06

Study of growth processes of one-dimensional Fe_xCo_{1-x} nanostructures and their influence on the magnetic properties of nanowire arrays

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A series of metal polymer composite samples based on Fe_xCo_{1-x} nanowire arrays with different elemental ratios and nanowire lengths were obtained. Using scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray diffraction analysis, the structure of the resulting nanowires and the dynamics of deposition kinetics during array growth, as well as the change in the Fe content in the nanowires depending on their length, were studied in detail. The dependence of the magnetic properties of the nanowire array on nanowire length and structural features was established.

Keywords: matrix synthesis, magnetic properties, nanowires, FeCo alloys, track membranes, growth kinetics, scanning electron microscopy, X-ray diffraction analysis.

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Ferromagnetic binary alloys of Fe-group metals are of particular interest, since their structural and magnetic properties may be fine-tuned by adjusting the ratio of elements in their composition [1]. Specifically, binary nanosized FeCo alloys have high saturation magnetization, weak magnetocrystalline anisotropy, and high magnetic susceptibility, which makes them promising for use in sensors [2]. On the contrary, one-dimensional nanoscale FeCo structures, such as nanowires, metal-polymer composites, and three-dimensional structures based on them have pronounced magnetic shape anisotropy and may be used to fabricate permanent magnets [3–5] and in magnetic recording devices [6]. One-dimensional FeCo nanostructures with a small diameter and the maximum saturation magnetization feature uniaxial anisotropy, which is induced by a pronounced contribution of magnetic shape anisotropy to the total effective anisotropy. Notably, the magnitudes of coercive force (H_c) in FeCo nanowires may be on the order of 1000 Oe and even higher [4,7,8].

Matrix synthesis is one of the most widely used methods for production of one-dimensional nanostructures of various types from Fe-group metals [9]. It involves electrochemical material deposition into the pores of a matrix. This method is not especially time- and labor-intensive and allows one to obtain all possible combinations of binary [7] and ternary [10] alloys of transition metals of the fourth period. However, the synthesis of nanostructures by this method has several known drawbacks. Owing to the specifics of electrochemical deposition processes, nanowires may have a non-uniform composition and, consequently, heterogeneous structural properties, which possibly affect the physical properties of nanostructures. The most significant effect here is anomalous Fe co-deposition, which is the accelerated deposition of Fe in the presence of other metals that causes a change in the elemental composition of nanowires [11,12]. In addition, the very deposition of material into the confined space of pores has a strong influence on the kinetics of deposition of metal ions [13,14] (e. g., accelerates or slows down their deposition due to local depletion of the diffusion layer [15] near the deposition region). This may result in inhomogeneities in the elemental composition, crystallite sizes, and geometric parameters of nanostructures throughout the entire volume of a metal-polymer composite consisting of a polymer matrix with an embedded array of nanowires.

The aim of the present study is to identify possible differences in the magnetic properties of arrays of FeCo nanowires with different ratios of elements and to determine the values of H_c of a metal-polymer composite based on nanowires as functions on the aspect ratio of the latter. The obtained data will enable consistent production of arrays of one-dimensional nanostructures and materials based on them with specific magnetic properties.

Arrays of one-dimensional nanostructures in the form of FeCo nanowires in polyethylene terephthalate (PET) matrices with a pore diameter of 100 nm produced by JINR (Dubna) were synthesized. Three electrolyte compositions with salts $CoSO_4 \cdot 7H_2O$ (16/16/2 g/l), $CoCl_2 \cdot 6H_2O$ (40/40/4 g/l), and $FeSO_4 \cdot 7H_2O$ (4/48/72 g/l) were used to examine the influence of Fe concentration on the structure and properties of the obtained nanowires. The concentration of salts was varied to obtain different ratios of metal ions,

