the dielectric permeabilities calculated from the capacitances of aniso- and isotropic samples filled with carbonyl iron particles are also given there. Similar results for specimens prepared on the basis of a pure nickel powder and those containing nickel-electroplated carbonyl iron particles and showing the best performance are presented in 3.2 and 3.3, respectively. The mechanisms resulting in certain effects are one more time discussed in the conclusion (4).

2 Experimental

2.1 Synthesis of magnetoactive elastomer

The process of sample fabrication based on polymerizing mixtures of the liquid silicone semi-product with magnetic powders has been described in detail in [13, 14]. The ingredients used in the given series of experiments were the SIEL-256 compound, a product of State Research Institute of Chemical Technologies of Organoelement Compounds,

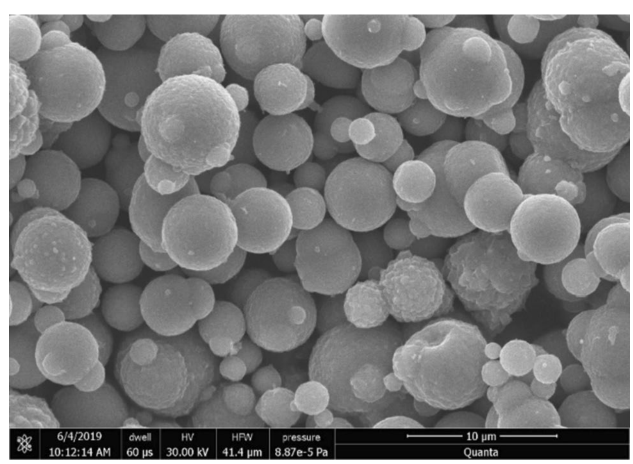

Fig. 1 Photographic image of spherical particles of carbonyl iron

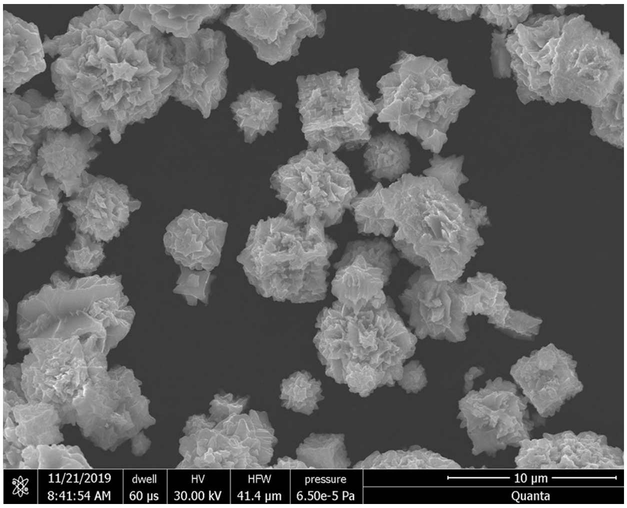

Fig. 2 Photographic image of spherical particles of carbonyl nickel

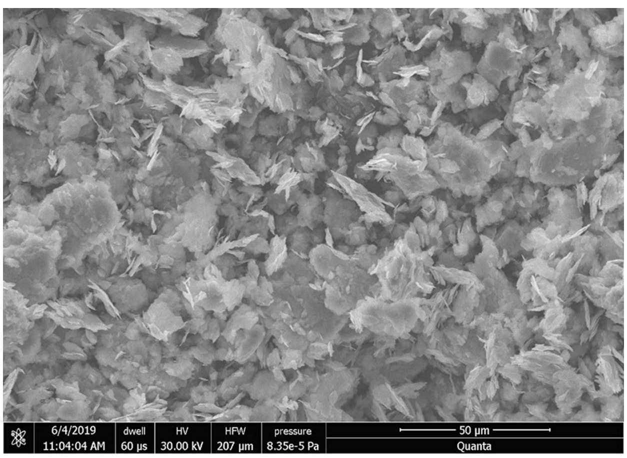

Fig. 3 Photographic image of splinter particles of carbonyl iron obtained by grinding a powder with spherical particles in *n*-heptane followed by electroplating with metal nickel (the concentration of nickel in the pure powder is 2.46 wt%)

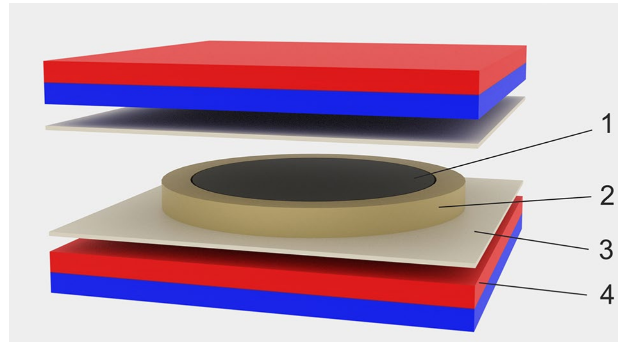

Fig. 4 Fabrication of an MAE sample with anisotropic structure: 1—MAE sample, 2—mold, 3—glass plate, 4—permanent magnet

