

Novel highly elastic magnetic materials for dampers and seals: Part I. Preparation and characterization of the elastic materials

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The new generation of magnetic elastomers represents a new type of composites, consisting of small (mainly nano and micron-sized) magnetic particles dispersed in a highly elastic polymeric matrix. The combination of polymers with magnetic materials displays novel and often enhanced properties. Highly elastic magnetic composites are quite new and understanding of the behavior of these materials depending on the composition, external conditions, and the synthesis processes is still missing. Thus, the aim of this work is the study of fundamental principles governing the preparation of these materials as well as their structure and elastic properties. Copyright © 2007 John Wiley & Sons, Ltd.

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INTRODUCTION

Materials whose mechanical and rheological properties can be varied by the application of external magnetic field can be divided into three major groups: solid hard bodies, magnetic fluids or suspensions, and magnetic elastomers.

The change in the length of a sample upon magnetization is a common phenomenon in several materials and this phenomenon is called linear magnetostriction. Most of the magnetostrictive materials are solids, which develop mechanical deformation when subjected to an external magnetic field. This phenomenon is attributed to the rotation of small magnetic domains in the solid body.

The magnetostriction phenomenon is evidenced in practice as a small change of geometry of the solid body when it is subjected to a magnetic field. In a material exhibiting magnetostriction the dimension along the magnetization is elongated, while the dimension perpendicular to it is shortened, keeping the volume constant. For

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monocrystalline materials the magnetostriction is highly anisotropic and even the sign of magnetostriction may vary for different crystallographic directions.

Until recently the observed magnetostrictive deformations were quite small. For iron, nickel, and cobalt the strain is of the order of 10^{-5} . The largest magnetostriction has been found in the rare-earth metals and compounds. Terbiumdysprosium-iron alloys offer strains up to 0.002. Although the magnetostrictive size changes are small, they are driven by strong interatomic forces, therefore traditional magnetostrictive materials find application where very strong forces, but not large movements are required.

Another class of materials whose mechanical (rheological) properties can be varied by the application of magnetic field is referred to as magnetorheological liquids and suspensions. These colloidal solutions contain small, dispersed particles in the size range from nanometers to micrometers. In the magnetorheological fluids the size of the magnetizable particles ranges typically from 0.01 to 100 µm. The controllable rheological response results from the interactions between the suspended particles. By the application of external magnetic field the induced magnetic dipoles cause the particles to form chain-like structures parallel to the applied field. Depending on the particle concentration, columnar structures may develop and restrict the flow of the



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liquid. The mechanical energy needed to yield these ordered structures strongly depends on the magnetic field intensity. As a result, magnetic-field-sensitive liquids have field dependent yield stress and viscosity. The most common magnetorheological liquids exhibit dynamic yield strengths in excess of 50 kPa when the magnetic field of 150– 250 kAm^{-1} is applied.¹

The rigid magnetic elastomers, i.e. composite materials consisting of rather rigid polymeric matrices filled with magnetic particles are long time known.² These materials are successfully used as permanent magnets, magnetic cores, and connecting and fixing elements in many areas. These traditional magnetic elastomers have low flexibility and practically do not change their size, shape, and elastic properties in the presence of external magnetic field.

The new generation of magnetic elastomers^{3,4} represents a new type of composites consisting of small magnetic particles, usually in the nano- or micrometer range, dispersed in a highly elastic polymeric matrix. Combination of magnetic and elastic properties leads to a number of striking phenomena that are exhibited in response to magnetic fields. Since the magnetic fields are convenient stimuli from the point of signal control, therefore it is of great importance to develop and study such flexible polymeric systems.

The appearance of the unique ability of such materials to change their size and mechanical properties in a reversible manner has inherent interest if for no other reason than the uniqueness of having a giant elastic response to magnetic polarization. Giant deformational effect, high elasticity, and quick response to magnetic field open new opportunities for using such materials for various applications.

Magnetoelastics have been gaining importance only in the last few years. Several groups have already started research in this area.^{4–20} The Ford Motor Company has patented an automotive bushing employing a magnetorheological elastomer.²¹ The controlled stiffness of the bushing is used to reduce suspension deflection.