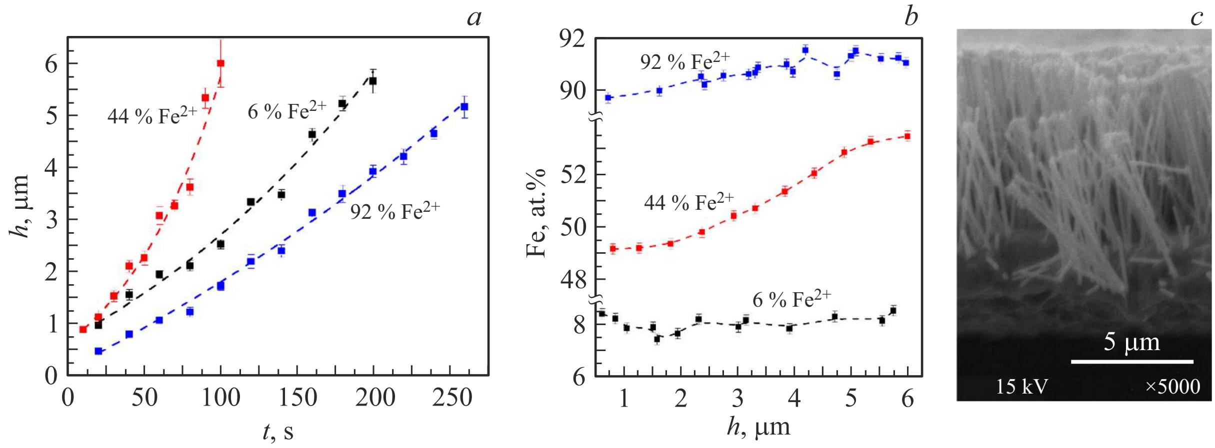

Figure 1. *a* — Dependence of the length of nanowires obtained from electrolytes with different fractions of Fe^{2+} ions on the time of their deposition; *b* — Fe distribution throughout the length; *c* — example array of nanowires.

namely the percentage ratio of Fe^{2+} or Co^{2+} ions to the total number of metal ions in the electrolyte: Fe^{2+} (6%)/Co^{2+} (94%), Fe^{2+} (44%)/Co^{2+} (56%), and Fe^{2+} (92%)/Co^{2+} (8%), respectively. Sodium lauryl sulfate (1 g/l) and boric (25 g/l) and ascorbic (1 g/l) acids were used as additives. Deposition was carried out under a constant cell voltage of 1.5 V with a Fe anode in a two-electrode arrangement [16]. A Cu film applied to one side of the matrix served as the cathode. The matrix area involved in the reaction was 2 cm^2. The electrolyte volume was 30 ml. The anode area was 6 cm^2, which was many times larger than the actual cathode area (~ 0.19 cm^2). The anode–cathode distance was 5.5 cm. A series of samples with different growth times were obtained for nanowires of each composition in order to examine the structural and magnetic properties of the resulting structures as functions of their length.

The morphology, length, and elemental composition of nanowires were investigated by scanning electron microscopy (SEM) using a JCM 6000 plus (JEOL, Japan) microscope with an energy-dispersive X-ray spectroscopy (EDX) attachment. In preparation for SEM studies, the PET matrix was etched in a 6N NaOH solution. The structural features of nanowires were examined via X-ray diffraction analysis (XRD) with a Miniflex 600 (Rigaku, Japan) powder X-ray diffractometer. In preparation for X-ray studies, the Cu cathode layer was etched to eliminate Cu reflections induced by the substrate. Measurements were carried out in the Bragg–Brentano geometry using CuK_α radiation (0.154 nm). The H_c values of samples were determined by vibration magnetometry (MV-07, Russia) in two field directions relative to the polymer matrix: along the normal to the plane (OOP) and perpendicular to it (IP).

SEM studies provided an opportunity to estimate the dependence of average lengths of the nanowire array on the deposition time (Fig. 1, *a*). An example microphotographic image of arrays of one-dimensional nanostructures is shown in Fig. 1, *c*. Dependences $h(t)$ of lengths of nanowires on their growth time are nonlinear in nature and are approximated well by exponential dependence $h(t) = A_1 \exp(t/t_0) + h_0$. This nanowire growth pattern may be shaped by a number of factors, such as the nonlinear kinetics of Fe and Co deposition in a limited pore volume that leads to changes in the ion concentration in the near-cathode space.

This assumption is presumably verified by the results of EDX analysis (Fig. 1, *b*), which demonstrate that the Fe content varies (increases) in the direction from the origin to the end of nanowires.

This non-uniform distribution of Fe along the length of nanowires may be attributed to an uneven supply of Fe^{2+} ions to the working area. At the onset of growth, the working area is depleted of ions due to their deposition, but a diffusion layer forms after that in pore channels and on the matrix surface, leading to an influx of ions into the working deposition area from the electrolyte volume. It should be noted that the rates of diffusion in pore channels and deposition of Co^{2+} and Fe^{2+} ions differ; the additional increase in concentration of Fe^{2+} ions due to anode dissolution is also worthy of note [17]. The difference in magnitude of changes corresponding to different compositions may be attributed to discrepant concentrations of deposited metal ions: this concentration is almost 2 times higher for the composition with 44% Fe^{2+}. However, the changes for each electrolyte composition are similar in nature.

