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Synthesis features, structure, magnetometry and NMR spectroscopy of nanowires of various types

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Various types of nanowires obtained by matrix synthesis — homogeneous (from iron) and heterogeneous (layered) — have been studied. A technique for obtaining arrays of layered nanowires with alternating thin layers of magnetic and non-magnetic metals (Co/Cu, Ni/Cu) has been developed and described. Microscopy methods (SEM and TEM with elemental analysis) have been used to study the topography of the resulting structures, the diameters of nanowires and the thicknesses of individual layers, and the features of interlayer interfaces. Methods of synthesis of nanowires with thin layers and clear boundaries are proposed — dilution of the electrolyte, use of a reference electrode, control of the leaked charge. Layered nanowires have been studied by magnetometry methods and it has been shown that the magnetic properties of an array of layered nanowires (in particular, the direction of the axis of light magnetization in the Co/Cu-NP array) depend not only on the aspect ratio of the magnetic layer, but also on the ratio of the thickness of the magnetic metal layer to the thickness of a non-magnetic spacer (copper layer). The nuclear magnetic resonance (NMR) method was used to study two types of nanowires. The NMR method (on 59-Co nuclei) studied the layer structures of Co/Cu: it is shown that in nanowires with layers of smaller thickness (and, accordingly, with a large contribution of interfaces), a large proportion of Co atoms coordinated by Cu atoms is observed. The high proportion of atoms coordinated by copper suggests that an admixture of copper enters the cobalt layers. Homogeneous iron nanowires (NMR on 57-Fe nuclei) were compared with bulk iron samples. A shift of the line towards high frequencies (by 0.3 MHz) was detected, indicating an increase in the field by about 0.2 T. A significant broadening of the line and a decrease in the spin-lattice relaxation time may indicate a significant variation in the local magnetic field values.

Keywords: nanowires, matrix synthesis, microscopy, elemental analysis, magnetic properties, NMR.

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1. Introduction

One-dimensional nanostructures-nanowires (NW) are of great interest, in particular as materials with special magnetic properties. The uniqueness of the characteristics is determined by both the small size (nanoscale magnetism) and the high aspect ratio (determining the form anisotropy). One way of producing such structures is matrix synthesis — electrochemical metal filling of pores in a special matrix (template). Porous aluminium oxide [1] or polymer track membranes [2] are the most commonly used matrices. The parameters of the obtained structures are typically arrays of many parallel NW connected by a common base and/or in a growth matrix — are determined by the matrix, growth electrolyte composition and growth conditions. The matrix synthesis allows for a wide range of geometric parameters (diameter, orientation and location) and composition. The development of the method proceeded in the direction of complexity of synthesized structures: initially, homogeneous NW were produced from one metal, then homogeneous

NW from alloys of two or more elements. The next step was to get heterogeneous NW structures consisting of periodically alternating layers of different compositions. Note that in all cases, the most important parameter (besides the matrix and the composition of the electrolyte) is the growth voltage, which for each metal correlates with its equilibrium deposition potential. The features of obtaining such structures by matrix synthesis are described in many studies — we can note the fundamental reviews of [3,4] and the Russian study [5].

Different types of NW can be used for a variety of purposes. It seems that the most interesting are the magnetic properties of NW, for which samples are synthesized from metals of iron group. Below are some effects and their applications.

The GMR effect (giant magneto-resistance — modification of the electroresistance in the external magnetic field) was detected first in the flat layers of Fe/Cr [6], and then in the layered NW [7]. The effect quickly found practical application (recording heads) and its authors were awarded the Nobel Prize (2007). The effect has been investigated by many authors — note the study of the GMR value from the ratio of thicknesses of Co and Cu [8].

The use of high-density magnetic recording was proposed as early as the end of the last century — see, e.g., [7]. In work [9] this idea was developed: it was proposed to increase the density of the record by using different regions of a single NW (e.g., the layered one). The authors of [10] suggested using the „division“ of binary NW to stabilize their magnetic state.

In a number of studies the features of the application of layered NW in microelectronics and spintronic is observed. It has been noted that a critical value may be the thickness of the layer: in some cases, it should be less than the length of the electron mileage or the length of spin relaxation: often this value should not exceed 5–10 nm. The immutability (uniformity) of this parameter along the length of the NW is also essential. Equally important is the nature of the boundary between the layers (interface): the boundary should be sufficiently clear (without diffuse areas) and flat. Monitoring of these parameters — both by direct methods (microscopy) and indirect – seems very important.

