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DEVELOPMENT OF MÖSSBAUER SPECTROSCOPY FOR MAGNETIC
NANOMATERIALS AND DYNAMICS OF MACROMOLECULES

Summary of doctoral dissertation

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VILNIAUS UNIVERSITETAS

JONAS REKLAITIS

MESBAUERIO SPEKTROSKOPIJOS TAIKYMAS MAGNETINIŲ
NANOMEDŽIAGŲ IR MAKROMOLEKULIŲ DINAMIKOS TYRIMAMS

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Introduction

The attention to the investigation of dynamics of atoms, and especially the collective dynamics of biomolecules and proteins, has increased recently because of the importance of their biological functions [1]. Informative methods e.g. X-ray dynamical analysis, nuclear magnetic resonance and neutron scattering are used for these investigations.

Unfortunately, the traditional Mössbauer spectroscopy has one essential drawback — the material under investigation should contain (as the main component or as impurity) identical Mössbauer nuclei as in the source. Thus, the application of traditional Mössbauer spectroscopy is unyielding to the investigation of the dynamics of biomolecules and proteins. Therefore, a new method based on the same Mössbauer effect but not requiring Mössbauer isotopes to be in the samples, the Rayleigh scattering of Mössbauer radiation (RSMR), has been developed and used. The Mössbauer γ -radiation is scattered by the electron cloud of atoms of the investigated materials. The part of elastic scattered γ -quanta (f_R) or the spectrum shape is analyzed by the absorber containing Mössbauer nuclei. The RSMR method is capable of detecting variations in the energy of the diffracted photons comparable with the typical width of the Mössbauer line ($\sim 10^{-9}$ eV for ^{57}Fe). This high energy resolution allows the experimental separation of elastic and inelastic scattering.

The newly created information rate grows exponentially, with its amount being doubled every three years [2], therefore the storage of information is relevant issue these days. It is theoretically assessed that the density of conventional longitudinal magnetic recording storage device is only 7.75 GB/cm². However the density of patterned perpendicular media may potentially have a much higher storage density than 15.5 GB/cm². One of the most promising candidates for recording medium of perpendicular magnetic recording could be a highly ordered array of magnetic nanowires in porous anodic aluminum oxide (AAO) template. One bit of information corresponds to one single domain nanowire, which can be magnetized "up" and is equivalent to a logical 1, or "down" — 0. In this way the recording density is determined by the

density of nanowires, thus the information storage density may be controlled with the control of nanowire density.

The increasing speed of technological progress and the constant miniaturization of electronics create a growing demand for smaller elements. The main problem encountered is that the properties of a small elements — nanostructures — have properties different from those of a "bulk" material.

Manufacturing of nanomaterials is a relevant topic, and its methods are of various complexity: mechanical grinding, chemical precipitation, electrical discharge, nanolithography etc. However, not all of these methods have an easily controlled dispersion of properties of nanomaterials.

The main tasks:

Classical Mössbauer spectroscopy has been applied to studies of iron nanowires and thus the following tasks were set:

1. preparation of different anodic alumina oxide matrices filled with iron nanowires;
2. investigation of the effectiveness of different electrolytes used in nanowire deposition;
3. exploration of the aging effects on chemical composition of nanowires;
4. investigation of the influence of annealing parameters on the chemical composition.

By expanding the possibilities of Mössbauer spectroscopy the following tasks were set:

1. to design the proper upgrades for the classical Mössbauer spectrometer, so that it could be used to detect scattered radiation;
2. to show the possibilities of the method by analyzing thermal movement of the carbon atoms in a polystyrene sample.

Statements of defence

1. The Mössbauer radiation Rayleigh scattering method extends the use of Mössbauer spectroscopy in the research of the dynamic characteristics of organic and polymer atoms, and the use of a high-resolution semiconductor detector increases the sensitivity of the analysis.
2. A new type of water based electrolyte is created for uniform growth of iron nanowire colonies inside anodic aluminum oxide templates;
3. Iron nanowires (with a diameter of 8 nm – 30 nm and a length of up to 2500 nm), fully encapsulated inside anodic aluminum templates, in the process of annealing (at temperatures between 250 °C – 600 °C) reacts with anodic aluminum oxide.