The diffraction patterns of arrays of nanowires with the maximum length were analyzed in order to obtain in-depth data on the structural features of FeCo for the synthesized samples (Fig. 2). Peaks characteristic of phases based on BCC Fe are observed for nanowires obtained from an electrolyte with 44 and 92% Fe^{2+}. In the case of nanowires produced from an electrolyte with 6% Fe^{2+}, peaks of

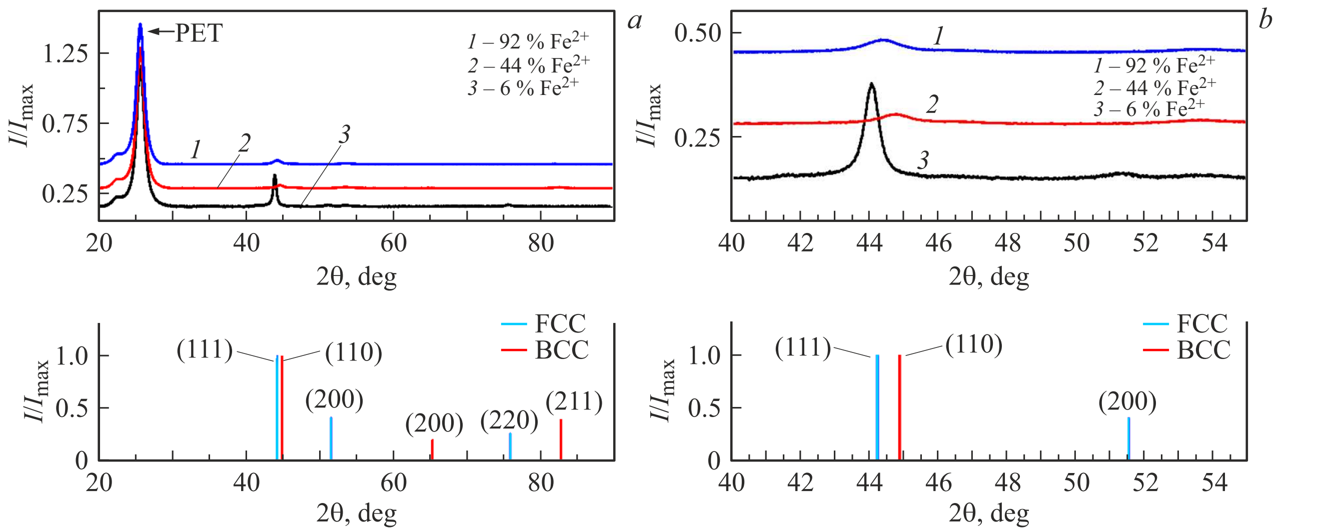

Figure 2. Diffraction patterns of nanowires of three compositions and line diagrams of BCC and FCC phases based on the Fe–Co system. $2\theta = 20-90^\circ$ (*a*) and $40-55^\circ$ (*b*).

FCC FeCo are seen. The obtained XRD data provided an opportunity to estimate crystallite size L, coherent scattering region (CSR) sizes D, and microstrain ε by the Williamson–Hall method (see the table).

It follows from Fig. 2 and the table that the crystallite and CSR sizes depend strongly on the Fe content in nanowires. An increase in concentration of Fe ions in the electrolyte may lead to an increase in contribution of the effects of anomalous Fe co-deposition, which, in turn, affect the growth kinetics and the size of crystallites of the array. The increase in microstrain at lower concentrations of Fe ions in the electrolyte may be associated with the size of crystallites in a limited pore volume: large crystallites may exert greater pressure on the pores and adjacent crystallites.

The dependences of H_c on length, which demonstrate a non-monotonic behavior (Fig. 3), were obtained by processing the vibration magnetometry data for nanowires of three compositions. It should be noted that the value of H_c for all compositions is greater in the IP position that corresponds to the direction perpendicular to the nanowire axis. This is indicative of a strong Magnetic dipole interaction of nanowires with each other [18].

The non-monotonic dependence of H_c in a rather complex system of nanowires enclosed in a polymer track membrane with a random arrangement of pores [19] may be induced by a variety of different factors. For example, magnetic anisotropy may be affected strongly by the interaction of nanowires with each other at their different lengths [20]. Another important factor is the variation of elemental composition and crystallinity along the length of nanowires, which was verified by EDX and XRD.

Thus, specific features of deposition of FeCo alloys with different ratios of elements were revealed. It was found that the distribution of Fe along the nanowire length varies nonlinearly with the Fe concentration increasing in the process of nanowire growth. It was also established that the crystallite size and the microstrain magnitude depend strongly on the Fe content in the nanowire array. As the Fe concentration increases, crystallite sizes and microstrain magnitudes tend to decrease. These effects are associated with the diffusion properties of Fe^{2+} ions and a local change in concentration of these ions near the working area due to depletion at the onset of growth with subsequent restoration due to anode dissolution. The magnetometry results revealed that the H_c values for all nanowire compositions depend non-monotonically on their length, which is attributable to variations of the elemental composition and average crystallite size along the length of nanowires and to their magnetic interactions with each other. Note also that the easy magnetization axis of nanowire arrays lies in the IP plane, which may be associated with strong magnetostatic interactions within the array.