Of particular interest are the magnetic properties of NW and the possibility of their modification. In studies [11,12], NW were studied with alternating layers of iron or cobalt with copper layers. It is shown that the nature of interaction and magnetic properties depend on the thickness of the nonmagnetic layer — the spacer. The authors of [13] have proposed the use of layered NW consisting of two different magnetic metals to generate terahertz frequency electromagnetic radiation. In study [14] it was proposed to use layer-based NW consisting of magnetic metal (nickel) and a non-magnetic layer („sacrificial“ layers of copper) to produce cylindrical magnetic nanoparticles. (The latter may be used for targeted drug delivery and opening of the polymer capsule.)

The application of nuclear resonance techniques to assess the magnetic state is very promising. Thus, the Mössbauer spectroscopy method allows to determine the magnitude of the field on the kernel and change this parameter when the local environment of the kernel and magnetization changes. By this method, ferrous specimens (FeCo and FeNi NW) have been studied in works [15,16]. The influence of growth voltage (and thus growth rate) and NW diameters on the parameters of the spectrum was determined and correlation of the latter with X-ray crystalline data was shown. The ratio of lines in the sextet showed that when the diameter of the NW is reduced to 30 nm the direction of magnetization of the NW almost coincides with its axis. The field value at the kernel was estimated to be significantly higher in iron-cobalt alloys than in iron-nickel alloys. For both formulations, an increase in the field at the kernel was detected as iron concentrations increased.

However, it is known that the Mössbauer spectroscopy allows to estimate the magnetic field profile only approximately — by solving the opposite problem. Another method, NMR, can determine the distribution of the magnetic field directly (in the zero field, by frequency shift). Cobalt-containing NW have been studied in work [17] by NMR and X-ray diffraction analysis; it has been shown that either hexagonal or cubic structure can be obtained depending on growth conditions (primarily speed). Crystallite sizes were estimated. However, the NW in this work was obtained in a different way — growth was conducted in a matrix of polycarbonate. In work [18] the study of NMR spectra ^{59}Co in cobalt NW with copper impurity obtained in the matrix from PET has been initiated and it has been shown that the appearance of impurities causes the appearance of new lines in the spectrum. The method of estimation of impurity quantity by parameters of spectrum is offered. It should be noted that, in general, there is very little work in which the NMR method was used to study NW arrays.

2. Experiment and results

2.1. Materials and instruments

In the work, the growth (template) matrices used track membranes with pores of 100 nm diameter and a surface density of 10^9 pores per cm^2 (produced by Joint Institute for Nuclear Research, Dubna). One of the surfaces of the polymer membrane was metallized with a thin layer of copper to create an electrically conductive layer. For the galvanic process, most of the electrolytes with sulfate salts of the respective metals were used. Additives were also used: buffer, for wetting and to prevent iron ions transition from bivalent to trivalent state.

The growth of NW masses was conducted in a galvanic cell. The source was the ELINS potentiostat-galvanostat. For the growth of homogeneous NW (from pure metals or from alloys), a potentiostatic mode was used, in which case the current-to-voltage dependence was recorded to control the process in real time. At the growth of heterogeneous

Electrolyte compositions and growth modes for different types of NW

<table>
<tr><th>№</th><th>Precipitable metal</th><th>Salt of metal</th><th>Conc., g/l</th><th>Additives</th><th colspan="2">Deposition potential, V</th></tr>
<tr><td>1</td><td>Fe</td><td>$FeSO_4 \cdot 7H_2O$</td><td>120</td><td>Boric acid (40 g/l);
Sodium Lauryl Sulfate (0.5 g/l);
Ascorbic acid (1 g/l)</td><td colspan="2">1</td></tr>
<tr><td rowspan="2">2</td><td rowspan="2">Ni/Cu</td><td>$NiSO_4 \cdot 7H_2O$</td><td>196.5</td><td rowspan="2">Boric acid (30 g/l)</td><td>For Ni</td><td>1.8 layer</td></tr>
<tr><td>$CuSO_4 \cdot 5H_2O$</td><td>6.3</td><td>Two layers Cu</td><td>0.6</td></tr>
<tr><td rowspan="2">3</td><td rowspan="2">Co/Cu</td><td>$CoSO_4 \cdot 7H_2O$</td><td>200</td><td rowspan="2">Boric acid (30 g/l)</td><td>For Co</td><td>1.5 layer</td></tr>
<tr><td>$CuSO_4 \cdot 5H_2O$</td><td>5</td><td>For Cu</td><td>0.6 layer</td></tr>
</table>

(layered) NW, pulses of voltage of different size and duration were used. (Options for process control in this case are described in the Results section).

