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# Polymer matrix composite materials reinforced by $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ magnetostrictive particles

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**Materials**

## ABSTRACT

**Purpose:** The goal of this work was to describe manufacturing process of polymer matrix composite materials reinforced by $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles and to observe changes of physical properties (magnetic properties and magnetostriction) of samples with randomly oriented magnetostrictive particles in epoxy matrix and with aligning these particles in the matrix during fabrication process.

**Design/methodology/approach:** Polymer matrix composite materials reinforced by the $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ magnetostrictive particles fabricating method was developed during the investigations, making it possible to obtain materials with good physical properties. The influence of the concentration of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles on magnetic and magnetostrictive properties was estimated. Metallographic examination of powder's morphology as well as EDS and XRD analysis and observations the structure of composite materials were made.

**Findings:** The influence of magnetic particle alignment is observed in the magnetic and magnetostriction responses. The magnetostrictive response improves when the magnetic particles are oriented in magnetic fields and reaches approximately 184 ppm for oriented composite materials with 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles.

**Practical implications:** For potential applications in technological devices, such as sensors and actuators, it is desirable to form composite systems by combining magnetostrictive phases with matrix, in order to have giant magnetostrictive effect and, at the same time, to reduce disadvantages of monolithic material.

**Originality/value:** The originality of this work is based on manufacturing process, especially of applying magnetic alignment for ordering $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles during polymerization of epoxy matrix.

**Keywords:** $Td_{0.3}Dy_{0.7}Fe_{1.9}$; Magnetostrictive composite materials; Magnetic properties; Magnetostriction; Smart materials

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## 1. Introduction

Magnetostrictive materials are this class of materials that couple a magnetic field to mechanical motion. A highly magnetostrictive alloy of terbium, dysprosium and iron – $Td_{0.3}Dy_{0.7}Fe_{1.9}$ (commercially known as Terfenol-D) was discovered at the Naval Surface Warfare Centre (NSWC) in the 1980’s. Since then the hhh phase alloy $Td_{0.3}Dy_{0.7}Fe_{1.9}$ attracts many investigations [1-4] so it is well known that the performance of this material is highly dependent on its state [5]. Mechanical pre-stress ($\sigma_0$) is considered one of the primary factors, along with magnetic field intensity (H) and temperature (T), which influence a deformation $\Delta l/l$ [4-6]. To produce a length change the mechanical pre-stress on the material was applied to rotate magnetic domains relative the direction of the applied stress. It means that a part of the elastic strain produced by the pre-stress arising from the rotation or movement of magnetic domains before the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ rod is magnetized [2, 3, 7, 8].

Among others, Duenas at al. [9, 10] proved that maximum value of the magnetostrictive strain is enhanced by an axial compressive pre-stress when the magnetization of the rod approaches the saturation value. For instance, the saturated magnetostriction can reach 1200 ppm under the pre-stress at 5.3 MPa [11, 12].

The limited applications of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ monolithic material arises from the intrinsic brittleness, high price, large magnetic fields required to induce strain and development of eddy currents in high frequency range. Potential solutions are the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ based composites, which have been examined since it was noticed that the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ disadvantages could be overcome by combining the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ powder with polymer matrix. Interaction between the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles and bonding material is the key to the overall properties of the composite [13, 14]. Binder polymerization constitutes the source of internal stress for the magnetostrictive composite, eliminating the need for externally generated pre-stress [3, 7, 10, 15, 16].

While $Td_{0.3}Dy_{0.7}Fe_{1.9}$ composites have many advantages, the significantly reduced magnetization and magnetostriction in these materials as compared to monolithic $Td_{0.3}Dy_{0.7}Fe_{1.9}$has been reported [9, 11, 17]. It has been found [12, 17] that these properties would be improved when the particles are magnetically aligned in the one direction. The field aligns the particles so that the mechanical behaviour is similar to that exhibited by a continuous fibre composite. When a magnetic field is applied next, an effective magnetostriction has been observed due to the particle’s magnetostriction and the magnetic interaction force between particles [17].

A connectivity study [18, 19] shows that composites with magnetic particle alignment produces 20% more strain than a non-oriented composites.