Novelty of the work

A new type of the Rayleigh scattering Mössbauer radiation spectrometer is created, which allows the analysis of materials not containing Mössbauer nucleus.

It has been determined that iron nanowires do not have a core-shell structure inside the anodic aluminum oxide template.

The aging of iron nanowires was analyzed with a complex use of the Mössbauer spectroscopy, the conversion electron Mössbauer spectroscopy and X-ray diffraction.

The Mössbauer spectroscopy and X-ray diffraction were applied to determine a chemical composition of the annealed nanowires, encapsulated inside anodic aluminum oxide templates.

Experimental

Mössbauer spectroscopy

The Mössbauer spectra were measured at room temperature using the source of ^{57}Co in Rh matrix. The spectrometer (Wissenschaftliche Elektronik GmbH) was operating in a constant acceleration mode. The velocity scale was calibrated using the magnetic sextet of a high-purity iron foil absorber as a standard. The line width obtained for the α -Fe 25 μm thick foil, as a reference, was approximately $\Gamma = 0.28$ mm/s at ambient temperature. All experimental spectra were fitted using Lorentzian lines by the least-squares method.

The amount of ferromagnetic α -Fe and two species of paramagnetic iron embedded in the template were evaluated according to the area of the sextet and of two doublets.

Sample preparation

All the chemicals were chemical grade reagents and were used without further purification in these experiments. Deionized water obtained from a Milli-Q Millipore water system was used in preparations of all solutions and washings.

Porous alumina templates of the thickness from 4.5 μm to 13 μm with the average pore diameter of 30, 15, 13, and 8 nm were prepared via two-step anodization processes in a vigorous stirred and thermostated solution of sulfuric acid.

Anodized specimens were carefully rinsed with deionized water and assembled into one of two solutions for deposition of iron nanowires within the alumina nanochannels. The solutions used were a well-known solution composed of FeSO_4 , H_3BO_3 and ascorbic acid and a new solution composed of FeSO_4 , citric acid and MgSO_4 (named A and B respectively). Iron sulfate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, was applied as a Fe source, magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, was used to prevent the breakdown of the alumina barrier layer during ac electrolysis, and a citric acid was added as a complexing agent for Fe^{2+} ions. In order to obtain spectra of better quality solution "B" was enriched with ^{57}Fe up to 10% instead of 2.119% natural abundance.

After deposition, the samples were washed for several minutes and placed in boiling deionized water for 30 min to seal the pores encapsulating the deposited Fe⁰ nanowires. Afterwards the samples were annealed for 1.5 h at the temperature of up to 600 °C. For the samples with nanowires of the 15 nm diameter, additional samples were annealed at 450 °C for different durations up to 140 min.

The thermal annealing was performed in an open horizontal tubular furnace. The samples were inserted into the furnace and kept at the annealing temperature to ensure a rapid heating and cooling of the samples.

Sample analysis

The alumina templates intended for XRD and CEMS observations were separated from the electrode surface by one-side sequential etching of the electrode window in a solution of 1.5 mol/l NaOH and then in 0.1 mol/l CuCl₂ + 0.27 mol/l HCl. To disengage the deposited/annealed products, the barrier layer and a part of the alumina matrix were dissolved by short-term etching in 0.25 mol/l NaOH followed by a thorough rinsing, neutralization in 0.1 mol/l citric acid solution, rinsing again and drying in the desiccators.

Powder X-ray diffraction (XRD) patterns of the samples after synthesis and after annealing were collected with a D8 diffractometer equipped with a Göbel mirror for Cu K α radiation. The patterns were collected from 20° to 70° 2 θ with a step size of 0.02° and dwell time of 8 s per step.

SEM, models EVO 50 EVP and Philips 30L, were used for observations of electrochemically filled alumina templates and disengaged nanowire arrays.