Data on electrolyte compositions and growth modes of different types of NW are given in the table.

It should be noted that the deposition time for the NW from Fe was chosen from the chronoamperogram, and accounted for 90% of the time before the first overgrowth to the matrix surface. In the case of deposition of the NW layer, the time for the deposition of a certain thickness layer was determined by the charge run-off (see below). For example, a flow of 135 mC was required for a copper layer 100 nm thick and for a cobalt or nickel layer of the same thickness — of 140 mC. The chosen method kept the geometric parameters of the layers constant throughout NW.

Note that after the galvanic process, the resulting array of NW either separated from the growth matrix or remained in it. The polymeric matrix was separated before electron-microscopic studies were carried out, and other studies (magnetic and NMR) were carried out on the array of NW in the matrix.

The scanning microscope JEOL 6000 with the element analysis unit was used. The microscope operated in the mode of secondary and elastic reflected electrons, at accelerating voltage of 15 kV.

Also used was the FEI Tecnai Osiris Translucent Microscope with 200 kV Acceleration Voltage (high-resolution electron microscopy mode (VREM), Scanning Raster Electron Microscopy (PREM), with electronic diffraction mode). The elemental analysis was carried out with the help of a special system SuperX EDS, which includes four silicon detectors, the design of which allows you to shoot the large distribution maps of chemical elements in a few min.

The magnetic properties were investigated by vibration magnetometry on the coercive spectrometer [19] which allowed field dependencies of both the induced and residual magnetization of the specimen to be recorded in one measurement cycle. The magnetic hysteresis curves were recorded at room temperature at two limit orientations of the scanning magnetic field: parallel or perpendicular to the growth axis of the NW (respectively, relative to the matrix plane, in the out-of-plane and in plane geometry).

In cobalt-containing NW at the kernel ^{59}Co, the NMR spectra in the 140−250 MHz band were studied. The spectra were taken on a pulse NMR spectrometer at a temperature of liquid helium (4.2 K) in a local magnetic field — the external magnetic field was absent. The spin echo signal is formed by a sequence of two coherent radio frequency pulses $(\tau_{\mathrm{p}})_x - t_{\mathrm{del}} - (\tau_p)_y - t_{\mathrm{del}} -$ echo, which create a variable magnetic field with the amplitude of the circular component $H_1 \approx 10$ Oe in the resonant coil with the sample. The pulse duration of τ_p was 0.5 μs, the time between the pulses of t_{del} was 11 μs. The frequency change was approximately 1 MHz.

Measurements of NMR spectra on the nuclei ^{57}Fe (for iron-based NW) were made on a spectrometer based on a highly upgraded Bruker MSL-300 [20]. The signal was detected by the classic Khan spin echo method at a step-by-step carrier frequency change of F. The resulting spectrum was defined in two different ways: (i) as the integral of spin echo and (ii) as the sum of the Fourier images of the second half of echo [21].

3. Results and discussion

3.1. NW synthesis and microscopic layer control

The peculiarities of the production of layered NW with different layers of thickness were studied. The main tasks to be solved in this process are — control of the composition and length of different layers, control of the boundary between layers — interface. In the present work, a single-bed method was used to produce layered NW. In this case, during a low-voltage pulse, a metal with a low equilibrium potential (copper) grows, and during a higher-voltage pulse, magnetic metal (cobalt or nickel) precipitates predominantly. In this case, the ratio of metals in the magnetic layers of the substrate is determined by the ratio of concentrations in the growth electrolyte. Thus, to reduce copper impurity, its concentration should be minimal. In this work, the concentration of copper ions was 20 times lower than the concentration of the second metal ions.