The goal of this paper is to describe manufacturing process of the polymer matrix composite materials reinforced by $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles and to present of the investigation results for materials with randomly oriented magnetostrictive particles in epoxy matrix and with the particles aligned in the matrix.

## 2. Experimental procedure

### 2.1. Material and technology

The experiments were made with the $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles (Etrema Products Inc., USA) dispersed in a resin system. The epoxy system is composed of two very low viscosity components (~65cps), which were mixed in equal parts by weight (Figs. 1a, b).

The resulting composite pastes were mixed together (Fig. 1c) and cast into aluminium mould of 60x36x10 mm size (Fig. 1d). For oriented samples, in order to align the particles, magnetic field of a pair of NdFeB permanent magnets was employed during epoxy polymerization (Fig. 1e). These magnets produced magnetic field (120 μT measured in the centre) along the longitudinal direction of the mould and caused the particles to align with the magnetic flux lines, creating an anisotropic particle distribution.

The epoxy system polymerization process was performed in electric furnace (Thermolyne 6000) at the temperature of 70°C over the period of 10 h. Such thermal curing process was shown to result in axial and radial residual stresses of ~3 and ~0.7 MPa respectively, even though no external stresses were used [19].

After cooling, the specimen was removed from the mould, and ground using the abrasive paper (400 and 800 grade). The final shape of the composites was rectangular with dimensions of 2x3x4 mm.

In this work, two different types of the composite were made. The composites of the first type has the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles randomly oriented (non-oriented) and the second type the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles were oriented for the magnetic field, making them anisotropic. The volume fractions of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles equalled to 10% and 25%.

The present work on polymer-bonded $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ composites is performed in a similar way to the previous works [20, 21].

All the specimens, fabricated with the method described above were then subjected to magnetic and magnetostrictive examination. The measurements were taken both at the transverse ($\lambda\perp$) and longitudinal ($\lambda_{\parallel}$) directions on magnetic alignment of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles during polymerization.

The goal of this work was also to examine morphology of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ powder and structure of the composite materials, as well as to evaluate its phase and chemical composition using the X-ray analysis and EDS method for the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles.

### 2.2. Investigations

Examination of the $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ powder morphology was made on the DSM 940 OPTON scanning electron microscope with the Oxford EDS LINK ISIS dispersive radiation spectrometer at the maximum magnification of 500× using the secondary electron detection at the 20 kV accelerating voltage.

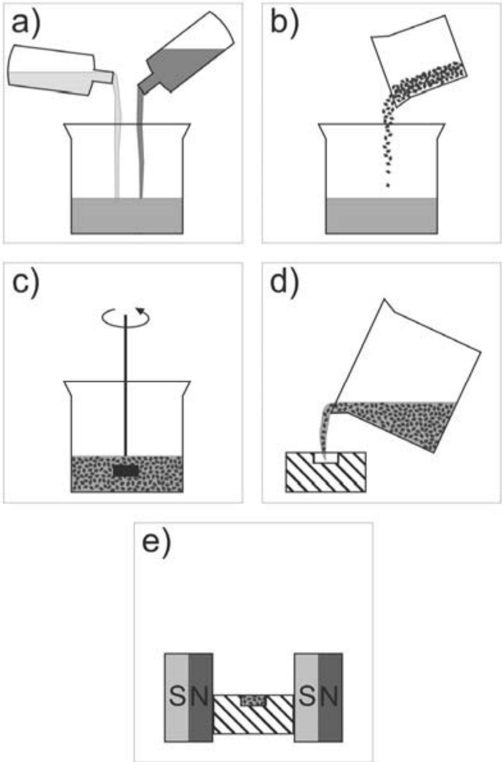



Fig. 1. Composite materials forming sequence: a) epoxy system mixing; b) incorporating $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles into the epoxy matrix; c) mixing; d) slurry casting; e) magnetic alignment of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles (optional)

Metallographic examinations of the resulting composite materials were made on the LEICA MEF4A light microscope equipped with the computer image analysis system at 100x magnification.