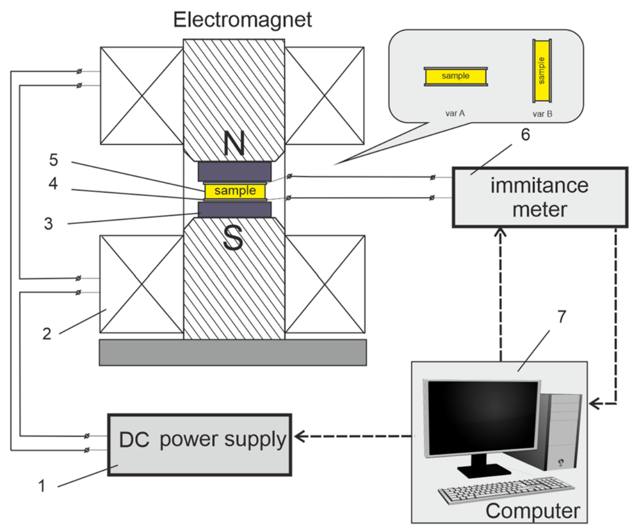

Fig. 5 Schematic of the experimental setup used for measuring the magnetoresistive and magnetodielectric properties of MAE: 1—power supply unit, 2—electromagnet, 3—dielectric spacer, 4—copper plates being the electric contacts, 5—MAE sample, 6—LCR meter, 7—PC supplied with an ADC and DAC

and powders of carbonyl iron (Fig. 1) and nickel (Fig. 2) and a product of grinding spherical iron particles in *n*-heptane with the following nickel-electroplating (Fig. 3).

Samples of magnetoactive elastomer were fabricated in the form of plates with a diameter of 20 mm and a thickness of 2 mm. For that, a liquid mixture of silicone resin and magnetic filler was poured into a mold placed on a glass plate (Fig. 4) to be covered with another glass plate after vacuuming and then put in the oven at 130 °C for 1 h for polymerization. Preparation of samples with anisotropic structure also required sandwiching the assembly in between two permanent magnets to maintain a magnetic field of 100 mT in the sample area. Extraction of the solidified specimen was followed by sandwiching it between two copper plates being the electrical contacts affixed for carrying out LCR measurements (Fig. 5). Featuring a capacitor containing polymer as the isolator connected in parallel with a resistor, the material was tested for the capability to vary its resistance and capacitance with the external magnetic field and frequency of the passing current. The measurements were done using an E7-20 LCR meter, a product of Minsk Research Instrument-making Institute, Belarus. Additional rheological tests performed on an Anton Paar 302 rheometer revealed an elasticity modulus of 40 kPa.

In this work, there were prepared and subjected to measurements the following MAE samples:

(1) 80 wt% of spherical particles of carbonyl iron, size 3–8 μm (Fig. 1), isotropic structure;
(2) 80 wt% of spherical particles of carbonyl iron, size 3–8 μm, anisotropic structure;
(3) 75 wt% of carbonyl nickel star-like particles (Fig. 2), isotropic structure;
(4) 72 wt% of splinter particles of carbonyl iron obtained by grinding a powder with spherical particles in *n*-heptane followed by electroplating with metal nickel (the concentration of nickel in the pure powder is 2.46 wt%) (Fig. 3), isotropic structure.

2.2 Experimental setup

Measurements of the resistance and capacitance to be then re-calculated into resistivity (electroconductivity) and dielectric permeability were carried out using a setup, whose schematic is brought in Fig. 5. The sandwich assembly (the sample) is placed between the poles of the electromagnet and may be positioned in various orientations with respect to the magnetic induction vector; in this series of experiments two possible orientations were used: sample plane perpendicular to the field (variant A) and sample plane parallel with the field (variant B).

The measurements were performed automatically by means of a PC used to set the current feeding the electromagnet to control the strength of the magnetic field. The outputs of the LCR meter were recorded every 4 s. After listing a certain array of parameters, the computer set the next current magnitude. The field-dependent active resistance and capacitance were selected as the descriptive functions for the material studied. On the basis the former the active resistivity was computed, and as has been mentioned above, the capacitance was processed into dielectric permeability according to the formula $\varepsilon = C_0\frac{d}{s\varepsilon_0}$ [6], where *d* and *S* are the thickness and area of the MAE bar.