The resulting NW were investigated by microscopy. In the first phase, scanning microscopy (SEM) was used. The

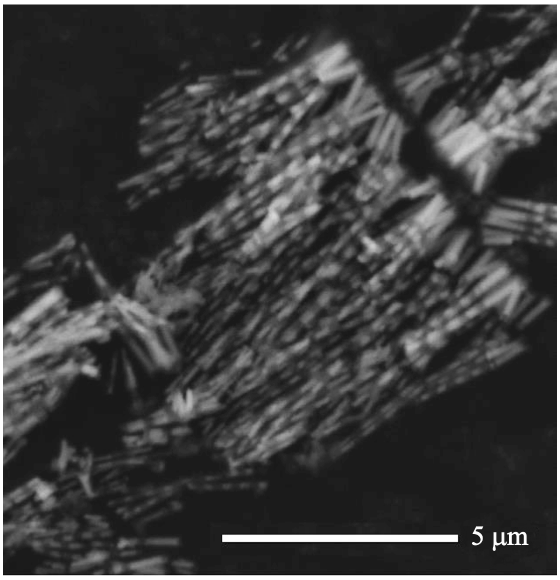

Figure 1. Co/Cu-NW SEM image.

example of the resulting image of a layered NW (Fig. 1) shows that this method can only be used for preliminary evaluation.

For detailed studies, a higher resolution of the TEM method is required, the application of which (on the example of Ni/Cu-NW) is described below.

3.2. Time control mode

Initially, the thickness reduction was achieved by the deposition time control mode — i.e., by reducing the growth time of the individual layer. An example of the obtained structures is given in Fig. 2.

The resulting images show that the NW have a layered structure. However, the thickness of the layers varies along the NW length. The elemental analysis conducted indicates the presence of copper impurity in magnetic layers of 20 order. In addition, the interface has an irregular shape: there is a bending and „flowing“ of one metal into the layer of another. Such results suggest the need to look for other growth regimes.

In order to eliminate the problem of changing the thickness of the layer in the NW the method of adjusting the duration of growth pulses based on the amount of the leaked charge (in accordance with the Faraday law) was used. The resulting images of NW arrays are shown in Fig. 3.

Results in Fig. 3, prove that the transition to the regime of control of the leaked charge has given significantly better results: the layers (both magnetic and non-magnetic) retain the same thickness along the entire NW.

The use of pauses between the growth of individual layers has been shown to be effective in increasing bulk samples. „Pauses“ — switching between the growth of different layers for a short time (a few seconds) — often resulted in leveling of concentrations and generally improved the structure of layers. In the case of NW, however, the desired result was not achieved. Although the nature of the interlayer boundaries changed (they became flat), the thickness of the layers themselves changed uncontrollably. This is assumed to be due to the dissolution of the deposited layers of magnetic materials during the „pauses“, and the deposition of copper layers (probably due to the resulting open circuit EMF).

The use of additives that would have led to more even growth and would have evened out the boundaries between the layers was another technique. Thus, the effect of butyndiol levelling additive has been studied. The TEM images of the samples showed that the interlayer boundaries became flat, but the copper layers became porous — perhaps due to the influence of butyndiol on the electrodeposition in narrow pores. Also used was the organic additive CDN-74 — the so-called glossy additive used to grind grain

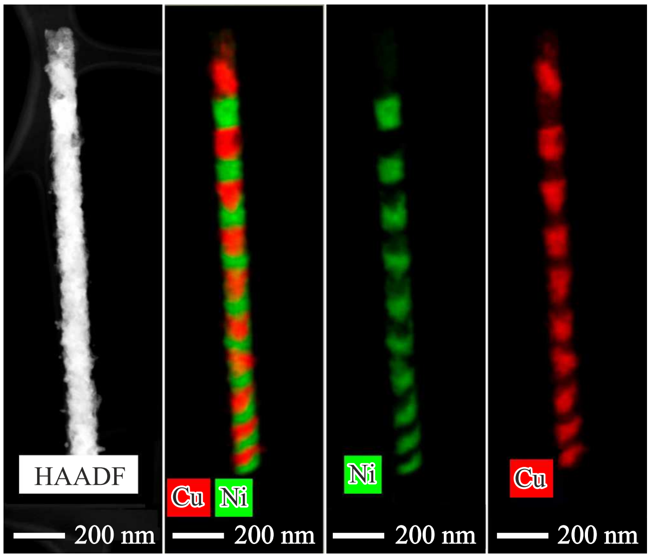

Figure 2. Image of layers in Ni/Cu-NW obtained in time control mode. (Left — PGEM image with z-contrast, near — element distribution cards.)

