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Magnetic anisotropy of FE $_X$ NI $_{100-X}$ nanotubes depending on their composition

Abstract: the article considers electrodeposition into porous polyethylene terephthalate matrices as an effective method for obtaining ordered arrays of magnetic nanostructures. By varying the electrolyte composition, it is easy to control the morphology and composition of nanowires/nanotubes, which determines their structural and magnetic properties. Ferromagnetic nanotubes based on nickel, iron, and their alloys have been synthesized in the pores of ion-track membranes. The compositions of electrolytes and synthesis conditions were determined, which makes it possible to obtain the following nanotube compositions: Fe, $Fe_{80}Ni_{20}$, $Fe_{60}Ni_{40}$, $Fe_{20}Ni_{80}$, Ni. A change in the composition of nanotubes significantly affects their crystal structure: with an increase in the nickel content, the lattice type transforms from bcc (characteristic of Fe) to fcc (characteristic of Ni), which is accompanied by a significant change in structural parameters. The process of magnetization of nanotube arrays depending on composition and crystal structure is analyzed, the main magnetic parameters are determined. Consequently, the magnetization vectors lie randomly in the walls of the pipe. Estimation of the main magnetic parameters from hysteresis loops measured in a magnetic field directed along the tubes and in the perpendicular direction reveals a complex magnetization process, including magnetization rotation and domain wall propagation.

Keywords: ferromagnetic nanotubes, 3d metals, template synthesis, crystal structure, magnetization.

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Introduction. One-dimensional magnetic nanostructures made of pure metals and alloys are being actively studied due to their specific surface properties, magnetic and optical properties, which, due to their small size, differ from those of bulk materials. The largest number of studies is devoted to iron-containing nanotubes, including iron-nickel and iron-cobalt alloys. One-dimensional nanostructures based on these alloys can potentially have unique magnetic and electrical properties [1], due to pronounced anisotropy of magnetic properties [2, 3], creation of domain walls, etc. In addition, having a low coercivity but a high magnetization value, iron-nickel nanotubes can be widely used in biomedicine as magnetic drug and protein carriers [4, 5]. Arrays of ferromagnetic one-dimensional nanostructures synthesized in porous templates can be used for high-density magnetic memory, to produce shields against high-frequency radiation, and for logic and memory devices based on domain walls [6, 7, 8]. Synthesized in polymer templates, such structures are of interest to produce flexible electronics [9].

The magnetic anisotropy of ferromagnetic nanowires can be manipulated by controlling the crystal structure [10], composition [11], and intrinsic exchange coupling [12]. In an external magnetic field, these magnetic properties can be easily controlled, which opens new possibilities for the application of such arrays.

The iron-nickel alloy demonstrates various physical characteristics when the atomic ratio changes and is one of the universal soft magnetic materials with a wide application range. The magnetic properties of homogeneous FeNi nanowires/nanotubes have been extensively studied [13, 14, 15]. However, there has not been carried out a systematic study of the influence of the composition of FeNi nanotubes on magnetic properties yet.

In our work, electrodeposition into porous polyethylene terephthalate matrices was used as an effective method for obtaining ordered arrays of magnetic nanostructures. By varying the electrolyte composition, it is easy to control the morphology and composition of nanowires/nanotubes, which determines their structural and magnetic properties.

Materials and methods. The PET-based porous template formation features were considered by us in [16], for the synthesis, membranes with parameters were used as templates: thickness 11.8 ± 0.2 μm, irradiation fluence 4*10^{7} cm^{-2} cathode layer 10 nm. The deposition was carried out in an electrochemical cell by a two-electrode method in a potentiostatic mode from electrolytes based on sulfate salts of iron and nickel in molar ratios of salts $FeSO_4 x7H_2O$: $NiSO_4 x7H_2O$ 1:1, 1:5, 1:10. To maintain acidity, H_3BO_3 (45 g / l), $C_6H_8O_6$ (3 g / l) were added to the electrolytes, the pH of the electrolyte was 3 at a temperature of 25 $^\circ$C and the deposition potential difference was 1.75 V. The morphology and composition of synthesized nanotubes were investigated by scanning electron microscopy (SEM, Hitachi TM3030) and energy dispersion analysis (EDA, Bruker XFlash MIN SVE). The internal diameters d of iron-nickel nanotubes for the entire series of compositions were determined by the gas permeability method at an excess pressure of 8 - 20 kPa with a step of 4 kPa [17]. X-ray diffraction analysis was performed on a D8 ADVANCE ECO diffractometer (Bruker, Germany) using CuKα radiation (γ = 15.4060 nm). The diffraction patterns were recorded in the angle range of 2θ = 30-90° with a step of 0,02°. The BrukerAXSDIFFRAC.EVAv.4.2 software with the international database ICDD PDF-2 was used to identify the phases. To measure magnetic characteristics, the universal measuring system "Liquid Helium Free High Field Measurement System (Cryogenic LTD, London, UK)" was used. Magnetic properties were examined in a magnetic field B = ± 20,000 Oe at 300 K.