Highly elastic magnetic composites are quite new and understanding of the behavior of these materials depending on the composition, external conditions, and the synthesis processes is still missing. Thus, one of the aims of this work is the study of fundamental principles governing the preparation of these materials as well as their structure and properties. We synthesize two types of magnetic composites and study their structural, magnetic and mechanical properties in external homogeneous magnetic fields. Some preliminary studies of possible technological applications of these materials are presented in the subsequent paper of this series.

EXPERIMENTAL

Synthesis of composite materials

Type 1 materials

In order to prepare magnetic field responsive PDMS composites (magnetoelasts), carbonyl iron (BASF), Fe₃O₄ (Bayern), Fe, Fe₃O₄·Fe, and γ Fe₂O₃ particles (prepared by the Institute of Chemistry and Technology of Organoelement Compounds (GNIIChTEOS), Russia) were used as magnetic



particles. The concentration of the filler particles was varied between 10 and 30 wt% in the polymer matrix. Poly(dimethyl siloxane) networks were prepared from a commercial product of a two-component reagent (Elastosil 604 A and Elastosil 604 B) provided by Wacker Co. These chemicals were used without further purification. Component A contains the polymers and the catalyst with Pt content and component B provides the cross-linking agent. The magnetite particles were dispersed in the Elastosil 604 A. After mixing it up with the Elastosil 604 B component, the solution was transferred into a cube-shaped mould.

It has to be mentioned that it was not possible to prepare PDMS magnetoelasts with the same cross-linking contents which were prepared from carbonyl iron (BASF), Fe₃O₄ (Bayern) in case of the magnetic powder prepared by GNIIChTEOS. The Fe₃O₄·Fe and γ Fe₂O₃ magnetic powder retarded the cross-linking reaction. For this reason 20 and 3.5 wt% component B was used, respectively. Because of the retardation from the Fe₃O₄·Fe powder only 10 wt% could be used for the preparation with 20 wt% B component. The settlement of the magnetic particle was strong in these samples. In the case of iron particles 3 wt% B component was used for the preparation.

Type 2 materials

Second type of materials is based on polymer matrices of compounds "SIEL" produced by GNIIChTEOS. Standard compound "SIEL" consists of two components A and B. The component A is the mixture of low-molecular vinyl-containing rubber (VR) and a hydride-containing cross-linking agent. The component B is prepared from VR and a complex platinum catalyst.

Magnetic filler was the powder of iron particles with the average size of $2-4 \,\mu$ m. To prevent particle aggregation and to enhance their compatibility with the polymeric matrix magnetic powders have been preliminary processed by hydride-containing silicone. As a result some moisture from particle surface was removed and the surface became more hydrophobic.

Processed magnetic particles were further dispersed in the compound SIEL. Composition polymerization was performed at 100–150°C at additional effect of electromagnetic field SHF with the frequency of 2.4 GHz.

Materials of the type 2 have much lower elastic modulus than the materials of the type 2.

In order to investigate the effect of uniform magnetic field on the elastic modulus of the sample, isotropic and anisotropic elastomers were prepared. The synthesis of the elastomers in the uniform magnetic field can be used to prepare anisotropic samples. If the polymerization reaction proceeds not too fast, then there is enough time to induce the pearl chain structure of the filler particles by applying uniform external field before the reaction is completed. The experimental setup is shown in Fig. 1. The uniform magnetic field was 400 mT. The formation of chain-like structure takes a few minutes and as a result, aligned particle aggregates parallel to the field direction are fixed in the network. Depending on the concentration of the magnetic particles, as well as on the value of applied magnetic field, the columnar structures of the magnetic particles built in the elastic matrix



Poles of the magnet

Figure 1. Preparation of uniaxially ordered polymer composite under uniform magnetic field.

can be varied in wide range. The understanding of the magneto-mechanical properties of magnetoelasts should assist the development of new type of dampers where the magnetoelastic response can be actively controlled in real time.