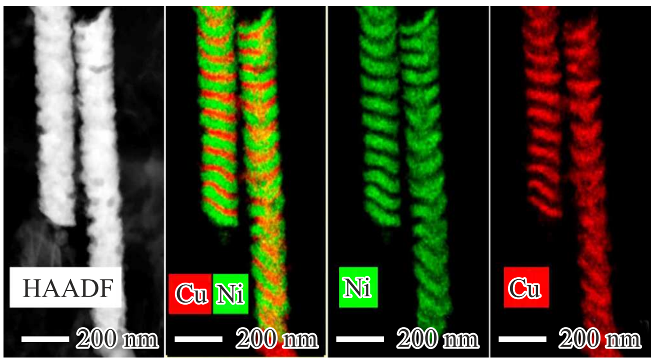

Figure 3. Image of layers in Ni/Cu-NW obtained in time control mode. (Left — PGEM image with z-contrast, then — element distribution cards.)

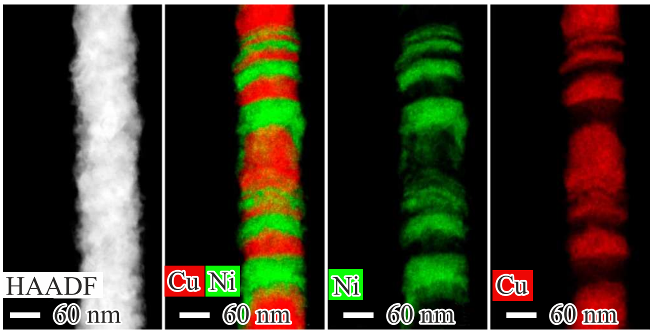

Figure 4. Image of layers in Ni/Cu-NW obtained using the third electrode. (Left — PGEM image with z-contrast, near — element distribution cards.)

and obtain a smooth surface. In this case, too, the interlayer interfaces were aligned. However, the thickness of the layers became significantly higher than the calculated — obviously, due to changes in electrodeposition conditions. It can be concluded that the application of this additive is possible only after adjusting the mode.

Electrolyte concentration change was another approach. It has been calculated that the production of thin layers (about 10 nm or less) at „conventional“ electrolyte concentrations requires a reduction in the deposition time of the layer to 1 s or less. However, due to the special features of diffusion in thin pore channels, such a short time — and therefore frequent change of modes — will lead to the uncontrolled change of diffusion conditions. Dilute electrolytes may be one solution: the deposition time increases and the process is more easily controlled. The results prove the possibility of reducing the thickness of the layers to 13–15 nm. However, the interfaces become uneven.

Use of the third electrode. It is known that at galvanic deposition the so-called third electrode — „comparison electrode“, is often used located at the surface of the growing metal and controlling the deposition process in the immediate vicinity of the working area. The TEM studies of NW samples (synthesized with a comparison electrode) showed the presence of layers with a successively decreasing thickness — the possibility of thinning the layers up to 7–10 nm was demonstrated. An example of NW with successively shrinking layers grown with dilute electrolyte is given in Fig. 4.

In general, the best results can be assumed to be thin layers with a good (flat) boundary between them obtained with a combination of several successful techniques: charge control, three-electrode circuit and electrolyte dilution.

3.3. Magnetic measurements

Magnetic properties were studied: for samples of Co/Cu layered NW with a diameter of 100 nm and with different thicknesses of cobalt and copper, hysteresis loops were removed. Two types of dependencies have been investigated: the influence of the length of the magnetic layers and the influence of the length of the nonmagnetic layers (spacers) on the shape of the hysteresis loops. The results obtained for the samples with different lengths of magnetic (cobalt) layers (and with the same lengths of non-magnetic copper spacers) are described in detail in work [12]. Thus, it has been shown that in the case of long Co layers (200 nm or more) the easy magnetic axis (EMA) is determined by the anisotropy of individual magnetic layers and lies along the axis of the NW. At the same time, for the layers of „medium“ length (equal to NW diameter) the NW array behaves like an isotropic material.

The second type of dependence is described in detail below. Samples with a copper layer length of 300 or 10 nm (cobalt layer thickness in both cases was about 50 nm). The results obtained are presented in Fig. 5.