Results and discussion. A typical view of a nanotube array in a polymer template is shown in Figure 1.

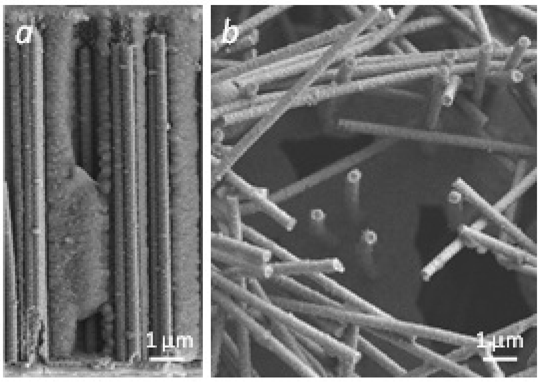

Figure 1 – Typical SEM images of an array of synthesized nanotubes: cleavage (a); scanning orthogonally to the surface (b)

The outer diameter of the synthesized NTs corresponds to the pore diameter of the template (D = 390 ± 20 nm). The length was controlled by the deposition time on the chronoamperogram. The average deposition time was 780 ± 10 s, during which NTs with a length of 10.5 ± 0.3 μm were formed to prevent the formation of a metal film on the surface. The obtained values were d = 180 ± 20 nm; therefore, the NT wall thickness was 90 ± 20 nm. To determine the atomic composition, the method of energy-dispersive analysis was used, the results are shown in Table 1.

X-ray diffraction analysis was carried out to determine the phase composition, as well as the structural parameter of NT arrays. Figure 2 shows X-ray diffraction patterns of the synthesized samples, Table 1 shows the phase composition of nanotubes.

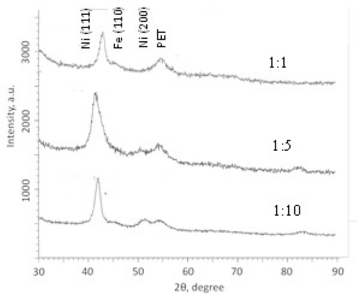

FIGURE 2 – X-ray diffraction patterns of the studied samples

As a result of processing X-ray diffraction patterns, the unit cell parameters and crystallite sizes were determined. The crystal lattice parameter was calculated using the Nelson - Taylor extrapolation function (1):

$$a = f\left[\frac{1}{2}(\frac{cos^2\theta}{sin\theta} + \frac{cos\theta}{sin\theta})\right] \tag{1}$$

The distance of coherent X-ray scattering (approximately equal to a crystallite size) was calculated according to the Scherer equation:

$$L = \frac{k_s\lambda}{\beta cos\theta} \tag{2}$$

where k $_s$ $= 0.9$ is the dimensionless particle shape factor (Scherrer constant), λ $= 1.54$ A is the X-ray wavelength, β is the parameter of FWHM.

The volume fraction of the phase contribution was determined using equation (3):

$$V_{admixture} = \frac{RI_{phase}}{I_{admixture} + RI_{phase}} \tag{3}$$

I $_{phase}$ is the average integrated intensity of the main phase of the diffraction line, I $_{admixture}$ is the average integrated intensity of the additional phase, R is the structural coefficient equal to 1.45.

An analysis of the diffraction patterns showed that nanotubes consisting of iron have a bcc lattice with a unit cell parameter a = 2.8627 (Table 2). When iron is diluted with nickel to Fe concentrations of 80% and 60%, bcc remains the predominant phase, while with an increase in nickel concentration, an increase in the crystal lattice parameter is observed up to 2.8794 Å for $Fe_{80}Ni_{20}$ and further to 2.8854 Å for $Fe_{60}Ni_{40}$. A decrease in the iron concentration to 40% leads to a rearrangement of the crystal lattice and the predominance of the fcc phase, which is characteristic of nickel. A further increase in the nickel content leads to an increase in the unit cell parameter to 3.5695 Å for samples with the $Fe_{20}Ni_{80}$ composition.