RESULTS

Optical and magnetooptical studies

Figure 2 shows different structures of magnetic particles formed in thin magnetoelastic layers (30–40 μ m) after their polymerization in the magnetic field. The photos were made with the help of optical microscope. As we can see from this figure, depending on concentration, the magnetic particles (with a size of 2 μ m) can form chains stretched along the field lines (Fig. 2a) or more complicated structures that are steady in this magnetic field. For large concentration of magnetic particles we have short chains as presented in Fig. 2(b) and as a result chains form the "net" structure. Figure 2(c) shows that for high concentration of magnetic particles they are densely packed. Figure 2(a)–2(c) has 60× amplification. Novel highly elastic magnetic materials for dampers and seals I

Figure 2(d) shows that even for large concentrations the particles form well-defined structure, oriented along field lines and composed of separate agglomerates. This figure has $600 \times$ amplification and one can see separate magnetic particles.

Optical properties and homogeneity of magnetoelastic films have been previously studied by us by the measurements of their transparency.²² The transparency spectra of magnetic composites have been compared with those of the polymer matrices without magnetic particles and those of magnetic fluids with homogeneous particle distribution. It has been shown that the spectra of transparency of magnetic composites are close to those of the polymer matrices without magnetic particles. Thus, (i) there exist some transparent polymeric areas without magnetic particles within the material giving the basic contribution to the spectral dependence of their transparency, and (ii) the distribution of magnetic particles in the polymeric matrices synthesized is non-uniform field (in comparison with magnetic fluids).

Magnetic properties

The magnetic properties of composites were studied by the magnetometer in the alternating magnetic field of 5 kOe. It has been shown that they are mainly determined by the magnetic properties of the magnetic powder fillers and their volume fraction in materials. As an example in Fig. 3 we present the loops of hysteresis for the iron magnetic powders (the size of the particles 300 A, Fig. 3a and 2 μ m, Fig. 3b) and the magnetite powders (the size of particles is 0.2 μ m, Fig. 1c) as well as for magnetic composites synthesized on the basis of the corresponding powders, Fig. 3a^{*}-c^{*}.

Iron particles of 300 A and $2 \mu m$ (Fig. 3a, b, respectively) have different values of the residual magnetization and the coercive force because of their different domain structures and different mechanisms of re-magnetization.

One can see from Fig. 3 that the general character of the loops is the same for the magnetic powders and composites.



Figure 2. Structures of magnetic particles in magnetoelastic material. Iron magnetic powder content in 5 vol% (a), 12 vol% (b,d), 25 vol% (c).





Figure 3. The loops of hysteresis for the iron magnetic powders (the size of the particles 300 A (a) and 2 μ m (b)) and the magnetite powders (the size of particles is 0.2 μ m, (c)) as well as for magnetic composites synthesized on the basis of the corresponding powders (a^{*}-c^{*}).

In particular, magnetoelastic samples on the basis of magnetic particles with more pronounced hysteresis also have more pronounced hysteresis and vice versa. However, the coercive force of the composites is smaller than that of magnetic powders because of the change in the concentration of the magnetic particles in the polymer matrices. Besides, the value of the saturation magnetization is defined by the volume fraction of magnetic particles in the material.

Magnetostriction of composite materials

The phenomenon of giant magnetostriction of magnetic composites is observed. Samples of materials of type 2 placed in a homogeneous magnetic field are extended along the lines of the magnetic field. The elongation of the samples depends on the intensity of the magnetic field. As an example the dependence of the relative deformation on the magnetic field for the sample containing 35 vol% of iron magnetic powder is shown in Fig. 4. The lower curve corresponds to increasing magnetic field while the upper curve is measured at decreasing H. One can see that the obtained dependence is characterized by a significant hysteresis.