Phase compositions of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ powder were determined by the X-ray diffraction method using the X-PERT PRO device with a cobalt lamp with 40 kV voltage. The data for X-ray diffraction lines were obtained by "step-scanning" method in 2θ range from 25° to 130°.

The magnetostriction measurements were performed at room temperature using three terminal capacitive techniques and with a magnetic field up to 1 T. The specification of this methodology has been described in details in previous works [20, 22]. The examination method is based on measurement of the sample which changes the dimension caused by magnetostriction. This is observed by variation in capacitance across those two electrodes: fixed and movable one (Fig. 2).

The magnetic behaviour of composite materials was characterized by hysteresis loop measurements using the vibrating-sample magnetometer (MagLab 1.2 T, Oxford Instruments, Ltd.) at room temperature and with the maximum field up to 1 T.

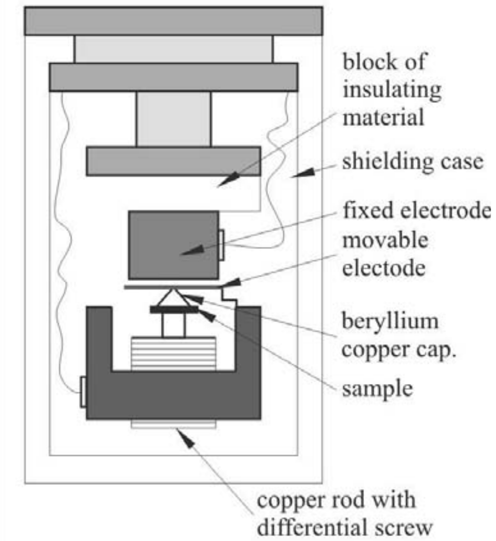



Fig. 2. The capacitive cell adapted for magnetostriction measurements [22]

In order to examine whether the manufacturing process has any influence on magnetic and magnetostriction properties, the tests were done in two perpendicular directions, respectively to the direction on magnetic alignment.

## 3. Description of the obtained results

### 3.1. Structure

Morphology of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ powder, observed on the scanning electron microscope is shown in Fig. 3. The sizes of the $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles used for fabricating the composite materials were from 38 to 106 μm.

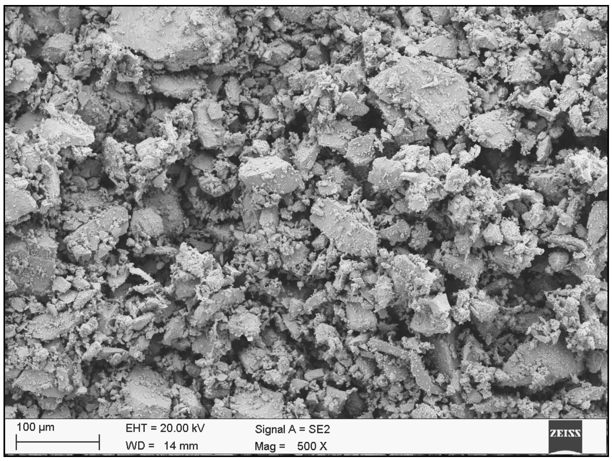
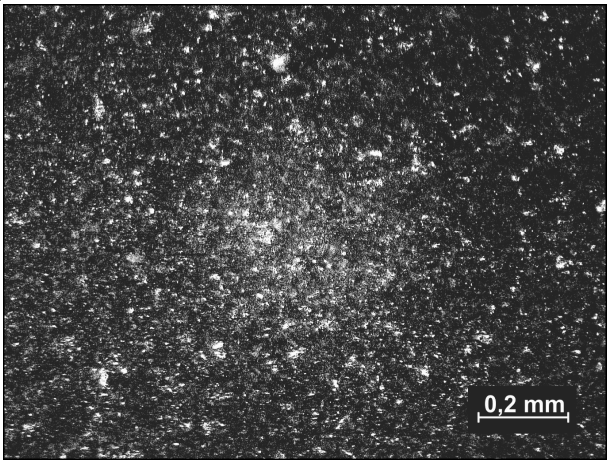



Fig. 3. $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ powder morphology observed by SEM

