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Neutron depolarization investigations of spring exchange interaction nanocomposites

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Neutron depolarization of the magnetic state of spring exchange interaction nanocomposites based on NdFeB is carried out. It is detected a different behavior for magnetically soft and rigid nanocomposite. Magnetically hard nanocomposite exhibits coercive force equal to field of maximal neutron depolarization and for a soft one a maximal depolarization field has a value smaller than coercive field. It was carried out also magnetic measurements using a vibrating sample magnetometer and X-ray diffraction in order to evaluate the crystallographic phases involved in a relationship with the magnetic behavior. The conclusion is made that it is connected with different magnetization regime and it is important for realization of spring regime.

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1. Introduction

Spring exchange interaction magnetic nanocomposite have a pronounced magnetic anisotropy phase (called hard magnetic material) and a low magnetic anisotropy ferromagnetic phase (called soft magnetic material) [1-8]. In ferromagnetic nanocomposites the linear size of soft magnetic phase is at most 2 times the length of the exchange interaction hard magnetic phase. The exchange interaction length is almost equal to the width of the Bloch wall hard magnetic material and takes place (the exchange interaction between the two phases) by the strong exchange hardening soft magnetic phase. The overall effect is like a lower magnetic anisotropy of nanocomposite (due to the anisotropy energy distribution of the hard phase in the entire volume of the nanocomposite) with an increase of the saturation magnetization - and therefore the remanent magnetization. The effect of increasing of the magnetization it is immediately reflected in the theoretical maximum magnetic energy density, that can be achieved with such nanocomposites: the nanocomposite $Nd_2Fe_{14}B$ / α -Fe magnetic isotropic 50/50 might get more than 200 kJ/m^3 and an anisotropic nanocomposite even 690 kJ/m^3! Values are much higher than the maximum expected for systems known (for example, sintered NdFeB magnets by about 500 kJ / m3). [9-11] Method of neutron depolarization is very useful in the investigation of structural inhomogeneities in the magnetic materials and superconductors. This technique involves shining a collimated polarized beam of neutrons onto a surface of the sample perpendicular to its plane and measuring the intensity of the beam passing through the sample as a function of neutron wavelength.

Spin-flippers are a device for changing the polarization of neutrons. So if we have polarizer and spin-flipper before sample and spin-flipper and polarization analyzer after sample, it's possible to get information about changing of neutron polarization after passing through the sample for different input polarization.

Neutron depolarization caused by Larmor precession in individual domains and it depends on the domain size, magnetization of a domain, the mean square direction cosines of the magnetization of the domains and the thickness of the sample.

Process of small angle-angle scattering occurs simultaneously with the process of depolarization. It also gives information about structural inhomogeneities with correlation length 1 nm – 1 μm.

Method of neutron depolarization described in details in works [12-14]. In this work all measurements were made on spectrometer REMUR.

The main objective of the paper is related to the investigation of physical properties of some spring exchange interaction, magnetic nanocomposites that can be good candidate precursors for permanent magnets having a lower content of rare earth, so cheaper magnets.

2. Experimental

Experimental researches were pursued in order of obtaining 3 compositional types of material based on NdFeB: $Nd_2Fe_{14}B$+5%Fe; $Nd_2Fe_{14}B$+10%Fe and $Nd_2Fe_{14}B$+15%Fe. In order to obtain Nd-Fe-B alloy (bulk) it was used the following master alloys (mass %): Nd_{84}-Fe_{16}, Fe_{80}-B_{20} and pure deoxidized Fe. It was used a Leybold-Heraeus induction furnace in the Argon protective atmosphere for casting the alloys used further. Then the Nd-Fe-B with Fe additions (5%, 10%, 15%) was rapidly solidified using a melt-spinning system, in different conditions in order to obtain amorphous ribbons. In table 1 described processing conditions of the NdFeB ribbons

investigated and the magnetic properties after measuring them with the VSM.

Table 1. Processing conditions of the NdFeB ribbons investigated

Sample No	**Alloy**	**V (m/s) Linear Speed**	**L (mm) Bandwidth**	**≠ (µm) strip thickness**
1	$Nd_2Fe_{14}B$ + 5% Fe	32	2	28.8
2	$Nd_2Fe_{14}B$ + 10% Fe	26	2	22
3	$Nd_2Fe_{14}B$ + 10% Fe	32	2.2	28.3
4	$Nd_2Fe_{14}B$ + 15% Fe	32	2.5	21.8
5	$Nd_2Fe_{14}B$ + 15% Fe	35	2.8	26.8
6	$Nd_2Fe_{14}B$ + 5% Fe	35	2.2	21
7	$Nd_2Fe_{14}B$ + 15% Fe	36	2	33
8	$Nd_2Fe_{14}B$ + 15% Fe	34	2.3	26
9	$Nd_2Fe_{14}B$ + 10% Fe	35	2	37
10	$Nd_2Fe_{14}B$ + 15% Fe	33	2.6	37