Memory effects

An applied mechanical force can change the shape of magnetic elastomers but samples usually restore initial shape when the mechanical force is switched off. However in the case, when the magnetoelastic sample is very soft and concentration of magnetic particles is rather high, a new effect has been observed. Namely, stretched or compressed samples of type 2 can maintain their new shape in the magnetic field after switching off the mechanical force and restore their initial shape only when the magnetic field is switched off. This new effect of plasticity in external magnetic fields or the shape memory effect is schematically demonstrated in Fig. 5. The arrows in this figure show the direction of an external magnetic field. In Fig. 5A an initial undeformed sample in the absence of magnetic field is shown. Then, the magnetic field is switched on and the sample is slightly elongated along the lines of the field (Fig. 5B). Figure 5(C)-5(F) shows various deformations of the sample in magnetic field. They just demonstrate that one can deform the sample in the magnetic field in various ways and the sample maintains various new shapes. In the presence of the magnetic field deformation of the sample after complete stress release remains practically unchanged with time. After



Figure 4. Deformation of composites under the influence of a homogeneous magnetic field.



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Figure 5. Schematic representation of pseudo-plasticity or memory effect of magnetic composites in an external magnetic field.

switching off the magnetic field (Fig. 5G) the deformation totally disappears and the sample adopts the initial shape (Fig. 5A).

This memory effect may be explained as follows. Under applied homogeneous magnetic field the magnetic particles in composites tend to align into chains. As a result, internal deformations of the polymer matrix between magnetic particles increase. The filler particles change their initial positions with respect to the state without magnetic field and each of them falls into an equilibrium state under influence of dipole-dipole forces and internal stresses of the polymer matrix. The whole sample of the composite appears in one of the local energy minimum of the system. Every time an applied external mechanical force breaks initial chains of magnetic particles in the magnetic field but new chains are formed and each filler particle falls into a new equilibrium state. Due to dipole-dipole interactions of magnetic particles the sample maintains its new shape in the magnetic field and the whole system appears in a new local energy minimum of the system. If we reduce the external mechanical force to zero without changing the magnetic field we observe some residual strain of the sample. Only when the magnetic field is switched off, the forces of dipole-dipole interaction decrease and the initial shape of the sample is restored due to elastic forces of polymer matrix.

Magnetic elastomers in uniform field, a theoretical approach

The nominal stress, $\sigma_{\rm el}$ can be calculated by a standard method: 23,24

$$\sigma_{\rm el} = \left(\frac{\partial W_{\rm el}}{\partial \lambda_x}\right) \tag{1}$$

and the stress–strain dependence of unidirectional deformation of an ideal polymer network can be described by neo-Hookean stress–strain relation:^{23,24}

$$\sigma_{\rm el} = G(\lambda_x - \lambda_x^{-2}) = G_0 D \tag{2}$$

where $D = \lambda_x - \lambda_x^{-2}$. It follows from the above equation that the elastic modulus can be defined as follows:

$$G_0 = \frac{1}{3} \lim_{\lambda_x \to 1} \left(\frac{\partial \sigma_{\rm el}}{\partial \lambda_x} \right) \tag{3}$$

It is worth mentioning that in the small strain regime one can approximate the experimental data by Hooke's law with Young modulus of E = 3G.

Comparing eqns 1 and 3, one can express the elastic modulus by the following alternative expression:

$$G_0 = \frac{1}{3} \lim_{\lambda_x \to 1} \left(\frac{\partial^2 W_{\rm el}}{\partial \lambda_x^2} \right) \tag{4}$$

In the case of a magnetoelast the overall energy density is the sum of magnetic, $W_{\rm M}(H_{\rm eff})$, and elastic, $W_{\rm el}(\lambda_x)$, energy contributions:

$$W(\lambda_x, H_{\text{eff}}) = W_{\text{el}}(\lambda_x) + W_{\text{M}}(H_{\text{eff}})$$
(5)

where H_{eff} stands for the effective magnetic field strength. The magnetic energy density, W_{M} of a piece of magnetoelast is related to the effective field:

$$W_{\rm M} = \mu_0 M(H_{\rm eff}) \cdot H_{\rm eff} \tag{6}$$

where the function $M(H_{\text{eff}})$ describes the magnetization curve and μ_0 denotes the magnetic permeability of the vacuum.

Deformation of a magnetoelast involves a change in the particle arrangement, which affects the magnetic energy density, $W_M(H_{eff})$. As a consequence, the deformation of magnetoelast requires more energy in an uniform magnetic field since one must overcome both the change in the elastic, and the magnetic energy as well. In practice, this magnetic effect manifests itself as an increase in the elastic modulus.