Results of the metallographic examinations of the composite materials, carried out on the light microscope are presented in Fig. 4. The observations show the uniform distribution of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles in epoxy matrix for the non-oriented composites (Fig. 4a). Moreover, it was found out that the result of magnetic field operating during the epoxy matrix curing is to arrange $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles into chain-like columns (Fig. 4b). The $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles are aligned in one direction, arranged in parallel along magnetic flux lines produced by NdFeB permanent magnets.

a)




b)

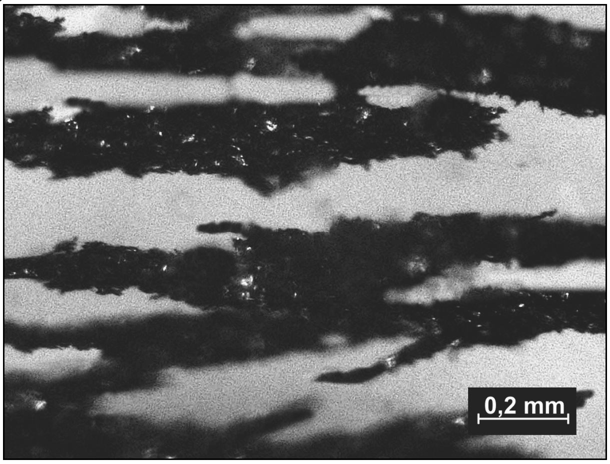



Fig. 4. Microstructure of composite materials with 10% volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$: a) with randomly oriented $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles in the epoxy matrix; b) with aligning $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles in the matrix; magnification 100x

## 3.2. EDS and XRD analysis

Examinations of the chemical composition on the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles made by the X-ray energy dispersive spectrometer (EDS) confirm presence of iron, dysprosium and terbium (Fig. 5). Information about the weight and atomic ratios of the particular elements is given in Table 1.

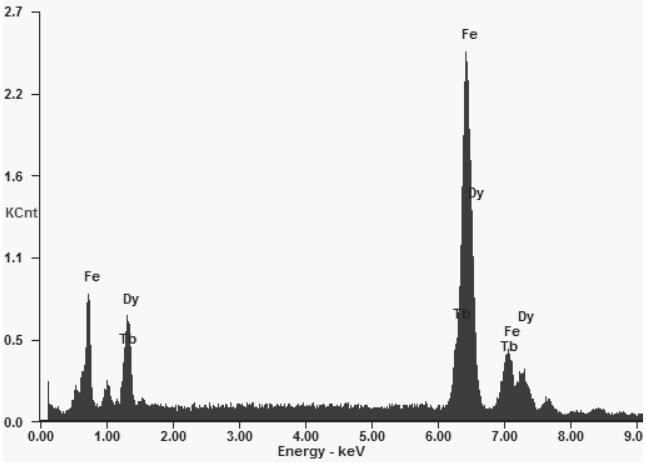



Fig. 5. EDS analysis of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ powder

Table 1.
Chemical composition of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ powder

| Element | Concentration of element | |
|---|---|---|
| | wt., % | at., % |
| Dy | 41.93 | 23.77 |
| Fe | 39.77 | 65.61 |
| Tb | 18.31 | 10.61 |

The X-ray diffraction pattern obtained for $Td_{0.3}Dy_{0.7}Fe_{1.9}$ powder displays numerous diffraction peaks, which reflect the diffraction intensities. As it is seen in Fig. 6, $Td_{0.3}Dy_{0.7}Fe_{1.9}$ is generally considered to be the $RFe_2$ phase (where R = rare earth elements Tb or Dy) so it can be considered as a magnetostrictive material.