Results and discussion

RSMR

Radiation from ^{57}Co Mössbauer source in rhodium matrix, was collimated by lead shielding. Collimated beam (beam divergence 2 deg) was scattered by polystyrene sample. Spectrometer was fitted with two different detectors — SI-PIN semiconductor detector, manufactured by "AmpTek", and the proportional gas counter, manufactured by "Wissenschaftliche Elektronik GmbH". There were two absorbers tried to analyze the Rayleigh scattered radiation — one was $\text{K}_2\text{Mg}[\text{Fe}(\text{CN})_6]$ enriched with ^{57}Fe (96 atomic %), another one was $(\text{NH}_4)_3(\text{FeF}_6) \cdot \text{Li}_3(\text{FeF}_6)$ (also known as "black" absorber). Analyzer was placed in one of two positions (Fig. 1): between source and scatterer (A_1) or between scatterer and detector (A_2).

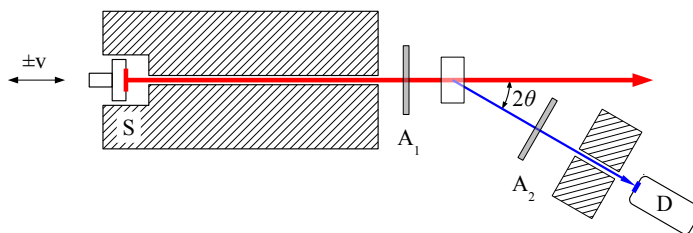


Figure 1 A sketch of RSMR spectrometer.

S — source, D — detector, A_1 and A_2 — position of analyzer, 2θ — scattering angle, $\pm v$ — drive velocity.

One can clearly see (Fig. 2), that energy spectra of ^{57}Co source, recorded with different detectors, differ in quality. One can easily recognize the iron K line, the Mössbauer line, and Rh fluorescence in spectra, recorded with the proportional gas counter (Fig. 2a). The same spectra recorded with the semiconductor detector give a much better energy resolution (Fig. 2b). It was possible to determine the structure of X-ray energy spectra: iron K series (K_α — 6.40 keV and K_β — 7.04 keV) and lead L series (L_α — 10.5 keV and L_β — 12.6 keV).

The Mössbauer linewidth for both spectra was evaluated. For the proportional gas counter the full width at half maximum (FWHM) is 1.3 keV (Fig. 2a), and for

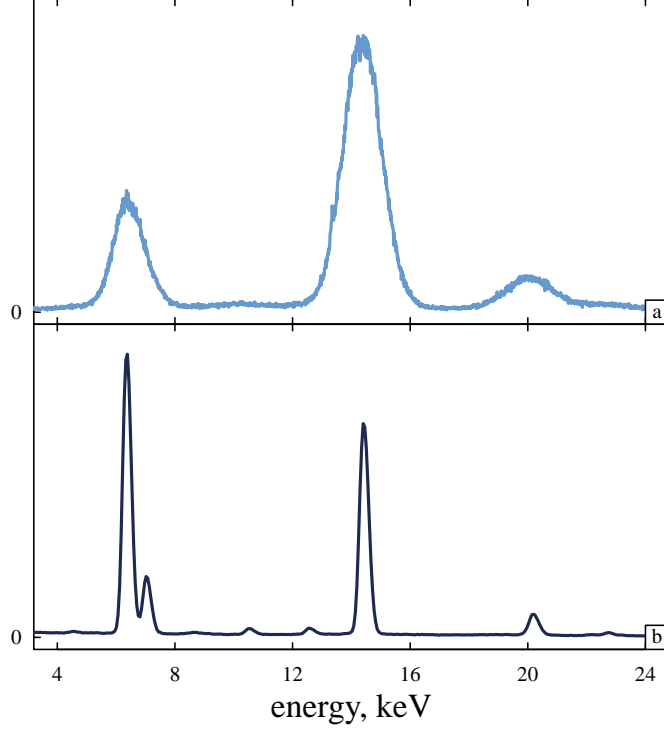


Figure 2 Energy spectra of $^{57}\text{Co}(\text{Rh})$ source.

a — proportional gas counter, *b* — semiconductor detector

the semiconductor detector FWHM is 0.3 keV (Fig. 2b).

Using "black" absorber several Mössbauer spectra were recorded using different detectors and scattering angle 2θ . For simplicity a spectrum of the "black" absorber was fitted to sextet (hyperfine magnetic interaction with low field $B_{ef} = 4.63 \pm 0.03$ T, isomer shift $\delta = 0.431 \pm 0.005$ mm/s, line width $\Gamma = 1.06 \pm 0.02$ mm/s, quadrupole splitting $\Delta = 0.0$ mm/s). These parameters were fixed and used for fitting the other RSMR spectra.