3 Results

3.1 Sample filled with particles of iron

A series of pilot experiments was carried out on isotropic and anisotropic elastomers containing the 'classic' filler being a powder of carbonyl iron with spherical particles, 3–8 μm in size. The goal was to determine the influence of anisotropy, including the direction of the external magnetic field with respect to the interior orientation, on the performance defined as the decimal logarithm of either the ratio of the resistivity at the maximum field to that at zero field or the ratio of the dielectric permeability at zero-field to that at the maximum field (selected to be 330 mT in the given work). Figure 6a, b depict the graphs of the magnetic field-dependences of the active resistivity and dielectric permeability demonstrated by an isotropic and anisotropic sample containing 80 wt% of carbonyl iron at different frequencies of the passing current.

The obtained results suggest that the influence of magnetic field results in the increasing of the electroconductivity and dielectric permeability of the material. As may be seen, samples with the 'classic' formula exhibit an insignificant magnetoresistive effect of 20–40, whereas the dielectric permeability increases by factors of 1.38 and 2.7 at 1 MHz and 25 Hz, respectively. The stronger outputs are exhibited by samples with anisotropic structure (Fig. 7).

As follows from the results, the resistivity decreases under the influence of magnetic field independently of

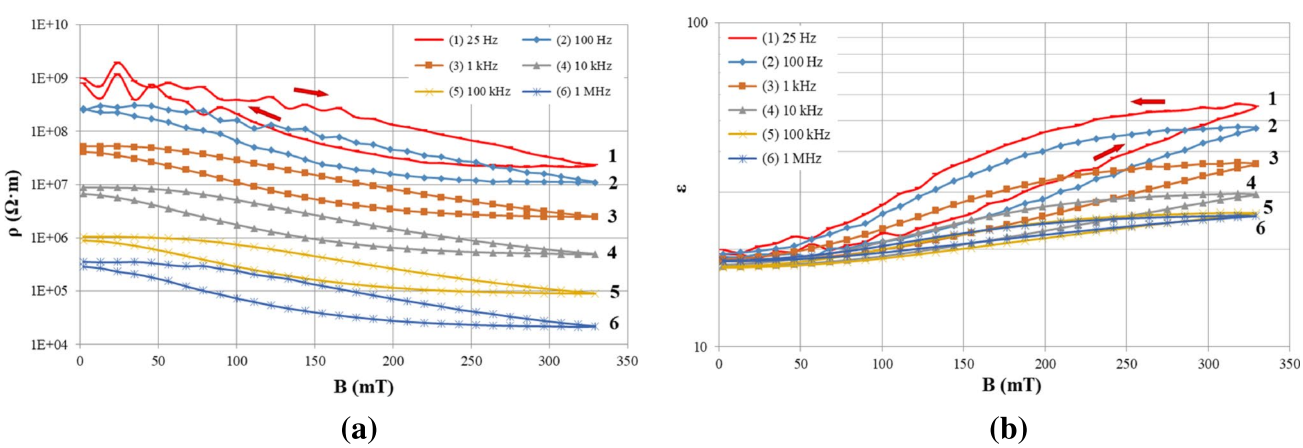

Fig. 6 Resistivity (**a**) and dielectric permeability (**b**) as functions of magnetic field at different frequencies demonstrated by an MAE sample with isotropic structure, containing 80 wt% of spherical particles of carbonyl iron, size 3–8 μm. The way the specified parameters change with the field is shown with arrows