TABLE 1 – Crystal structure of synthesized nanotubes

Sample	Crystal lattice type	Crystal lattice parameter, Å	Average crystallite size, nm	Phase content			Atomic ratio, %	
				Ni	Fe	FeNi	Ni	Fe
$Fe_{81}Ni_{19}$	bcc	2.8794	19	5	82	13	19	81
$Fe_{62}Ni_{38}$	bcc	2.8854	18	26	59	15	38	62
$Fe_{21}Ni_{79}$	fcc	3.5195	13	68	7	25	79	21

The volume fraction of the phases shows that the phase composition is close to the atomic ratio specified and determined by the EDA method.

In addition, for pure iron, the predominant crystallographic direction is (110), the severity of which decreases with an increasing nickel content in the composition of nanotubes. At the same time, the texture coefficient (111) of the nickel component increases simultaneously. The transformation of the crystal structure with a change in composition occurs under the influence of the mechanism of nanotube growth in the pores of polymer templates [10], which is the layer-by-layer formation of nanotube walls, because of which the priority crystallographic direction becomes the direction along their main axis for the predominant metal in the composition. This is also related to the rearrangement of the crystal structure from bcc to fcc lattice with an increase in the nickel content in the composition of nanotubes.

The field dependences of the magnetization obtained as a result of the measurement are shown in Figure 3.

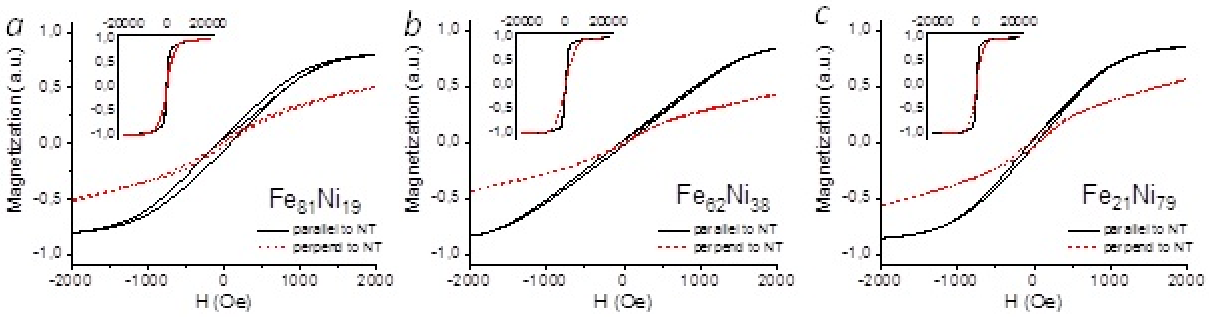

FIGURE 3 – Field dependences of the magnetization of arrays of iron-nickel nanotubes in the perpendicular (black solid line) and parallel (red dotted line) directions of the magnetic field relative to the main axis of the nanotubes. The compositions of nanotubes are signed directly on the figures)

The magnetic hysteresis loops measured for two magnetizing field directions (perpendicular and parallel with respect to the NTs axis) are presented in Fig. 3 for different NTs composition. The characteristic magnetic parameters: coercivity and squarenesses ratio $\frac{SQ=M_S}{M_S}$ of remanence magnetization M_r to saturation magnetization M_s are found from the hysteresis loops (Fig. 3) and presented in Table 2.

TABLE 2 – Basic magnetic characteristics of FeNi NT for parallel and perpendicular directions of the magnetic field

Composition	$H_{C\parallel}$	$SQ_{\parallel}$	$H_{C\perp}$	$SQ_{\perp}$
	Oe		Oe	
$Fe_{81}Ni_{19}$	87	0,0605	42	0,0192
$Fe_{62}Ni_{38}$	41	0,0237	36	0,0162
$Fe_{21}Ni_{79}$	43	0,0325	53	0,0388

Studies of the magnetization of NT arrays showed that the behavior of hysteresis loops has the form characteristic of ferromagnetic materials [18]. Previously, it was shown that magnetic nanotubes are characterized by anisotropy of magnetic properties, mainly associated with shape anisotropy, which is most characteristic of pure metals and alloys containing Ni [11], [19]. Such materials are characterized by the orientation of the easy magnetization axis along the nanotube axis. For samples consisting of alloys, anisotropy may not be pronounced [20], and with a high defectiveness of the structure and small sizes of crystallites, domain walls, and vortexes can be observed [21], most often this effect is observed in permalloys. The samples of FeNi nanotubes under consideration are characterized by a low coercivity, in contrast to pure metals. For samples with a high iron concentration, the values of the magnetic parameters for the direction of the magnetic field along the axis of the nanotubes are several times larger than for the perpendicular one. For samples with a Fe content below 60%, no pronounced anisotropy is observed, and the coercivity differs insignificantly for both directions of the magnetic field. This behavior of the magnetic parameters is associated with the predominance of crystallographic anisotropy, which is characteristic of the fine-grained structure of nanotubes. In addition, it is important to note that in these two compositions, the crystal structure is rearranged from bcc, which is characteristic of iron, to fcc, which is characteristic of nickel, which will affect the magnetic characteristics of the NT.