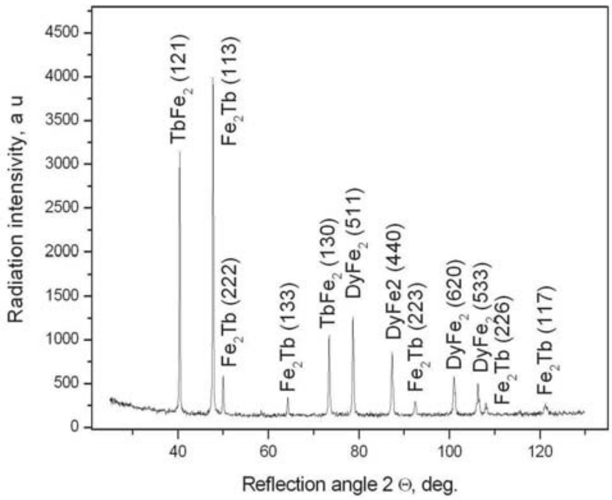



Fig. 6. X-ray diffraction pattern of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ powder

## 3.3. Magnetostrictive and magnetic results

From Figs. 7 and 8 it is possible to compare the magnetization and magnetostriction as the function of the external magnetic field for the non-oriented and oriented (Figs. 9 and 10) $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles composites for the various $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fractions.

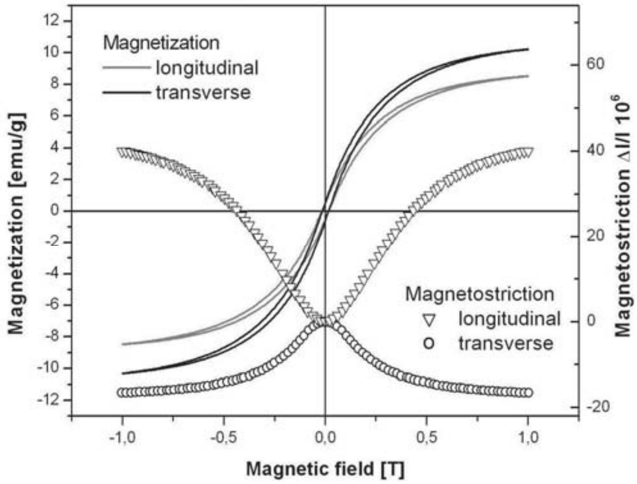



Fig. 7. Relationship of magnetization and magnetostriction with magnetic field, including the external longitudinal field applied (grey curves) and transverse one (dark curves) to the internal bias field for the non-oriented composite materials with 10% volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$

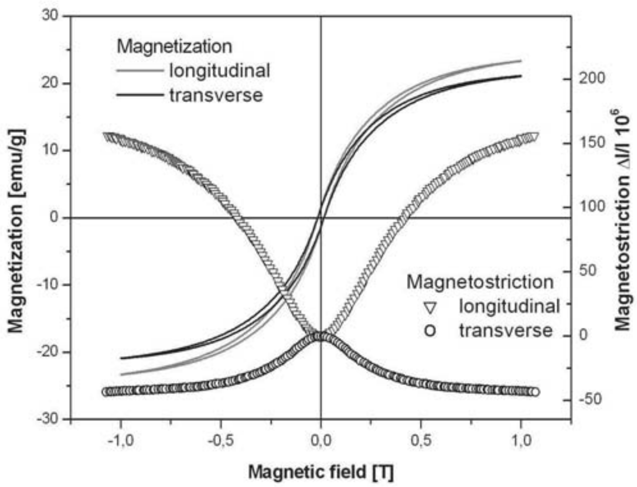



Fig. 8. Relationship of magnetization and magnetostriction with magnetic field, including the external longitudinal field applied (grey curves) and transverse one (dark curves) to the internal bias field for the non-oriented composite materials with 25% volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$

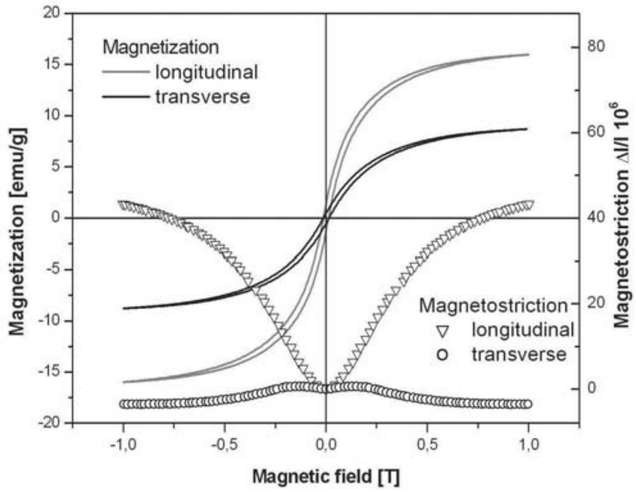



Fig. 9. Relationship of magnetization and magnetostriction with magnetic field, including the external longitudinal field applied (grey curves) and transverse one (dark curves) to the internal bias field for the non-oriented composite materials with 10% volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$