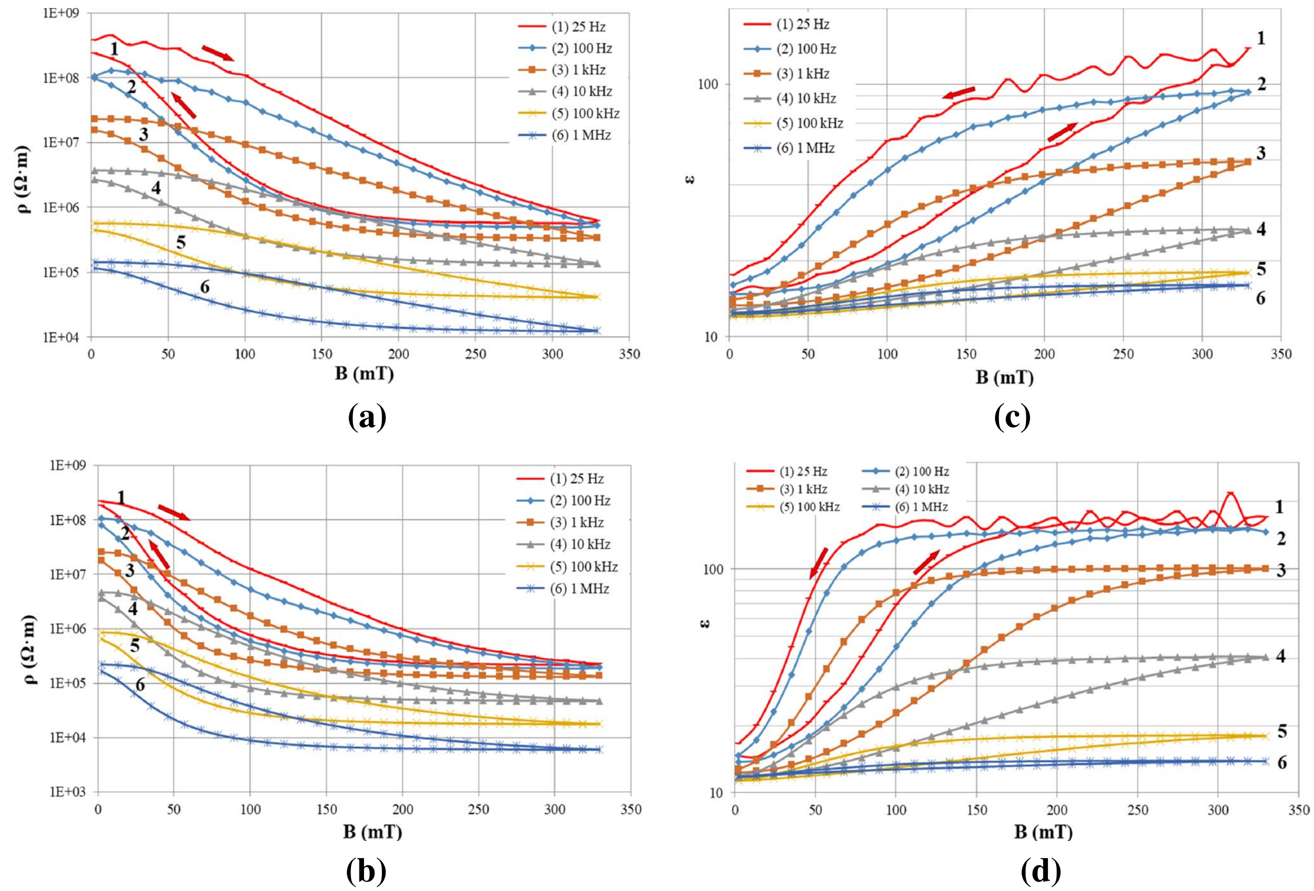

Fig. 7 Resistivity (**a**, **b**) and dielectric permeability (**c**, **d**) as functions of magnetic field at various frequencies exhibited by an MAE sample with interior anisotropy, containing 80 wt% of spherical particles of carbonyl iron, size 3–8 µm. The exterior magnetic field is applied in a parallel (**a**, **c**) (variant A, ref. to Fig. 5) and perpendicular (**b**, **d**) (variant B, ref. to Fig. 5) way to the normal of the sample surface. The way the specified parameters change with the field is shown with arrows

its direction; on the other hand, the dielectric permeability increases. A decrease of the resistivity by three orders of magnitude is also observed when the frequency of passing current changes from 25 Hz to 1 MHz. This phenomenon is evident, because the composite system being considered features a collection of capacitors connected in series and in parallel, and the capacitor impedance drops with frequency. It should be noted that these field-dependences are most pronounced at low frequencies. For instance, a sample influenced by a magnetic field directed parallel to its plates (variant B) exhibits a decrement of 200 kΩ·m or 3200% at 1 MHz, whereas at 25 Hz its resistance drops by 200 MΩ·m or 100,000% (Fig. 7b). Making the direction of the exterior field perpendicular (variant A) to the plates changes the decrements to 140 kΩ·m or 900% and 300 MΩ·m or 50,000%, respectively (Fig. 7a).

The dielectric permeability shows similar tendencies and demonstrates more pronounced increases with magnetic field when the frequency is low. Application of a field directed parallel to the plane of the sample at 1 MHz results in a 16% increment of the parameter—from 12 to 14 units—whereas at 25 Hz the permeability increases by 1100%, namely from 14 to 170 units (Fig. 7d). The increments observed when the field had the perpendicular direction are 15%—from 13 to 15—and 900%—from 15 to 150,—respectively (Fig. 7c). It should be emphasized that the relative magnitudes are somewhat higher when the field is parallel to the plates of the sample.

The fact that the direction of the exterior magnetic field has little effect on the observed phenomena remains partially unclear. According to the currently existing understanding, the given relationships are determined by the structuring processes, in which the particles participate driven by the field along its vector. Application of a field along the normal of the plane of the sample is expected to cause the lining-up of the particles between the plates, thus closing contacts and decreasing the overall resistance. At the same time, turning the sample by 90° to make its plane parallel with the field supposedly has to result in the formation of chains along the contact plates, thus causing a reduction of the capability to conduct electric current. On the other hand, it may be suggested that this phenomenon is based on the high concentrations of magnetic particles, which makes it possible to form structures along all directions including those co-linear with the exterior field.