The structural properties of the samples were investigated by X-ray diffraction, using a D8Discover (Bruker) instrument, having a PSD LynxEye detector, Gobell mirror, Cu Kα radiation in Bragg-Brentano geometry. It was evaluated the mean crystallite size using the Scherrer equation. We report the values for the both crystalline phases identified α-Fe and $Nd_2Fe_{14}B$, taking into account the fact that some ribbons were found to be amorphous.

Time of flight method is the method of measurement neutron count $J(t)$ in dependence of time of flight by neutron t, the distance L between neutron source and neutron detector, as result the neutron wavelength λ and $J(\lambda)$. The time of flight is:

$$t = \frac{L}{\upsilon} = \frac{Lm}{p} = \frac{Lm\lambda}{h},$$

where L – distance between neutron source and detector, λ – wavelength of neutron, m – mass of neutron.

3. Results and discussion

Hysteresis loop of some samples showed in Fig. 1.

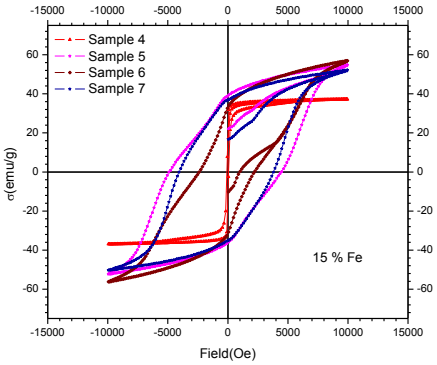

Fig. 1. Hysteresis loop of samples 4, 5, 7, 8 $Nd_2Fe_{14}B$ + 15% Fe

X-Ray diffractograms for ribbons samples are showed on Fig. 2 and some structural properties of the samples evaluated by X-ray diffraction showed in Table 2.

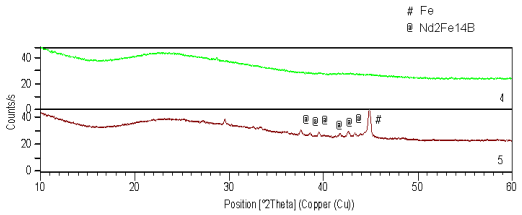

Fig. 2. XRD diffractogram for ribbons samples 4 and 5 corresponding to $Nd_2Fe_{14}B$ + 15% Fe

Table 2. Mean crystallite size D

Sample No	**Addition content of Fe**	**V (m/s) Linear Speed**	***D (nm) for α-Fe***	***D (nm) for $Nd_2Fe_{14}B$.***
1	5%	32	29,5	50.8
2	10%	26	31,7	27,7
3	10%	32	21,1	9,5
4	15%	32	40,7	20,8
5	15%	35	35,3	37,3
6	5%	35	36	39,2
7	15%	36	34,6	36,5
8	15%	34	37,3	35,4
9	10%	35	35,1	25,6
10	15%	33	40,7	32

There are dependences of R-ratio integral by wavelength from magnetic field on Fig. 3. The minimum of the R-ratio corresponds to a maximal magnetic field perpendicular to magnetization. Experimental data generalized in Table 3.

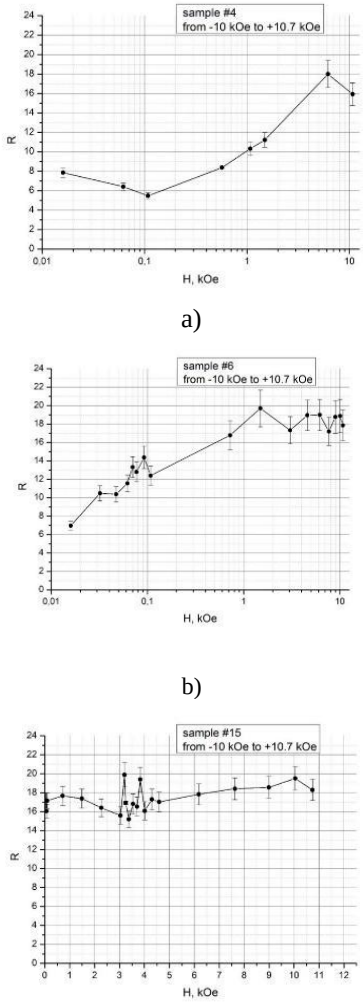

Fig. 3. Dependences of R-ratio integral by wavelength from magnetic field; a – sample №2, b – sample №3, c – sample №8

