

National Research University Higher School of Economics

as a manuscript

Doludenko Ilia

**Investigation of the formation, structure and physical properties of nanowires
of complex composition**

Dissertation summary

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Professor Bondarenko G.G.

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Relevance of the research topic

Currently, there is an increasing interest in nanomaterials and structures based on them. Many works of both fundamental and applied nature are devoted to the study of the methods of obtaining them, the study of their structure and properties. The variety of methods of obtaining, structure and properties of nanostructures provides a wide field of their applications.

One of the types of nanostructures with great potential in practical applications are one-dimensional nanostructures or nanowires (NW). There are a number of methods for obtaining them. One of them is matrix synthesis. The main idea is as follows: At the moment, a large volume is one-dimensional magnetic nanoparticles. This is due to the use of the ever-increasing pace of studying nanoscale, quantum and spin effects, and the need for new materials and structures for them in chemistry, radio electronics, microelectronics and medicine.

The matrix synthesis method has been known since the 90s of the twentieth century, however, many aspects of the preparation procedures, structural features and functional particles are found in full. Most of the work is detected over a wide range of reception modes, not fixed for the observed relationship and the ability to predict the detected structure and properties. Establishing the dependence of structures and, as a result, the properties of the nanostructure on the modes of their reception, as well as the creation of increased sensitivity to geometric and structurally high parameters of approximate studies in the field of practical application in various fields and technology, which determines the relevance of the topic of this study.

The degree of development of the research topic

As noted above, “the method of matrix synthesis is based on filling a pre-prepared matrix with the necessary material. Porous alumina (POA) [1] and track membranes (TM) [2] are most often used as a matrix for creating one-dimensional nanoparticles. These types of matrices differ in the methods of preparation and, as

a result, in physicochemical properties, geometry and joint arrangement of pores in the volume of the matrix, and, most importantly, in the ability to control and change individual parameters in a controlled manner. Thus, TM, based on a polymer film, are flexible and elastic, which makes it possible to use structures based on them in elements of microelectronics and radio electronics [3]. The process of obtaining them implies the ability to controllably change the pore diameter and, accordingly, the diameter of the obtained nanoparticles. It should be noted that such changes in the diameter can be independent of the pore density, which cannot be fully achieved when creating matrices from POA [4]. Another advantage of TM is the possibility of changing the angle of inclination of the pores with respect to the plane of the matrix during its preparation, while maintaining the specified parameters throughout the volume. In addition, the process of obtaining TM is in-line and has been developed at the Joint Institute for Nuclear Research (JINR, Dubna), while maintaining the geometric arrangement of pores in the matrix and their selectivity in diameter, which is an important circumstance in the creation of nanoparticles, for example, for medical applications [5]. Based on the above, track membranes were chosen as the matrix in this work.

The main method for filling matrices to obtain magnetic nanowires is the method of electrochemical deposition. This method is distinguished by the ability to change the structural parameters of the obtained materials, thereby changing their properties, as well as to obtain a wide range of NW materials [6, 7].

Promising materials for creating magnetic nanoparticles are iron-based alloys, namely alloys with cobalt or nickel [8]. For bulk materials, it is known that by varying the ratios of these elements, one can obtain completely different magnetic properties. Thus, the Fe₂₀Ni₈₀ alloy (permalloy) is magnetically soft, and the alloy with an equiatomic ratio of iron and cobalt is a magnetically hard alloy. It seemed interesting to find out whether the above magnetic properties are characteristic of the nanosized structures of these alloys. To do this, it was necessary to develop methods for controlling the composition and structure of the obtained nanoobjects. It should be noted that the method of electrochemical deposition allows one to vary

the ratio of deposited metal ions in the electrolyte; this ratio is not always preserved in the deposited material. Thus, the process of bulk materials is characterized by the effect of anomalous co-precipitation of iron. It lies in the fact that during electrochemical deposition from solutions containing ions of several metals, one of which is Fe^{+2} , the ratio of deposited atoms of various ions will differ from the ratio of ions in the electrolyte in the direction of increasing iron. The question of the manifestation of this effect during deposition into the pores of track membranes is complicated by the diffusion restrictions imposed on the matrix pores. This raises a number of questions, such as whether this effect will be retained during deposition in a limited volume, how this effect will depend on the type of deposited ions, their ratio and deposition rate.