On the basis of eqns 4 and 5 the elastic modulus measured under uniform external field can be expressed as the sum of two contributions:

$$G = G_0 + G_{\rm M}^{\rm E} \tag{7}$$

where $G_{\rm M}^{\rm E}$ denotes the magnetically induced excess modulus. We can characterize the magnetic field intensity by the magnetic induction, *B* which is the product of the magnetic permeability of the vacuum and the field intensity: $B = \mu_0 H_{\rm eff}$. The magnetic contribution of the modulus can be calculated from the change of magnetic energy due to the distortion of the composite. Similar to eqn. 3 the excess modulus can be defined as follows:

$$G_{\rm M}^{\rm E} = \frac{1}{3} \lim_{\lambda_x \to 1} \left(\frac{\partial^2 W_{\rm M}}{\partial \lambda_x^2} \right) \tag{8}$$





Figure 6. Five possibilities to determine the elastic modulus of the anisotropic magnetoelasts. White arrows indicate the direction of the force; black arrows show the direction of magnetic field.

Since the magnetic polarization is a fast process, $G_{\rm M}^{\rm E}$ develops quickly when the external field is turned on and disappears immediately when the external field is turned off. The analytic calculation of the magnetic excess modulus, $G_{\rm M}^{\rm E}$, is a rather complicated task due to the fact that the magnetic energy density W(H_{eff}) cannot be given in an analytical form.

Mechanical measurements under uniform magnetic field. Comparison of mechanical behavior of homogeneous and anisotropic composites in magnetic field

The elastic modulus of magnetoelasts of type 1 was measured under uniform magnetic field at 293 K using two different homemade apparatus. The magnetic induction, *B* was measured by a Phywe Teslameter. Comparing the direction of the force and the magnetic field there are two different situations: the force and the magnetic field are either perpendicular or parallel.

If the magnetoelast contains magnetic particles dispersed randomly there are two basic experimental situations: The compressive force (F_x) and the direction of magnetic field (characterized by the magnetic induction, *B*) can be either parallel or perpendicular.

The elastic modulus of the anisotropic magnetoelasts can be determined using five different experimental setups (as shown in Fig. 6.), depending on the direction of the magnetic field, the columnar structure and the mechanical stress.

Mechanical measurements under uniform magnetic field.

Isotropic mechanical behavior

Depending on the direction of the field to the mechanical stress, two kinds of experimental arrangements were used: parallel and perpendicular (See Fig. 7). One can see in both figures that there is a slight increase in the modulus due to the external field. It is also seen in the figures that the increase in the concentration of the filler particles leads to the increase in the field free modulus, G_0 . Larger amount of Fe particles results in larger elastic modulus.

We have performed systematic studies of the magnetic reinforcement effects. All of the experimental arrangements shown in Fig. 6 were investigated. The magnetic field intensity was varied and the elastic modulus was measured as a function of magnetic induction, *B*. The effect of the concentration of magnetic particles has been investigated. It has been shown that the elastic modulus increases with an increasing magnetic filler content.

Anisotropic mechanical behavior

Compressive force is perpendicular to the direction of particle chains

The effect of the uniform magnetic field on the elastic modulus was studied when the applied mechanical stress was perpendicular to the particle alignment. The cross-



Figure 7. Dependence of the elastic modulus on the magnetic field intensity for magnetoelasts containing different amount of Fe particles. The concentrations of the filler particles are indicated in the figure. The cross-linking densities of the PDMS samples are the same (the concentration of component B equals to 3 wt%).





Figure 8. Effect of the magnetic field intensity on the elastic modulus. The crosslinker content is 3.0 wt% in every case. The iron content of the elastomers is indicated in the figure. The white and black arrows show the direction of the force and the uniform magnetic field, respectively.

linking density was constant, the concentration of the magnetic particles was varied. By varying the direction of the applied magnetic field and the columnar structure of the particles, we have three possible experimental arrangements: the direction of the field is perpendicular to the particle chains (Fig. 8a, b) and parallel to the particle chains (Fig. 8c).