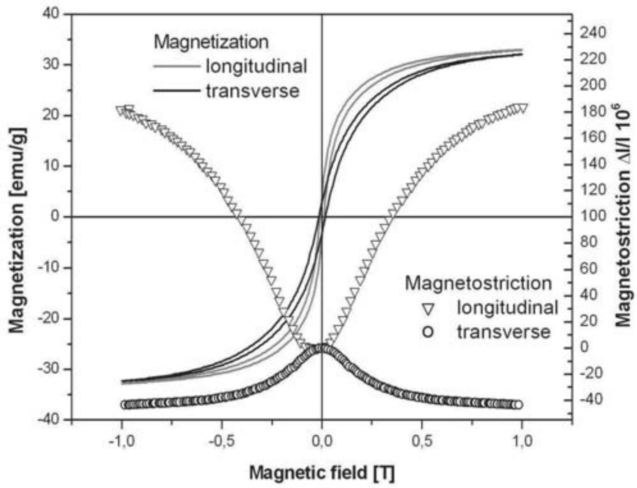



Fig. 10. Relationship of magnetization and magnetostriction with magnetic field, including the external longitudinal field applied (grey curves) and transverse one (dark curves) to the internal bias field for the non-oriented composite materials with 25% volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$

The effect of magnetic particle alignment is observed in the magnetostriction response as seen in Figs. 9 and 10. The magnetostrictive response improves when the magnetic particles are oriented in the magnetic fields and reaches approximately 184 ppm for the oriented 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles.

Differences between the longitudinal and transverse measurements for specimens with the same $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fraction arise from the anisotropic distribution of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles into epoxy matrix. For this reason, the magnetostriction for the non-oriented composites reaches the highest values in transverse direction then for oriented composites but at the same time – the smaller longitudinal response.

Fig. 11 shows the maximum longitudinal and transverse strain response as a function of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fractions. The 25% $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fraction oriented composite yields the highest response (measured in the longitudinal direction) and the 25% volume fraction non-oriented composite yields the lowest response of magnetostriction (measured in the transverse direction).

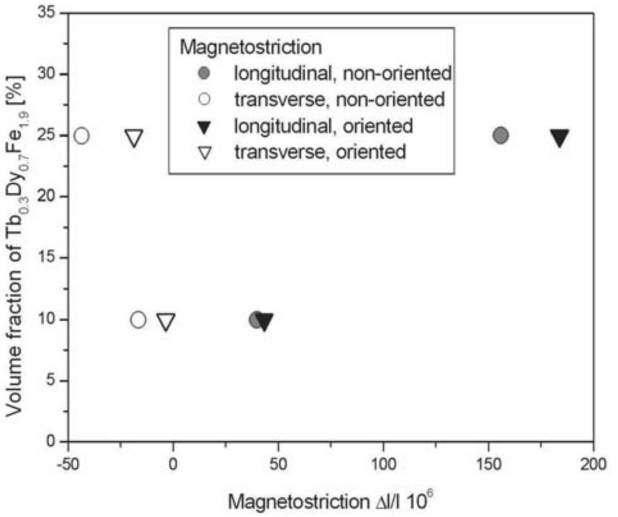



Fig. 11. Maximum magnetostrictive response as a function of volume fraction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$. Measurements were made at B = 0.95 T

Saturation magnetostriction (Table 2) measured in the longitudinal direction ranging from 40 ppm (at magnetic field equals 0.96 T) for the non-oriented composite with 10% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ up to 184 ppm (at magnetic field equals 0.97 T) for oriented composite material with 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$.

It can be found that the saturation magnetostriction of the non-oriented sample with 10% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ is about four times bigger than that of the oriented sample with 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$.