The obtained data expand our understanding of the kinetics of electrochemical deposition of Fe-group alloys in a limited volume and may be used to obtain arrays of one-dimensional nanostructures with specified magnetic parameters and their anisotropy for various applications.

Structural parameters of the obtained nanowires

Fe^{2+} content of the electrolyte, %	Crystallite size L, nm	CSR size D, nm	$\varepsilon \cdot 10^3$
6	17	12	7.4
44	10	8	5.3
92	7	7	1.6

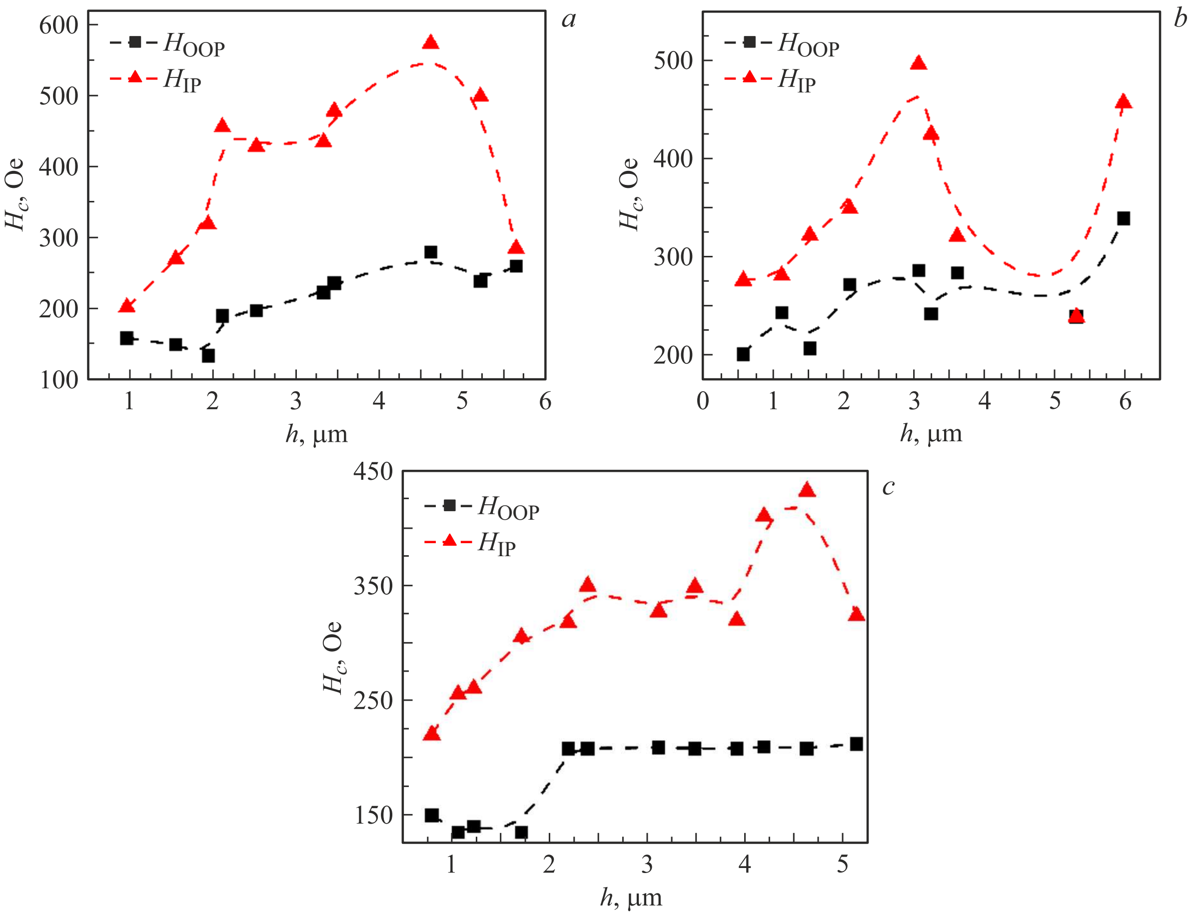

Figure 3. Dependence of the coercive force of nanowires of different compositions on their length. *a* — 6% Fe^{2+}, *b* — 44% Fe^{2+}, *c* — 92% Fe^{2+}.

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Conflict of interest

The authors declare that they have no conflict of interest.

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