It took almost two weeks to obtain the spectrum of scattered radiation using proportional gas counter (Fig. 3a). The observed effect was very small ($f \sim 1\%$) and errors/uncertainty were very high due to low intensity of the registered spectrum (5900 counts per channel). After swapping a regular detector to a semiconductor one, quality leap was obtained: the measurement time decreased to a few days and the observed effect increased to 40% (Fig. 3b).

A semiconductor detector was chosen for further experiments, seeing that semi-

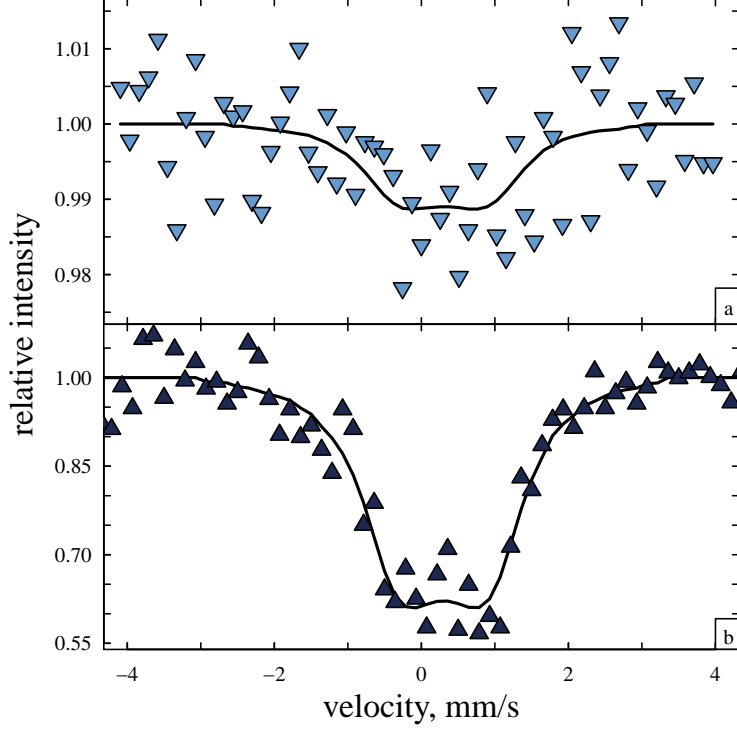


Figure 3 RSMR spectra of "black" absorber $2\theta = 6.0^\circ$.

a — proportional gas counter, *b* — semiconductor detector

conductor detector is significantly effective in scattering experiments. $\text{K}_2\text{Mg}[\text{Fe}(\text{CN})_6]$ was chosen as an analyzer for the scattered radiation because it has a very narrow line ($\Gamma = 0.31 \pm 0.02$) so one could observe the line broadening related to the dynamics of the sample. During experiment it became clear that spectra statistics is insufficient to properly evaluate line broadening. Mössbauer spectra (Fig. 4) of this absorber are fitted with the following parameters: $\delta = -0.095 \pm 0.001$ mm/s, $\Gamma = 0.313 \pm 0.004$ mm/s. These parameters were used to fit the other spectra with poorer statistics and evaluate the area of the spectra.

It is known that the peak area is proportional to the intensity of the elastic scattering, thus the ratio of the peak areas is equal to the probability of the elastic Rayleigh scattering f_R [3]. Dependence of f_R estimate on the scattering angle is shown in Fig. 5.