Such results have been obtained by us previously [6, 9, 10], although then the experimental sample was filled with a magnetically hard NdFeB powder. Comparison of the abovementioned picture with that observed in samples containing a filling material at small or moderate concentrations may help select the correct direction of thinking.

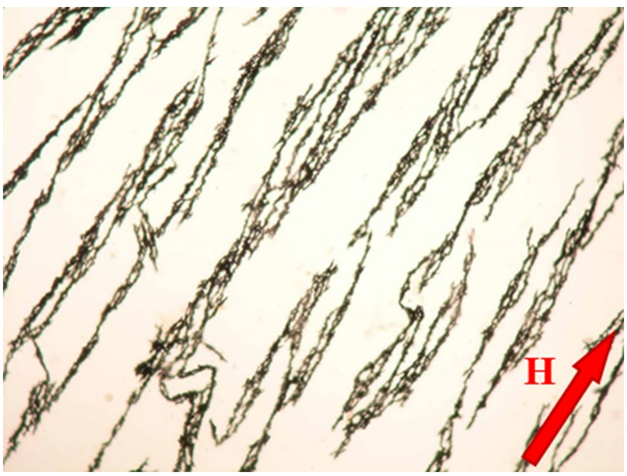

Fig. 8 Interior structure of MAE forming in a magnetic field at a small filler concentration (2–3 wt%)

Fabrication of a sample with anisotropic structure is based on lining the particles up using an exterior magnetic field. The buildups being formed have a pronounced orientation along the force lines. At the same time, they have a non-zero degree of branching and the limbs may have intersections. As may be seen from Fig. 8, at a concentration of 2–3% the branching is rare and the limbs look extended. Figure 9a suggests that the degree of branching increases with concentration, as at 20% intersections are way more multiple. Under the influence of magnetic field, the system forms dense chains along one direction having multiple intersections in perpendicular directions. Such a mechanism of structuring manifests itself as the increasing of the conductivity of the material in all directions independently of how the exterior magnetic field is oriented (Fig. 9b).

There is a chance that a sample with a low concentration of filler may exhibit a more pronounced electroconductivity anisotropy depending on the direction of the exterior field. However, its detection is complex owing to the extremely high resistivity taking place when the amount of conducting particles is insufficient.

It should be noted that the overall variation of electroconductivity caused by the influence of magnetic field is determined by the capability of the magnetized particles to move inside the polymer matrix. Their displacements followed by the formation of chains after the application of a field and the destruction of the buildups with the following migration of the particles to their previous positions when the field is off may be observed in a thin layer of material under the microscope. The details of this process are depicted in this video [15].

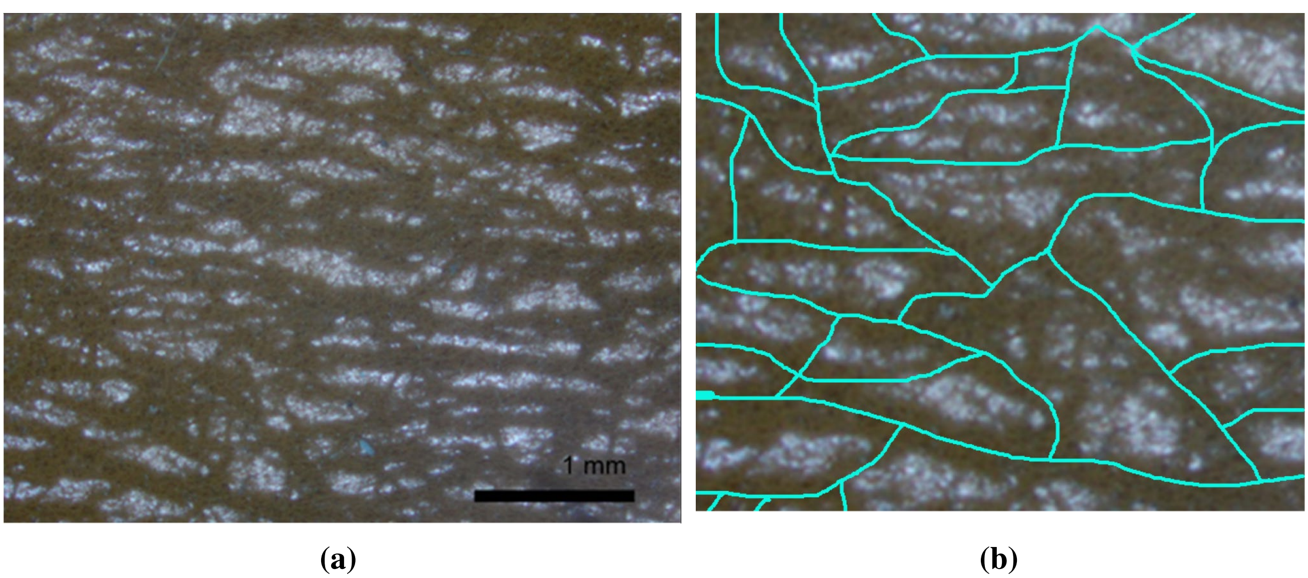

Fig. 9 Interior structure of MAE forming in a magnetic field at a medium filler concentration (20 wt%) (**a**). Formation of possible electrically conductive channels (**b**)