The results show that in the case of a long EMA spacer, the direction is perpendicular to the axis of the NR — its direction is determined by the „flattened“ form of cobalt layers (diameter is two times the thickness). On the contrary, in the case of thin copper layers because of the interaction of the adjacent magnetic layers the NW as if „does not feel“ the non-magnetic layers — EMA lies along the axis of the NW. Both examples show the possibility of changing the magnetic anisotropy (in particular, the direction of EMA) by changing the ratio of thicknesses of magnetic and non-magnetic layers.

It may be noted that in the case of layered NW, the magnetic properties are determined both by the individual magnetic layers and the distance between them, and by the interaction between the adjacent NW. The latter relationship requires a separate study on samples with different pore densities (and, respectively, with different distances between NW).

3.4. NMR method

NMR (on^{59}Co kernels) was used to study layered Co/Cu-NW. Previously, a comparison was made between pure cobalt and cobalt NW with copper [18]. In addition to the cobalt main line of 220 MHz (corresponding to pure metal, where the cobalt atom is coordinated by twelve adjacent cobalt atoms) lines with frequencies of about 200, 180, 160 MHz etc. are shown to be detected when copper is added to NW in their NMR spectrum. Their emergence indicates a consistent substitution of copper atoms for cobalt atoms, with increasing concentrations. The role of interfaces in different NW has been assessed in this work. As an example in Fig. 6 the NMR spectra of NW with cobalt layers of 30 and 7 nm are given.

It can be seen that as the cobalt layer thickness decreases, the intensity of the lines increases at a lower frequency. This indicates an increase in the number of cobalt atoms with copper atoms in their coordination sphere. Estimation of the ratio of intensity of resonant lines of NMR spectrum allows to judge the distribution of types of configuration of the first coordination sphere: with decrease of layer thickness

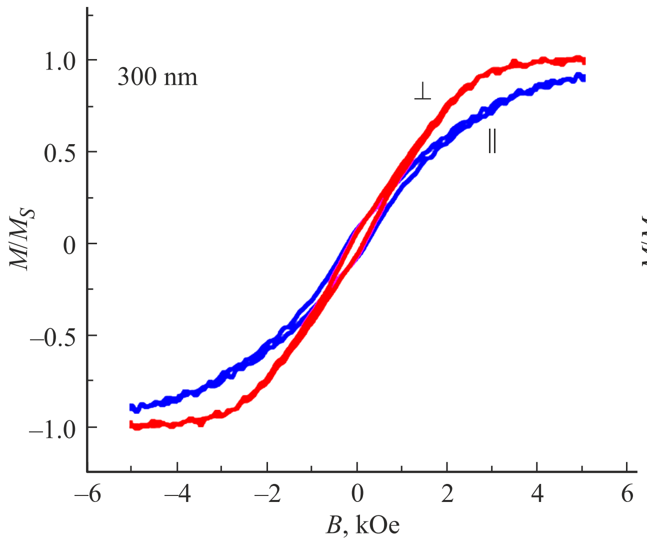

Figure 5. Hysteresis loops for samples with copper layers *a*) 300 nm and *b*) 10 nm (in both cases the cobalt layer thickness was 50–40 nm) (Fig. from work [12]).