Another feature of the electrochemical method of filling matrix pores is the possibility of layer-by-layer deposition of a number of metals; it is implemented due to the difference between the equilibrium deposition potentials of various metals [9]. A material with a lower equilibrium potential with a uniform increase in the deposition potential will begin to precipitate earlier; in this case, the process of deposition of the second type of ions will not proceed. Only after the value of the equilibrium potential of the second metal is exceeded will the ions of two different metals co-precipitate. The selection of the ratio of ions in the electrolyte makes it possible to minimize the impurity content at a higher potential. The selection of deposition modes and electrolytes makes it possible to obtain layer-by-layer deposits with a sharp switching of potentials. However, the use of this method for obtaining nanowires is associated with a number of problems, such as determining the optimal conditions for the deposition of individual layers, developing methods for controlling the layer thicknesses to obtain a periodic structure with conserved geometric and structural parameters, etc. [10].

In the light of the stated state of the degree of elaboration of the topic, this thesis is devoted to the study of the features of filling track membranes by the electrochemical method to obtain nanowires from iron-based alloys and heterostructural nanowires in order to establish dependencies between production

modes, structure and properties, which in the future will allow them to be used to create flexible elements. microelectronics and as components of systems for local delivery of drugs to the focus of disease in the body.

Purpose and objectives of the study

The purpose of this work is to reveal the patterns of formation, morphology, growth kinetics, and the influence of various factors on the structure and properties of homogeneous and heterostructural nanowires of a given geometry.

To achieve this goal, the following tasks were solved in the work:

- development of methods and development of modes for obtaining homogeneous and heterostructured nanowires with a given composition and uniform distribution of elements;
- establishing patterns of formation, morphology and growth kinetics of nanowires of complex composition;
- development of a method for controlling the thickness of layers of heterostructural nanostructures;
- revealing the patterns of changes in the physical properties of nanowires depending on their geometry, composition and structure.

Scientific novelty of the dissertation work

- A new method for obtaining homogeneous nanostructures consisting of Fe-Ni and Fe-Co magnetic alloys has been proposed and practically implemented.
- For the first time, experimental data on the growth kinetics of nanostructures from Fe-Ni alloys were obtained and a model for the growth of nanostructures was proposed on their basis, according to which it is possible to predictably control the geometry of nanostructures;
- For the first time regularities of formation of cylindrical magnetic nanoparticles and changes in their structure depending on the mode of electrodeposition were established; when obtaining nanostructures from Fe-Ni alloys, the effect of anomalous (up to 35%) deposition of iron ions into the pores of the matrix was discovered, and a physical mechanism of the effect was proposed;

- Regularities of changes in the electrical properties of arrays of nanoparticles depending on their aspect ratio (X) were revealed, while a decrease by an order of magnitude (from 10^6 to 10^5 Ohm) in the electrical resistance of a metal-polymer composite based on the resulting nanoparticles was found with an increase in X from 20 to 70 units;
- A method for controlling the thickness (30-500 nm) of layers of heterostructural nanowires at the growth stage has been proposed and practically implemented, which is distinguished by the possibility of obtaining layers of a given geometry along the entire length of the nanowire;
- For the first time, a method was proposed for obtaining a suspension of calibrated cylindrical magnetic nanoparticles for local drug delivery in the human body, based on the use of anisotropic magnetic nanoparticles;
- Regularities of changes in the electrokinetic potential (zeta potential) of a suspension of cylindrical magnetic nanoparticles (CMNP) depending on their aspect ratio were revealed. It was found that the change in the electrokinetic potential is inversely proportional to the increase in the aspect ratio of CMNP.