The structuring and destructing processes are characterized by a significant hysteresis, owing to which hysteresis loops are persistently present in all graphs related to electroconductivity. The observed reduction of the capability to conduct electric current follows the corresponding weakening of the exterior field with a noticeable latency. This phenomenon is determined by the fact that whereas the elastic forces of the polymer linearly depend on strain, the attraction force between two magnetized particles is inversely proportional to the fourth power of the distance separating them and rapidly grows when they move closer to each other. Therefore, whereas strong magnetic fields cause particles to move closer to each other, weak fields no longer suffice for keeping them in close contact. Thus, in order to pull them apart, the influence of magnetic field has to be significantly reduced. This mechanism manifests itself through the hysteretic character of the field-dependences of the properties [18].

3.2 Sample filled with particles of nickel

In order to uncover the roles of various magnetic filling materials in the overall magnetoresistive effect, there was conducted an experiment with a sample with isotropic structure filled with a pure powder of carbonyl nickel, which exhibited high electroconductivity. There were recorded (Fig. 10) and studied the dependences of the resistivity and dielectric permeability on magnetic field and frequency $\rho = \rho(B, f)$ and $\varepsilon = \varepsilon(B, f)$, B and f being magnetic induction and frequency, respectively.

As may be seen from the graphs, despite the highest conductivity demonstrated by the pure nickel powder, the sample prepared on its basis demonstrates poor magnetoresistive and magnetodielectric properties. The given phenomenon is explained by the fact that metal nickel possesses moderate magnetic properties resulting in that its particles show a weak tendency to form structures when influenced by a magnetic field. At a frequency of 100 Hz and a field of 330 mT, the magnetoresistive and magnetodielectric effects amount to 130 and 100%, respectively, which are significantly lower in comparison with samples containing carbonyl iron particles.

The experimental material collected during this work has revealed the fact that the highest performance is demonstrated by samples filled with splinter particles obtained by means of grinding carbonyl iron powders with spherical grains in *n*-heptane. That sparked interest in a more detailed study of composite materials with such fillers. Figures 11 and 12 present the results of measurements of the resistivity and dielectric permeability of the corresponding experimental specimens.

3.3 Nickel-containing particles

The understanding existing presently suggests that the performance of magnetoactive elastomer may be improved by means of the introduction of particles with higher electroconductivity. Such filling materials may be obtained by grinding carbonyl iron powders, in pure form or mixed with other metal particles, and by the electrochemical deposition of other metals (nickel in the given series of experiments) onto the surfaces of iron particles.

In order to improve the performance of MAE, a powder of carbonyl iron was dispersed in *n*-heptane (Fig. 3) with

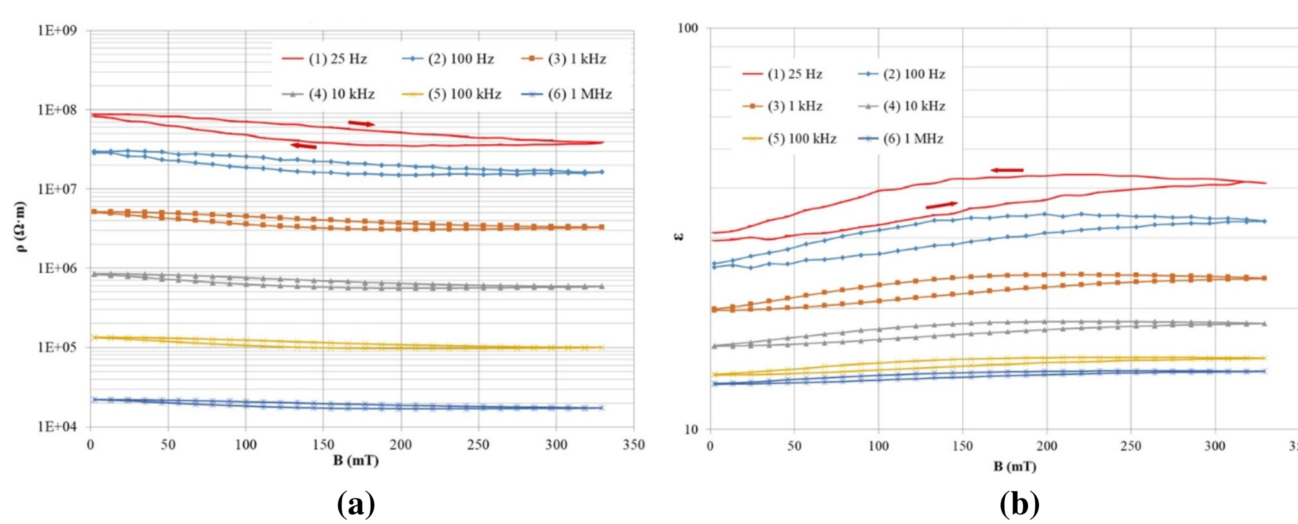

Fig. 10 Resistivity (**a**) and dielectric permeability (**b**) as functions of magnetic field at different frequencies demonstrated by an MAE sample with isotropic structure containing 75 wt% of carbonyl nickel star-like particles. The way the specified parameters change with the field is shown with arrows