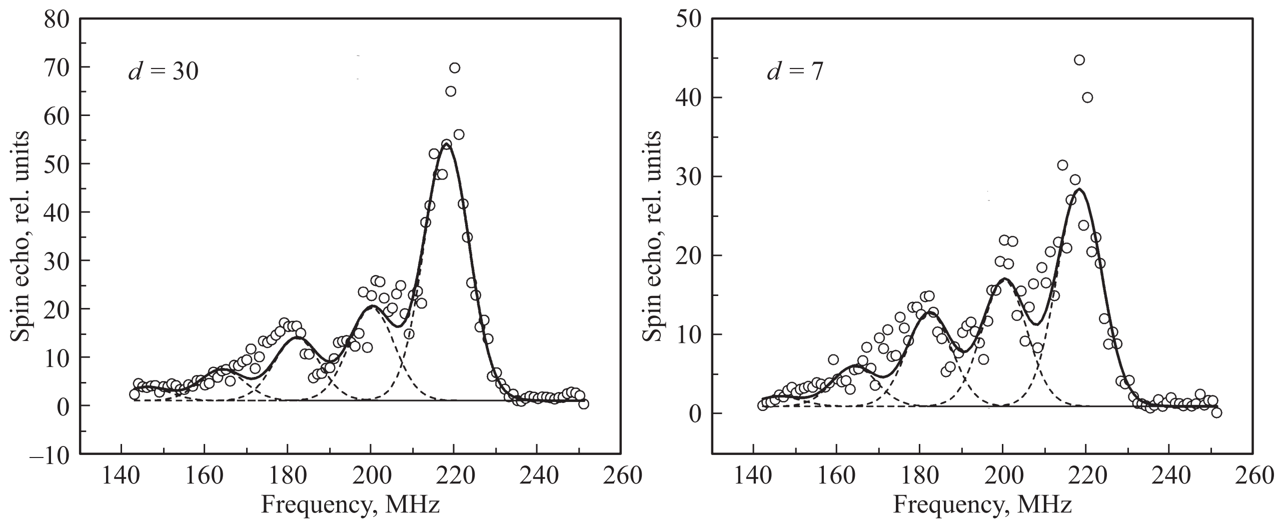

Figure 6. NMR spectra of layered Co/Cu-NW with the layer thickness of *a*) 30 nm and *b*) 7 nm.

the integral share of copper increases, at that the share of cobalt atoms with a large number of copper atoms in the surrounding environment is increasing.

The reasons for this may be an increase in the share of atoms in the border area, distortion of the shape of the boundaries themselves and an increase in the amount of copper in the cobalt layers themselves (in the latter case, it is possible to assume the formation of a solid solution with cobalt). The fractions of cobalt atoms coordinated by a different number of copper atoms (calculated by the formula from [18]) are 45% and 55% (for NW with layers thicknesses of 50 and 7 nm, respectively). Such a big value suggests that a significant amount of copper is also found in cobalt layers (in impurities). In this case, NMR gives integral information about Co atoms both in the „cobalt“ layer and at its boundary (in the interface). Note that the NMR method can evaluate the status of the Co/Cu interfaces: comparing the NMR data with the results of earlier TEM studies, it is possible to conclude that the interlayer boundaries are distorted while reducing the thickness of the layers. For more specific conclusions, further study of layered NW with different layer thicknesses is needed, as well as the application of X-ray diffraction analysis and/or high-resolution TEM.

The NMR method has also been used to study pure iron NW. The NMR spectrum at the ^{57}Fe kernels in a zero external magnetic field (ZF-NMR) at 4.2 K for homogeneous one-component (iron-only) NW with a diameter of 100 nm was measured. The resulting spectrum is represented in Fig. 7 together with the ^{57}Fe spectrum of bulk alpha-iron powder, also measured at $T = 4.2$ K.