Theoretical significance of the work

The theoretical significance of the dissertation lies in expanding the range of knowledge about the processes of obtaining, formation, growth kinetics and physical properties of complex nanostructures; an array of experimental data was obtained, regularities of the studied processes were established and analyzed, changes under the influence of various factors of the structure and properties of homogeneous and heterogeneous nanowires of the iron-nickel, iron-cobalt, copper-nickel, copper-cobalt systems. As a result of the analysis of the experimental data obtained in the study of the formation of nanostructures, a model for the growth of nanostructures is proposed and it is shown that this model can be used for predictable control of their geometry during growth. When obtaining nanostructures from Fe-Ni alloys, the effect of anomalous electrodeposition of iron ions into the pores of the polymer matrix was discovered, and a physical mechanism was developed that adequately explains this phenomenon.

The practical significance of the work

The practical significance of the work lies in the fact that the developed methods and the dependences obtained will make it possible in the future to obtain one-dimensional nanoparticles and structures based on them with specified structural, geometric parameters and physical properties. The developed methods make it possible to obtain NP arrays from magnetic alloys with a given composition and structure, which makes it possible to widely vary their magnetic properties. The method described in this work for obtaining a suspension of calibrated cylindrical magnetic nanoparticles makes it possible to obtain nanoparticles that can be used in medicine for local drug delivery to the focus of a disease in the body, as components of substrates in the study of giant Raman scattering, magnetic heating devices, etc. The resulting metal-polymer composites can be used to create sensors, sources of electromagnetic waves, and also as elements of flexible microelectronics and spintronics. An important circumstance that determines the practical application of the results obtained in the work is also the fact that the matrix used is produced by a well-mastered flow method, which makes it easy to scale the obtained volumes of nanoparticles and nanostructures based on them.

Research methods

In the work, one-dimensional nanoparticles from magnetic and non-magnetic metals were obtained and studied. The preparation method was based on matrix synthesis, namely, on the galvanic filling of the pores of track membranes. Track membranes manufactured by JINR, Dubna were used as matrices for creating NW. The matrices were produced by irradiating thin polymer films of polyethylene terephthalate with heavy ions of inert gases at a U-400 accelerator, followed by etching of latent tracks. Irradiation was carried out with a spread of pore inclination angles up to 30° in the direction of film rolling. This was necessary to increase the irradiation density while maintaining a constant pore diameter. “The

film thickness was 12 microns. The radiation dose was $1.2 \cdot 10^9 \text{ cm}^{-2}$. The pore diameter was varied due to the process of etching latent tracks and was 100 nm in most works [11].

For the synthesis of nanowires, we used the method of galvanic deposition into the pores of track membranes. The process was carried out in a special galvanic cell in which the anode and matrix were placed vertically. “The area of the track membrane sample into which the deposition was carried out was 1.8 cm^2 ; thus, the area of the working electrode (the surface area of the pores of the matrix) was 0.17 cm^2 . A potentiostat - galvanostat Elins R-2X was used as a source. The deposition potentials varied from 0.5 to 2 V. The process was carried out according to a two-electrode scheme. Electrodeposition and control of the growth process were carried out using an Elins P-2X programmable potentiostat-galvanostat [12]. To perform structural studies, a complex of several instruments was used for conducting scanning, transmission electron microscopy with attachments for elemental analysis, X-ray diffraction analysis. The morphology of the resulting nanoobjects, their composition, and growth rate were studied by scanning electron microscopy (SEM) using a JEOL JCM-6000plus scanning electron microscope equipped with an attachment for elemental analysis. X-ray diffraction studies were carried out on a MiniFlex-600 X-ray powder diffractometer [13].

The measurement of the electrokinetic potential (zeta potential) resulting from the accumulation of electric charges at the interface between the solid and liquid phases was carried out using an automatic analyzer Zetasizer Nano ZS (Malvern, UK) [14].