The same sample of polystyrene was analysed for the comparison using X-ray diffraction (XRD) ([3]). It has two maxima, at 19.96° and at 23.80° , which corre-

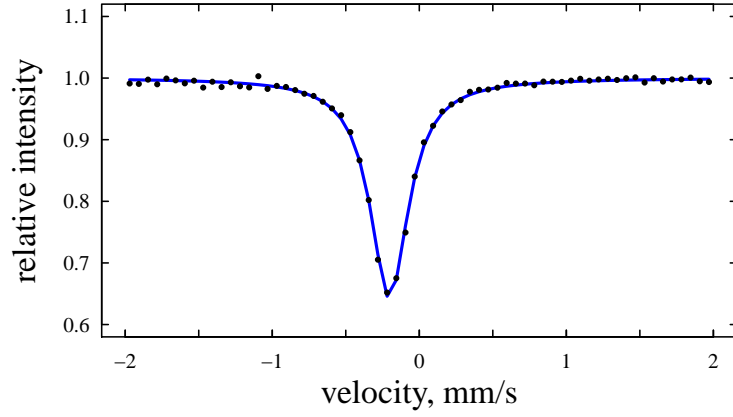


Figure 4 Typical Mössbauer spectra of $K_2Mg[Fe(CN)_6]$ (unscattered radiation)

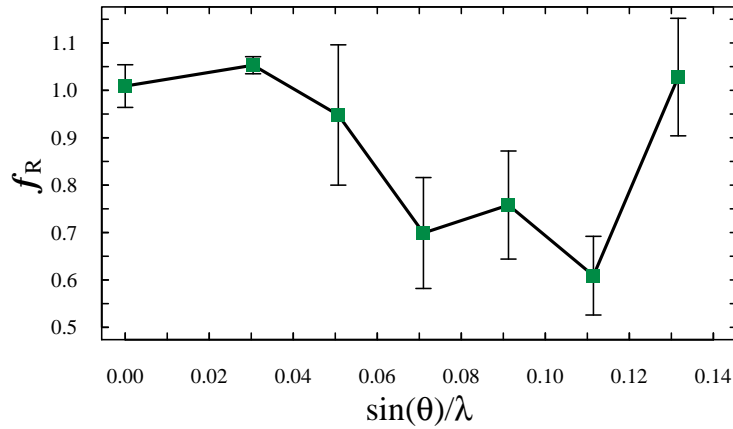


Figure 5 Dependence of RSMR estimate f_R on the scattering vector

spond to the distance between planes, 4.45 Å and 2.07 Å, respectively. In order to compare different types of measurements, the intensity of XRD was averaged over the angular step (Fig. 6a), which was used in the RSMR measurements (Fig. 6b).

Figure 7 shows the angular dependencies of the elastic and inelastic intensities. The Mössbauer radiation intensity was multiplied by the numeric value of scattering probability in order to obtain the elastic and inelastic scattering intensities.

Section conclusions:

- Our study has shown that applying the semiconductor Si-PIN detector (Amptek Inc.) for recording the RSMR spectra is much more suitable than other types of detection;

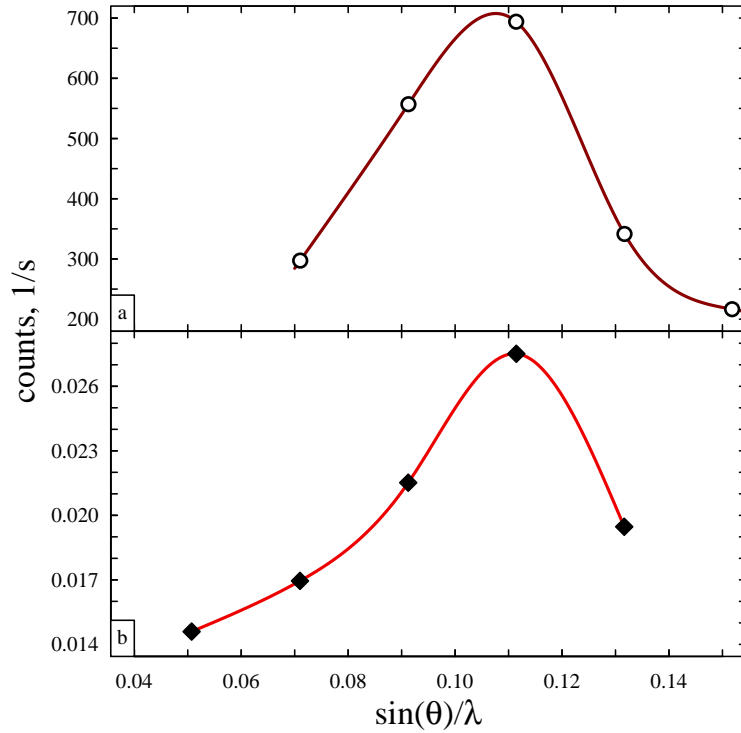


Figure 6 Dependence of averaged X-ray (a) and Mössbauer (b) radiation intensities on the scattering vector $\sin(\theta)/\lambda$