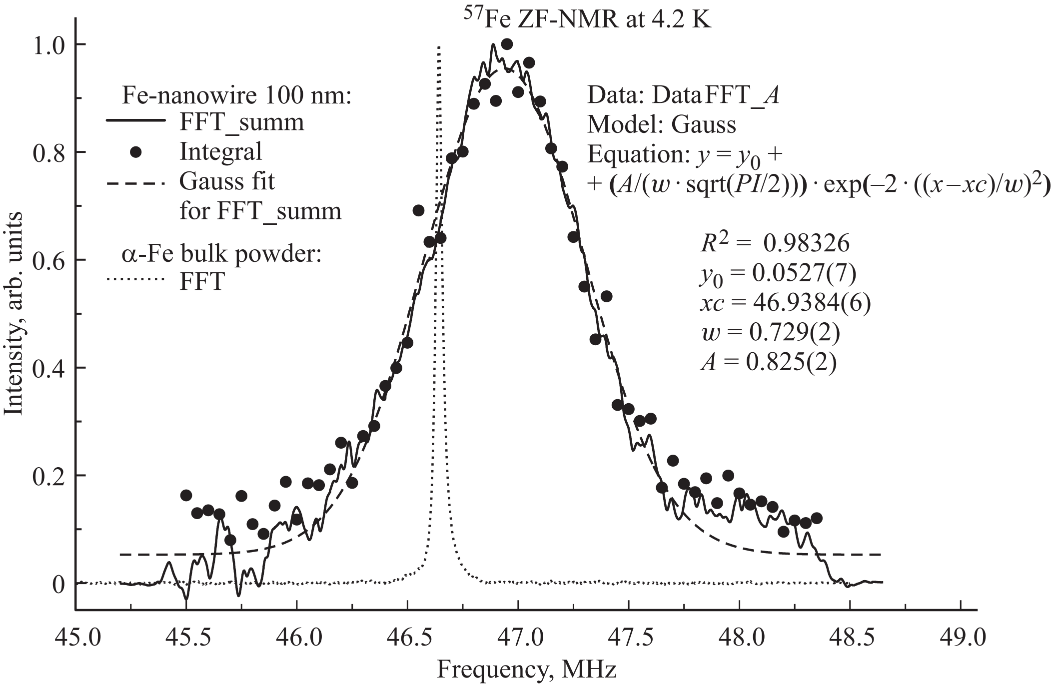

Figure 7. NMR spectra of ^{57}Fe in the zero external magnetic field (ZF-NMR) Fe-NW with a diameter of 100 nm measured at $T = 4.2$ K by integrating the integral spin echo (black circles) and the sum of the second Integral Echo of the Integral Echo signal (FFT_summ, solid line) when changing frequency step by step. The dotted line represents the best approximation of the FFT_summ curve by the Gaussian function. Also, the ZF-NMR spectrum of bulk powder α-Fe is given, measured at $T = 4.2$ K by the Fourier method of half spin echo signal at the frequency of maximum of ZF-NMR 46.642 MHz line (dots).

As you can see from Fig. 7, the ZF-NMR spectrum of NW demonstrates a significantly augmented line that is well approximated by the Gaussian function, reflecting the statistical nature of the distribution of local magnetic fields in these NW. The width of the Gaussian curve is 0.729(2) MHz, which is 20 times the width of the NMR line of bulk alpha-iron, which is 0.036(1) MHz at half the height. Since ^{57}Fe kernel with a spin of $I = 1/2$ does not experience the splitting of energy states under the action of an inhomogeneous electric field, such a large expansion of the NMR line indicates a very large spread of only local magnetic field values on iron in the amount of NW. It should also be noted that the center of the Gaussian distribution of the NMR line in the NW is offset by the value of $\Delta F = 0.297$ MHz towards higher frequencies. This corresponds to an increase in the average value of the internal magnetic field, the profile of which is directly determined by ZF-NMR spectrum, by a value of $\Delta B = \Delta F/\gamma(^{57}\mathrm{Fe}) = 0.216$ T. The nature of this increase, as well as the effect of the NW diameter on the width of the NMR line in it is to be determined in further research. In addition, there was a significant acceleration of spin-lattice relaxation in these NW ($T_1 = 1.02(6\,\mathrm{ms})$ compared to α-Fe ($T_1 = 13.9(6)$ ms), which also indicates an increased degree of disorder.

4. Conclusion

The article studies peculiarities of growth of layered two-component nanowires (NW). The article studies different approaches to obtaining layers with the same (on the length of NW) thickness, to the reduction of thickness of layers and to obtaining interfaces of regular (flat) shape. The application of the TEM method led to the conclusion that a number of techniques used for the growth of bulk samples proved to be ineffective in the growth of NW — the use of levelling additives, pauses between growth impulses. At the same time, some approaches have proven to be very effective in producing layer-based NW with thin layers and smooth interfaces. Among these approaches are the control of the amount of charge flowing, the use of a comparison electrode and electrolyte dilution, and combinations of these techniques. The use of these techniques resulted in NW with 7–10 nm layers and with flat interfaces.

The magnetometry method studies hysteresis loops: measurements are made in two geometries: along the wire axis and in a perpendicular direction. It is shown that the magnetic properties of the array of layer-based NW (in particular, the direction of the light magnetization axis in the Co/Cu NW array) depend both on the aspect ratio in the magnetic layers and on the ratio of the magnetic metal layer thickness to the non-magnetic spacer thickness.

The NMR method (at ^{59}Co kernels) revealed an increase in the proportion of Co kernels coordinated by copper atoms in NW with thinner layers. This is obviously due to the great contribution of interfaces. In general, a significant proportion of Co kernels surrounded by copper suggest that „cobalt“ layers contain a significant amount of copper as an impurity. Iron NW (NMR ^{57}Fe) were first studied: a comparison with bulk iron samples showed a shift in the line towards high frequencies, indicating an increase of approximately 0.2 T. There is also a significant (20-fold) line expansion and increase of more than an order of speed of spin-lattice relaxation. The latter proves the large variation of local magnetic field values.

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Conflict of interest

The authors declare that they have no conflict of interest.

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