Provisions for defense

- A new method for obtaining homogeneous nanostructures consisting of Fe-Ni and Fe-Co magnetic alloys;
- experimental data on the growth kinetics of nanostructures from Fe-Ni alloys and the model of nanostructure growth proposed on their basis;
- patterns of formation of cylindrical magnetic nanoparticles and changes in their structure depending on the mode of electrodeposition; the effect of anomalous

(up to 35%) deposition of iron ions into the pores of the matrix found during the preparation of nanostructures from Fe-Ni alloys, the physical mechanism of the effect;

- patterns of change in the electrical properties of arrays of nanoparticles depending on their aspect ratio;

- a new method for controlling the thickness (30-500 nm) of layers of heterostructural nanowires at the growth stage;

- a new method for obtaining a suspension of calibrated cylindrical magnetic nanoparticles for local drug delivery in the human body.

- patterns of change in the electrokinetic potential (zeta potential) of a suspension of cylindrical magnetic nanoparticles depending on their aspect ratio;

Reliability of the obtained results

The reliability of the experimental results presented in the dissertation work is confirmed by their reproducibility, as well as the use of modern experimental equipment, independent research methods: transmission electron microscopy, scanning electron microscopy, transmission scanning electron microscopy, electron diffraction, energy dispersive analysis, X-ray diffraction analysis. The interpretation of the obtained data is based on modern ideas about the formation processes and growth mechanisms of nanowires. The developed technical solution for the method of obtaining nickel nanowires was patented: a patent of the Russian Federation for the invention was issued.

Author's personal contribution

The author's personal contribution to the work consists in the study of the processes of obtaining nanowires and structures based on them, the development of a growth model for the studied nanostructures, the study of their morphology, structure, elemental composition by scanning electron microscopy with an attachment for elemental analysis and X-ray diffraction analysis, including sample preparation; development of electrodeposition modes for obtaining layered nanowires with controlled and constant layer thickness, as well as methods for selective etching of heterostructured nanowires to obtain a suspension of calibrated

magnetic nanoparticles; participation in the preparation and conduct of research on the magnetic and electrical properties of arrays of obtained nanowires, suspensions of nanoparticles and structures based on them; analysis of the obtained results; formulation of conclusions on the dissertation work.

Approbation of results

The main results of the dissertation were presented at the following national and international conferences:

1. 2017 Materials Research Society Spring Meeting and Exhibit, Report «Fabrication of aligned carbon nanotubes and magnetic nanowires using porous polymer template», Phoenix, USA, 2017, 17 – 21 April.
2. Magnetism 2019, Report «Template synthesis and magnetic properties of two-component nanowires», UK, Leeds, 2019, 8-9 April.
3. E-MRS 2019 Fall Meeting, 2019. Report « Nanowires of FeNi and FeCo alloys: matrix synthesis and structure», Poland, Warsaw, 2019, 16 – 19 September.
4. International Conference on Materials for Energy Applications (ICME-18), Report «Template synthesis of heterostructural nanowires Cu/Ni», India, Jaipur, 2018, 6 – 8 December.
5. The 9th International Conference on Physical and Numerical Simulation of Materials Processing (ICPNS'2019). Report «Characterization of nanowires of FeNi and FeCo alloys», Russia, Moscow, 2019, 10-14 October.
6. International Baltic Conference on Magnetism 2021. Report «FeNi and FeCo alloys nanowires: synthesis, structure and magnetic properties», Russia, Svetlogorsk, 2021, 29 August - September 2.
7. International Conference on Nanophysics and Nanoelectronics 2018 [in Russian]. Report «Production of layered nanowires, study of their structure and magnetic properties [in Russian]». Russia, Nizhny Novgorod, 2018, 12 – 15 March.
8. International Conference on Nanophysics and Nanoelectronics 2019 [in Russian]. Report «Structure of multilayer nanowires with alternating magnetic and nonmagnetic metals [in Russian]». Russia, Nizhny Novgorod, 2019, 11 – 14 March.