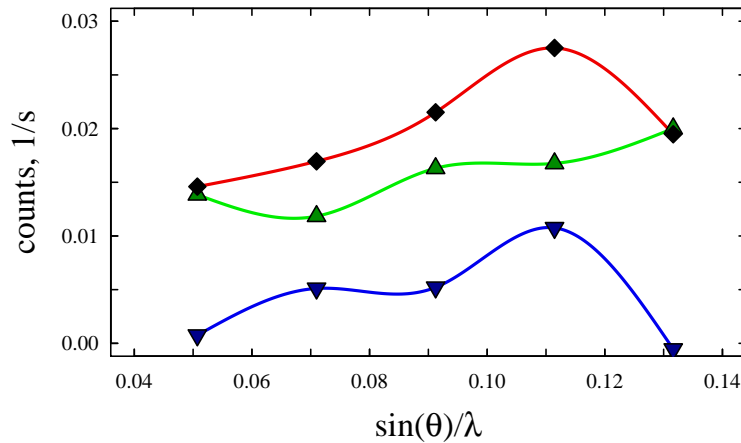


Figure 7 Angular dependencies of total (\blacklozenge), elastic (\blacktriangle) and inelastic (\blacktriangledown) intensities on scattering vector $\sin(\theta)/\lambda$

- Switching the proportional gas counter to the semiconductor detector, gives a great increase in performance: measured Mössbauer line (FWHM) in energy spectra has reduced from 1.3 keV to 0.3 keV; the observed effect has increased to 40%

- Using the RSMR method the scattering of the polystyrene sample was studied in the neighborhood of the main Bragg maximum, which corresponds to the distance between the polymer chains equal to $d = 4.44 \text{ \AA}$;
- A new kind of RSMR spectrometer was created, which could be used in analysis of dynamic processes, due to the suitable time scale of the scattering process.

Aging of iron nanowires in AAO template

Porous anodic alumina oxide (AAO) templates were prepared by method proposed by Masuda [4]. The pore diameter of AAO was evaluated to be 15 nm from anodizing conditions, and later confirmed by transmission electron microscopy (TEM). Prepared templates were filled with iron nanowires (*nws*) by the alternating current electrolysis protocol. Two solutions were used for electrolysis: one composed of FeSO_4 , H_3BO_3 and ascorbic acid [5,6], another one, created specially for this study, was composed of FeSO_4 , MgSO_4 and citric acid [7]. Templates were colored uniformly from light bronze to deep black tints.

X-ray diffraction (Fig. 8) measurements show only two diffraction peaks — an especially strong peak occurring at $2\theta=44,67^\circ$ and a weak peak at $2\theta=65^\circ$. In agreement with the data of the polycrystalline bulk Fe diffraction line standard (PDF: No 006-0696), large peak indicates the formation of the metallic α -Fe nanowire array with a preferred growth direction $[1\ 1\ 0]$ along the crystallographic axes.