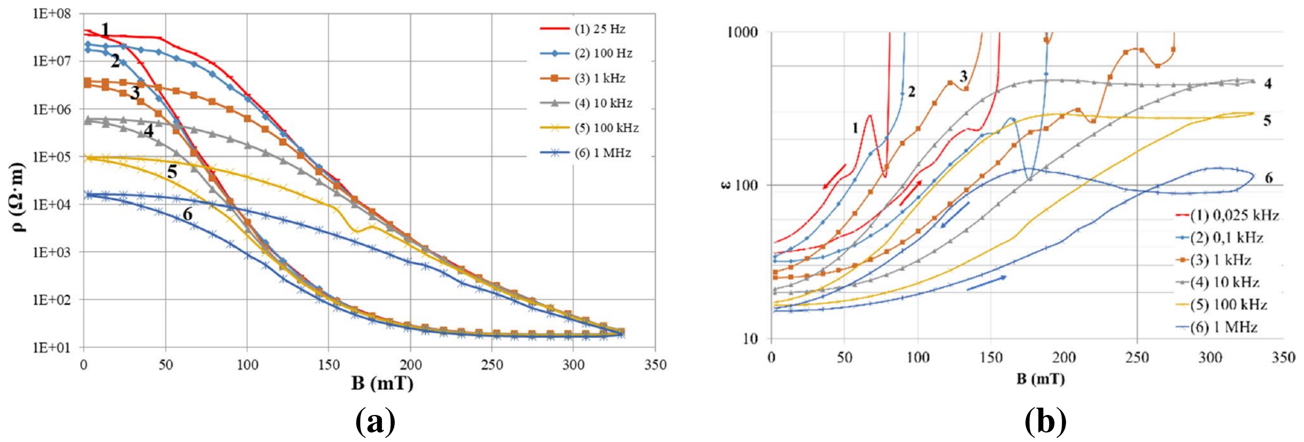

Fig. 11 Resistivity (**a**) and dielectric permeability (**b**) as functions of magnetic field at various frequencies exhibited by an MAE sample with isotropic structure, containing 72 wt% of splinter particles of carbonyl iron obtained by grinding a powder with spherical particles in *n*-heptane followed by electroplating with metal nickel (the concentration of nickel in the pure powder is 2.46 wt%). The way the specified parameters change with the field is shown with arrows