9. International Conference on Nanophysics and Nanoelectronics 2020 [in Russian], Нижний Новгород, Россия. Report «FeNi and FeCo nanowires: synthesis, structure and Mössbauer spectra [in Russian]». Russia, Nizhny Novgorod, 2020, 10 – 13 March.
10. XXVII International Conference "Radiation Solid State Physics" [in Russian]. Report «Application of track matrices for obtaining heterostructured Ni/Cu nanowires [in Russian]». Russia, Sevastopol, 2017, 10 - 15 July.
11. XXVIII International Conference "Radiation Solid State Physics" [in Russian]. Report «Application of nuclear filters to obtain layered nanowires [in Russian]». Russia, Sevastopol, 2018, 9 - 14 July.
12. XXIX International Conference "Radiation Solid State Physics" [in Russian]. Report «Electrochemical filling of nuclear filters (track membranes) for obtaining nanowires of variable composition [in Russian]». Russia, Sevastopol, 2019, 8 - 13 July.
13. XXX International Conference "Radiation Solid State Physics" [in Russian]. Report «Matrix synthesis and investigation of FeNi alloy nanowires [in Russian]». Russia, Sevastopol, 2020, 7 - 12 July.

List of published articles on the topic of the dissertation

The main provisions on the topic of the dissertation are set out in 6 publications indexed in the international Scopus system:

1. Structure of Cu/Ni Nanowires Obtained by Matrix Synthesis // O. M. Zhigalina, I. M. Doludenko, D. N. Khmelenin, D. L. Zagorskiy, S. A. Bedin, I. M. Ivanov / Crystallography Reports, 2018, Vol. 63, No. 3, pp. 480–484. DOI: 10.1134/S1063774518030379 (Q3);
2. Structure and Magnetic Properties of Nanowires of Iron Group Metals Produced by Matrix Synthesis // Zagorskiy D.L. ; Frolov K. V.; Bedin S. A.; Perunov I. V.; Chuev M. A.; Lomov A. A.; Doludenko I. M. / Physics of the solid state. Vol. 60, No. 11 pp. 2115-2126 (2018) DOI: 10.1088/1742-6596/1134/1/012071 (Q3);
3. Specific Features of Obtaining of Metal Nanowires by Replication of Pores of Track Etched Membranes // D. Zagorskiy, I. Doludenko, A. Shatalov/ Key Engineering Materials, 2018, Vol. 781, pp. 170-175. (DOI: <https://doi.org/10.4028/www.scientific.net/KEM.781.170>) IF: 0.39 (Q3);

4. Fabrication of Cylindrical Magnetic Nanoparticles for Functionalization of Polyelectrolyte Microcapsules // I. M. Doludenko, A. V. Mikheev, I. A. Burmistrov, D. B. Trushina, T. N. Borodina, T. V. Bukreeva, D. L. Zagorskii / *Technical Physics*, 2020, Vol. 65, No. 9, pp. 1377–1383. DOI: 10.1134/S1063784220090121 (Q3);
5. I. M. Doludenko. Aspects of Pore Filling in Synthesis of FeNi Alloy Nanowires Using Track-Etched Membranes / I.M. Doludenko // *Inorganic Materials: Applied Research*, 2022, Vol.13, No.2, pp. 531-535 DOI: 10.1134/S2075113322020125 (Q2);
6. I.M. Doludenko, I.S. Volchkov, B.A. Turenko, I.O. Koshelev, P.L. Podkur, D.L. Zagorskiy, V.M. Kanevskii, Electrical properties arrays of intersecting of nanowires obtained in the pores of track membranes, *Materials Chemistry and Physics* (2022), doi: <https://doi.org/10.1016/j.matchemphys.2022.126285>(Q2).

Intellectual property objects

Doludenko I.M., Zagorsky D.L., Trushina D.B., Burmistrov I.A. A method for producing nickel nanorods with a regulated aspect ratio. Patent for invention No. 2724264, 06/22/2020.