Table 3. Experimental data, L – thickness of the sample, H_c - coercive force, H_{exp} – coercive force from experiment with neutrons, B – magnetic induction, D – depolarization, d – size of the domain

S. №	L, μm	σ_s, emu/g	H_c, Oe	H_{exp}, Oe	$\eta = H_{exp}/H_c$	B, kGs	D	d, μm
2	22	58.67	1071	100	0.09	5.31	0.77	30.7
3	28.3	66.05	70	50	0.71	5.98	0.91	7.4
7	33	55.99	2212	1800	0.81	5.07	0.94	5.9
8	26	51.08	3831	3250	0.85	4.63	0.975	3.7

Magnetic induction was defined as sum of magnetic field and magnetization of the sample. Magnetization of the sample at H_c is known from magnetometry results. Wavelength of neutron accepted λ=1.3 Å.

For sample №8 were obtained dependences of *R* and *D* on wavelength (Fig. 4). For *R*-ratio from periodical oscillations at Δλ =2.7, 3.7, 4.75 Å and using the formula:

$$\varphi = 2\pi = 4.63 \cdot 10^{-6} \cdot B\,(Gs) \cdot L(\mu m) \cdot \Delta\lambda\,(Å)$$

we have B=2π(4.63·10-6·26·1.1)=5.2 T. Possible exist islands (with not big volume concentration) with induction 5.2 T. In Fig. 4 (b) is showed dependence of the depolarization on wavelength (averaged on 10 channels). Again, we see oscillations with period of order 1.1 Å.

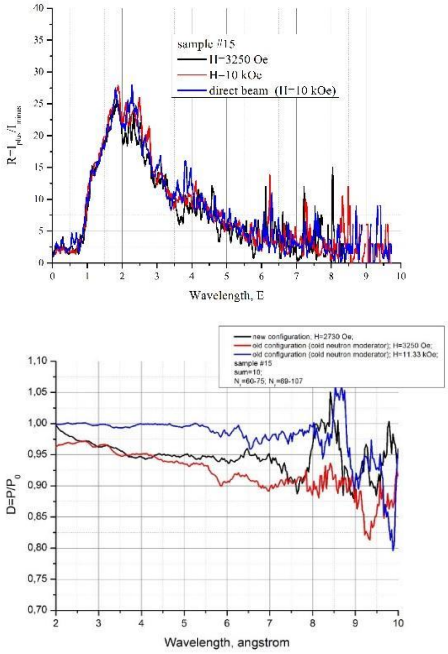

Fig. 4. Dependences of R-ratio (a) and D (b) on wavelength for sample №8

Sample №1 has enough thickness for complete depolarization of the neutron beam, simultaneously happens small-angle scattering, which increases with increasing of neutron's wavelength, and absorption of the neutron.

Experimental results showed that cross-section is close to proportional neutron wavelength dependence, this happening when absorption cross-section it is bigger that scattering cross-section. This is because Boron-element.

The magnetic measurements showed the influence of the addition of iron on the composition and the influence of the parameters of melt spinning.

The X-ray diffractogram and the magnetic loops reveal good magnetic properties at v=35 m/s.

The analysis of magnetic measurements of ribbons type $Nd_2Fe_{14}B$ + 15% Fe alloy, recrystallized at 620°C, showed the best magnetic coupling between hard magnetic phase $Nd_2Fe_{14}B$ and soft magnetic phase α-Fe. These magnetic characteristics recommend ribbons type $Nd_2Fe_{14}B$ + 15% Fe alloy, cast with v = 26m / s for further processing to obtain magnetic nanocomposites, densified by plasma sintering (Spark Plasma Sintering process). The processing parameters indicate the temperature of plasma sintering at around 600°C - 630°C and holding time of the order of minutes.

4. Conclusions

Neutron experiments revealed a ratio between anisotropy field and coercive field of 0.85 which indicates the possibility of increasing the magnetic properties by increasing of coercive field.

Both, the neutron experiments and classic magnetometry experiments confirmed the existence of the two crystalline phases, evidenced also by X-ray diffractometry, being a soft and a hard magnetic phases.

The magnetic domains were found to be in order of few microns in relationship with the ratio of 0.85 between the anisotropy and coercive field.

By neutron experiments were evidenced the existence of islands (with not big volume concentration) with induction 5.2 T, but they not contribute to the global magnetic properties, as it was evidenced by magnetometry experiments. It remains to investigate further if they are assumed with some particular crystalline phases.

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