It was also noticed, as expected, that the increase of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fraction in composite materials amplify the magnetostrictive response. This is due to the particle's magnetostriction and the magnetic interaction force between particles.

Tests of magnetic properties revealed that the highest magnetization (33 emu/g) was characteristic for the composite with 25% $Td_{0.3}Dy_{0.7}Fe_{1.9}$ volume fraction. Magnetization values decreased along with the lowering ratio of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles in the composite, reaching 8,53 emu/g for the composite with the 10 % volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$.

Table 2.
Results of the saturation magnetostriction for the non-oriented and oriented $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles composites

| Volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$, % | | Magnetic field, T | Magnetostriction, ppm |
|---|---|---|---|
| 10% | for non-oriented sample | | |
| | • longitudinal | 0.96 | 40.00 |
| | • transverse | 0.96 | -16.48 |
| | for oriented sample | | |
| | • longitudinal | -0.96 | 43.30 |
| | • transverse | 0.94 | -3.54 |
| 25% | for non-oriented sample | | |
| | • longitudinal | 0.94 | 156.00 |
| | • transverse | 0.94 | -43.50 |
| | for oriented sample | | |
| | • longitudinal | 0.97 | 184.00 |
| | • transverse | -0.97 | -18.68 |

It is observed that the magnetic tests realized in the longitudinal direction provide the highest responses that performed in transverse direction.

The magnitude of magnetization for composite materials varies in a regular way due to $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particle alignment during fabrication: for composite material with 10% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$, magnetization increase from 8.53 emu/g for non-oriented samples up to 16 emu/g for samples with oriented $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles for measurement conducted in longitudinal direction. During measurement performed in the transverse direction to the field applied, inverse dependence was observed: magnetization decreased from 10.24 emu/g for non-oriented samples to 8.71 emu/g for samples with oriented $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles.

The detailed analysis of data of magnetic properties (in particular low values of coercivity) indicates that $Td_{0.3}Dy_{0.7}Fe_{1.9}$ composite materials possess good magnetic softness.

## 4. Conclusions

This work gives valuable insight into how the behaviour of composite materials reinforced by $Td_{0.3}Dy_{0.7}Fe_{1.9}$ changes under aligning particles in the epoxy matrix during polymerization. It was found out that $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles magnetic alignment has been successfully developed for manufacturing the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ composites. Composites with preferential alignment orientation exhibit a great saturation magnetostriction value reaching 184 ppm for oriented composite material with 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ (measured in longitudinal direction). This shows the need for magnetic alignment of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles.

It was found that, based on microscope examinations, $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles are distributed uniformly in the entire epoxy matrix (in the case of non-oriented specimens). In oriented composite materials, $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ particles are arranged into chain-like columns, causing anisotropic properties of obtained samples.

Based on the investigation, it was noticed that low values of magnetostriction measured in transverse direction for composites are due to anisotropic distribution of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles. On the other hand it is a reason for the greater magnetostriction response during longitudinal measurements.

The additional benefit of achieved materials is saturation magnetostriction which equals 184 ppm for oriented composite material with 25% volume fraction of $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles when the monolithic $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ material range from 800 to 1200 ppm. The saturation value of the magnetostrictive strain is obtained when the magnetization of the rod approaches the maximum value.

The advantage of composite materials fabricated in this work is that they possess the internal preload exerted by the epoxy shrinking onto the particles during polymerization process. This effect overcomes the disadvantage of monolithic $Td_{0.3}Dy_{0.7}Fe_{1.9}$, which must be preloaded in order to initiate magnetization “jumping”.

The analysis of the magnetic properties test results of the polymer matrix composite materials reinforced by $Td_{0.3}Dy_{0.7}Fe_{1.9}$ magnetostrictive particles revealed that the soft magnetic properties of the composite are dependent on the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particles ratio in the composite. In addition, application of the magnetic alignment increases magnetization of the composite materials.

Further work is needed – among others – to identify the influence of the $Td_{0.3}Dy_{0.7}Fe_{1.9}$ particle size on the magnetostrictive and magnetic responses. Demagnetization effect should be also considered.

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