Conclusion

The following results were obtained in the dissertation research:

- a new method has been developed for producing homogeneous nanostructures with a high aspect ratio (up to 150 units) consisting of Fe-Ni and Fe-Co magnetic alloys, characterized by the ability to control the composition of alloys in increments of 10%;
- the dependences of the composition of nanowires on the composition of the electrolyte were determined; when studying nanowires from Fe-Ni alloys, the effect of abnormal (up to 35%) electrodeposition of iron ions into the pores of the matrix was found, unlike nanostructures from Fe-Co alloys, where this effect does not exceed 5%; the observed effect depends

on the composition of the electrolyte and increases with an increase in the number of iron ions in the electrolyte;

- when studying the effect of the electric deposition potential on the elemental composition of the nanowire array, it was found that for Fe-Ni alloys, a decrease in the electric potential (from 1.5 to 1.0 V) leads to a 10-20% increase in the number of Fe atoms in the nanowires; this effect increases with a decrease in the relative concentration of Fe^{+2} ions in electrolytes; a physical mechanism of the effect is proposed;
- when determining the nature of the distribution of elements in the obtained nanowires, it was found that in the case of Fe-Co alloys it is uniform, while in Fe-Ni the amount of iron at the final stage of nanowire growth is greater than at the initial stage; the magnitude of this effect increases with a decrease in the electric deposition potential;
- the regularities of changes in the electrical properties of nanoparticle arrays depending on their aspect ratio (X) are revealed; a decrease by an order of magnitude (from 10^6 to $10^5 \Omega$) in the electrical resistance of a metal-polymer composite based on the obtained nanoparticles with an increase in X from 20 to 70 units is established;
- the dependences of the electrical resistance of a metal polymer composite on the degree of filling of the matrix with $\text{Fe}_{20}\text{Ni}_{80}$ alloy are obtained; calculations of the probability of crossing pores and, accordingly, nanowires in the matrix depending on the degree of its filling are performed; correlation of experimental and calculated data is found;
- a new method has been developed for obtaining and controlling the thickness (30-500 nm) of layers of heterostructural nanowires, characterized by the possibility of forming layers (copper /nickel, copper/cobalt) while preserving structural and geometric parameters along the entire length of the nanowire;
- The regularities of changes in the electrokinetic potential (zeta potential) of a suspension of cylindrical magnetic nanoparticles (CMNP) depending

on their aspect ratio are revealed. At the same time, it was found that the change in the electrokinetic potential is inversely proportional to the increase in the aspect ratio of the CMNP;

- a method has been developed for separating heterostructural nanowires along a copper layer to obtain a suspension of cylindrical magnetic nanoparticles of calibrated size (from 30 to 500 nm in length); a method for producing nickel nanorods with an adjustable aspect ratio is protected by a patent; these particles can be used in medicine as components of polyelectrolyte microcapsules for local delivery of drugs of high toxicity.

Recommendations, prospects for further development of the topic

- The resulting metal-polymer composites or individual nanowires can be recommended for use in the creation of sensors, sources of electromagnetic waves, as well as as elements of flexible microelectronics and spintronics;
- Cylindrical magnetic nanoparticles can be used in medicine as components of polyelectrolyte microcapsules for local delivery of highly toxic drugs to the patient's body and as components for magnetic heating.

In further studies, it is expected to evaluate the effect of the geometry of the location of pores and their density on the electrical properties of metal-polymer composites, as well as to obtain the dependence of their magnetic properties on the degree of filling. In addition, it is planned to work out methods for modifying the resulting metal-polymer composites with magnetically sensitive components to improve their sensory properties and create logic cells. As part of the development of the CMNP research direction, it is planned to conduct experiments on the opening of polyelectrolyte microcapsules with embedded magnetic particles in the patient's body, as well as testing the possibility of improving the processes of introducing particles through the selection of material and external influences.

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