On the basis of the experimental results shown in Fig. 8 it may be concluded that the spatial orientation of the force, the field and the particle arrangement play a decisive role in the temporary reinforcement effect. A weak effect has been found when the field is perpendicular to the particles alignment (Fig. 8a, b).

If the columnar arrangements of the particles are parallel to the direction of the magnetic field, the elastic modulus increases significantly (Fig. 8c). At small field intensities of up to 100 mT the modulus increases significantly. At higher field intensities (from 100 mT) the elastic modulus tends to level off.

It is also seen that by increasing the concentration of the iron particles in the polymer matrix the elastic modulus, G_{0} , also increases.

Compressive force is parallel to the direction of particle chains

Figure 9 shows the reinforcement effect when the direction of the applied mechanical stress is parallel to the particle



Figure 9. Dependence of the elastic modulus on the magnetic field. The arrangements of the particles in the polymer networks are parallel to the applied mechanical stress while the applied uniform magnetic field is parallel (a) or perpendicular (b) to the columnar structure. The cross-linker content is 3.5 wt%. The concentration of the carbonyl iron particles in the polymer matrix is indicated in the figure.

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alignment. In this case there are two different experimental setups. The direction of the magnetic field is parallel or perpendicular to the direction of the columnar structure as seen in Fig. 9. It was found that when the applied uniform magnetic field is perpendicular to the direction of the particle chains (Fig. 9b), the magnetic field does not influence the elastic modulus. The strongest magnetic reinforcement effect was found when the applied uniform magnetic field is parallel to the particle alignment and to the mechanical stress (Fig. 9a). In this case the relative increment of the modulus has the highest value.

When the iron content is increased, the mutual particle interaction increases, as well as the elastic modulus, as it is predicted.

If the arrangements of the particles are parallel to the direction of the uniform magnetic field and to the applied force (Fig. 9a), the elastic modulus of the elastomers increases significantly with the magnetic field intensity. The increment in the modulus can be approximated by a quadratic relation with the field intensity.

For magnetic elastomers of type 2 a giant increase in the elastic modulus in the external homogeneous magnetic field



Figure 10. Dependence of elasticity modulus *G* for magnetic elastomers of type 2 with different volume contents of magnetic powder.

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perpendicular to the compression force has been observed even for isotropic samples. In contrast to the materials of type 1, this increase was two orders of magnitude (see Fig. 10) and it is higher for materials with higher powder content. We believe that such a considerable increase in the modulus in the magnetic field is connected with the fact that the initial elastic modulus of the samples of type 2 was very small (several kPa), much smaller than that of the samples of type 1. In this case, particles can easier move under the influence of the field to form magnetic structures of denser chains.

DISCUSSION

The main objective of the paper is the development of the new highly elastic magnetic field controlled materials and prospective for the use in devices on the basis of these materials. The novel feature of these materials is the ability to undergo quick and controllable large-scale deformations and essential changes in elastic and viscous properties in magnetic field. These peculiar magnetoelastic properties can be used to create high-reliability seals with enhanced properties in comparison with existing analogs and tuneable vibration absorbers.

Samples of highly elastic magnetic composites with homogeneous distribution of magnetic particles have been synthesized. It has been shown that the magnetic properties of these materials are similar to those of magnetic powders that are fillers of composites. The viscoelastic properties of the synthesized materials have been studied under compression. It has been shown that viscoelastic properties are strongly dependent on the external magnetic field, namely, two orders increase of the elastic modulus of the materials has been observed. The "memory effect" (large residual deformations of material in a magnetic field) has been found.

The synthesis of composites in an external magnetic field has been carried out. In the uniform magnetic field the solid particles form chain-like structure and this structure becomes fixed in the course of polymerization reaction. It has been shown that the resulting magnetic elastomers are anisotropic in terms of mechanical properties.

The analytic calculation of the magnetic modulus is a rather complicated task due to the fact that the demagnetizing coefficient cannot be given in an analytic form and it depends on the spatial distribution of the magnetic particles in the elastic matrix. The analytic calculation of the magnetic modulus and damping possibilities of the magnetoelasts will be the topic of the forthcoming paper.



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