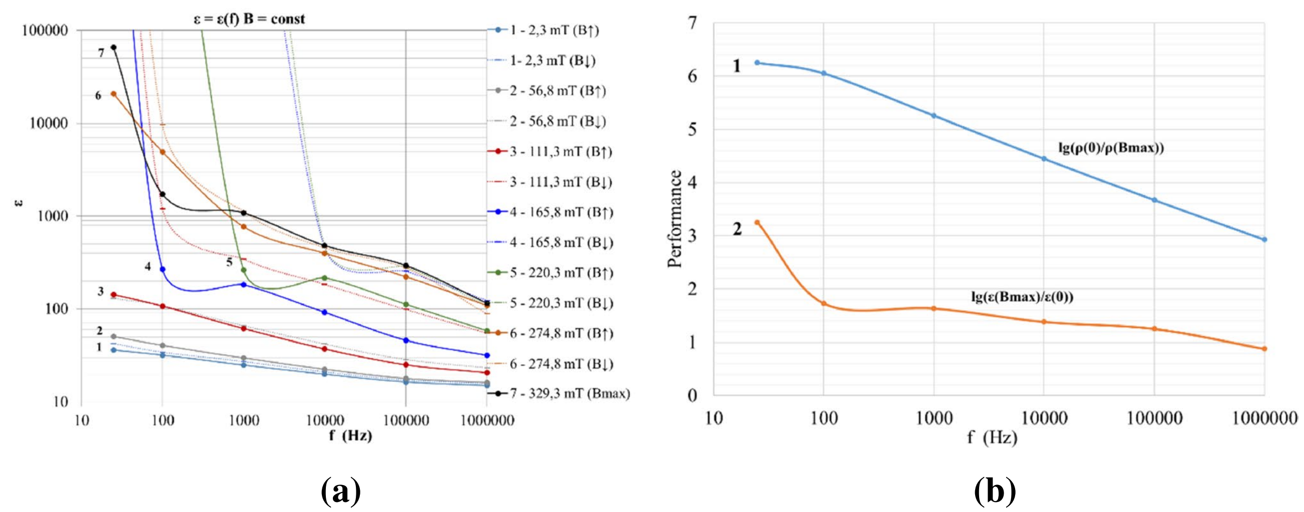

Fig. 12 Performance, B_{max} being 330 mT (**a**) and dielectric permeability at various magnetic induction magnitudes while the field increases ('↑', solid lines) and decreases ('↓', punctured lines) (**b**) as functions of the frequency of the passing current demonstrated by an MAE sample with isotropic structure, containing 72 wt% of iron particles obtained by the grinding of a carbonyl iron powder with spherical particles in *n*-heptane followed by nickel-electroplating (concentration of nickel in the pure powder—2.46 wt%)

its following electroplating with metal nickel [16]. Dispersion in heptane conditions the change of particle shape from spherical to splinter, which to some degree may be approximated to irregular ellipsoidal. This results in the intensifying of magnetic interactions among the particles thus promoting the formation of structures. A similar tendency was observed previously in magnetorheological experiments when samples filled with permalloy splinter particles showed the best performance [17].

The relationship between magnetic field strength and the resistivity of material containing 72 wt% of carbonyl iron particles, previously subjected to grinding in *n*-heptane followed by nickel-electroplating, is shown in Fig. 11a. A simple comparison with the behavior of samples filled with pure carbonyl iron makes it possible to see that the presence of nickel intensifies the field-dependence, as the performance of such specimens is higher. Indeed, increasing the exterior magnetic field from zero to 330 mT results in a resistivity reduction by 1×10^3 and 2×10^6 times at 1 MHz and 25 Hz, respectively (Fig. 11 a)). At the same time, the magnetic field-dependence of the dielectric permeability is also strong (Fig. 11b). It should be noted though, that significant reduction of the resistance of a sample makes the measurements at low frequencies difficult, as the output signal of the impedance meter becomes unstable resulting in inadequate indications.

An analysis of how the performance demonstrated by the sample at 330 mT depends on frequency is presented in Fig. 12. The illustrations suggest that the strongest sensitivity for magnetic field exhibited by the material is observed in the low-frequency range. Indeed, the most intensive increase of the resistivity and dielectric permeability, namely by factors of 2×10^6 and 150, respectively, occurs at 25 Hz, the lowest frequency value allowed by the equipment (Fig. 12a). (The runs of lines 1 and 2 were determined on the basis of the experimental data brought in Fig. 11). A more detailed diagram depicting the frequency-dependences of the dielectric permeability at different external field strengths (Fig. 12b) shows a set of converging lines, by which also confirming the assumption regarding the functioning condition.

4 Conclusions

An important achievement of the investigations done within the frames of this work is the successful preparation of samples of magnetoactive elastomer exhibiting strong field-dependences of their resistivities and dielectric permeabilities. The best performance is demonstrated by the sample filled with splinter iron particles obtained by grinding carbonyl iron powders with spherical grains in a planetary mill with following nickel-electroplating. The variation of parameters shown by the material in magnetic field may be explained by the reversible structuring processes, which the particles are engaged in. When magnetizable particles approach each other the local magnetization field is increased and the mutual attraction force is so to say self-enhancing. Upon reduction of the external magnetic field the locally induced magnetization is still enhanced compared to the state where the particles were separated. Only as the external field is further reduced also the induced magnetization becomes small enough for the elastic restoring force to overcome the magnetic force. The effect of enhanced magnetization and magnetic forces upon magnetizable particle approaching each other is described in detail in [18].

The novelty of the study is the discovery that the relationships between the resistivity or dielectric permeability with magnetic field remain practically indifferent to the orientation of the sample with respect to the magnetic induction vector. This phenomenon may be explained by the fact that at high filler concentrations the chain-like structures demonstrate a high branching degree, which promotes high electroconductivity along all directions. It disproves our assumption, according to which the capability of a sample to conduct electric current decreases when a magnetic field is directed parallel with its plates.

Along with that, a sample capable of reducing its resistivity by six orders of magnitude in a magnetic field of 330 mT has been synthesized. At the same time, we applied a formal approach based on interpreting observed capacitance variations as dielectric permeability changes. Despite the fact that the mechanisms of this phenomenon remain unknown, as the case considered does not assume observation of spin-dipole coupling described in [19, 20] and presents only an example of interior restructuring occurring within the bulk of magnetic composite, it is worth mentioning that in similar experiments carried out at previous works, the recorded variations of the 'effective' dielectric permeability did not exceed a factor of 10 [9].

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Author contributions All authors read and approved the final manuscript.

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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