Conclusion. A series of samples of nickel-iron alloy nanotubes were synthesized by electrodeposition in the pores of ion-track membranes made of polyethylene terephthalate using the potentiostatic mode. The influence of the electrolyte composition on the resulting composition of nanotubes is determined. A series of samples demonstrated the effect of deposition conditions on the structural characteristics of the alloy. With a decrease in the iron content in the composition of nanotubes below 60%, the crystal lattice is rearranged from bcc (typical for Fe) to fcc (typical for Ni), which is associated with a change in the priority direction of crystallites. In accordance with the change in the type of crystal lattice, the lattice parameter changes significantly. Changes in the composition of the samples and the corresponding structural changes determine the specific features of the magnetic properties of nanotube arrays. The coercive force and stockiness of the hysteresis loops for alloy samples are significantly reduced compared to pure metals. The shape anisotropy is dominant in all cases, while the crystalline anisotropy is randomly distributed. Consequently, the magnetization vectors lie randomly in the walls of the pipe. Estimation of the main magnetic parameters from hysteresis loops measured in a magnetic field directed along the tubes and in the perpendicular direction reveals a complex magnetization process, including magnetization rotation and domain wall propagation. The results of our work can be useful for the creation of composite materials with oriented magnetic nanotubes, as well as sensitive elements of nano- and microelectronics.

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FE $_X$ NI $_{100-X}$ нанотүтіктерінің құрамына байланысты магниттік анизотропиясы

Аннотация. Иондық-тректі мембраналардың кеуектерінде никель, темір және олардың қорытпалары негізінде ферромагниттік нанотүтікшелер синтезделеді. Fe, Fe80Ni20, Fe60Ni40, Fe20Ni80 Ni нанотүтіктік композицияларды алуға мүмкіндік беретін электролиттердің құрамы мен синтез жағдайлары анықталды. Нанотүтікшелер құрамының өзгеруі олардың кристалды құрылымына айтарлықтай әсер етеді: никель мөлшері ұлғайған сайын тор түрі денеге бағытталған текшеден (Fe үшін тән) бет центрлік текшеге (Ni үшін тән) өзгереді, бұл құрылымдық параметрлердің айтарлықтай өзгеруімен бірге жүреді. Нанотүтікшелер массивтерінің магниттелу процесі құрамы мен кристалды құрылымына байланысты талданды, негізгі магниттік параметрлер анықталды. Құрамы мен кристалдың құрылымына байланысты нанотүтік массивтерінің магниттелу процесі талданады, негізгі магниттік параметрлері анықталды. Демек, магниттелу векторлары құбыр қабырғаларында кездейсоқ жатады. Түтіктер бойымен және перпендикуляр бағытта бағытталған магнит өрісінде өлшенген гистерезис контурларынан негізгі магниттік параметрлерді бағалау магниттелудің айналуын және домен кабырғасының таралуын қамтитын күрделі магниттелу процесін корсетеді.

Түйін сөздер: ферромагниттік нанотүтікшелер, 3D металдар, шаблондық синтез, кристалды құрылым, магниттелу.

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Магнитная анизотропия FE $_X$ NI $_{100-X}$ нанотрубок в зависимости от их состава

Аннотация. В порах ионно-трековых мембран синтезированы ферромагнитные нанотрубки на основе никеля, железа и их сплавов. Определены составы электролитов, условия синтеза, позволяющие получить следующие составы нанотрубок: Fe, Fe80Ni20, Fe60Ni40, Fe20Ni80, Ni. Изменение состава нанотрубок существенно влияет на их кристаллическую структуру: с увеличением содержания никеля происходит трансформация типа решетки от ОЦК (характерного для Fe) к ГЦК (характерному для Ni), что сопровождается значительным изменением структурных параметров. Проанализирован процесс намагничивания массивов нанотрубок в зависимости от состава и кристаллической структуры, определены основные магнитные параметры. Проанализирован процесс намагничивания массивов нанотрубок в зависимости от состава и кристаллической структуры, определены основные магнитные параметры. Следовательно, векторы намагниченности лежат в стенках трубы случайным образом. Оценка основных магнитных параметров по петлям гистерезиса, измеренным в магнитном поле, направленном вдоль трубок и в перпендикулярном направлении, выявляет сложный процесс намагничивания, включающий вращение намагниченности и распространение доменных границ.

Ключевые слова: ферромагнитные нанотрубки, 3d-металлы, шаблонный синтез, кристаллическая структура, намагниченность.

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