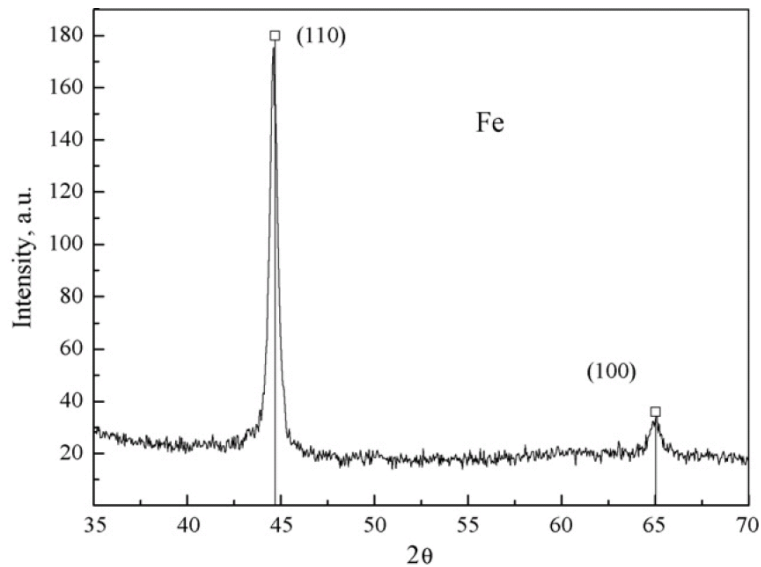


Figure 8 XRD pattern of the sulfuric acid alumina template colored black in the solution containing $0.05\text{M FeSO}_4 + 0.025\text{M MgSO}_4 + 0.05\text{M citric acid}$ by *ac* electrolysis for 45 min.

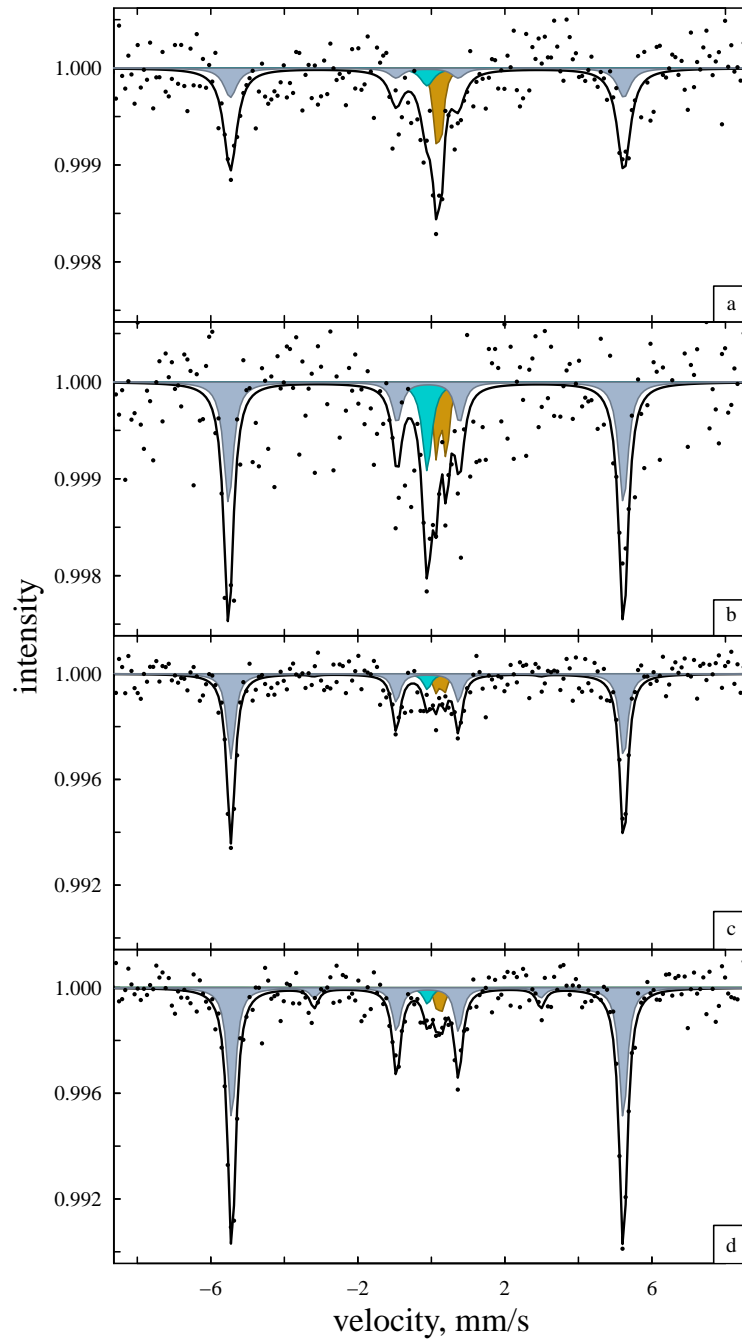


Figure 9 Typical Mössbauer spectrum of the Fe nws array within AAO template

AAO pore diameter 15 nm a — duration of electrolysis 5 min., b — 15 min., c — 25 min. and d — 40 min.

The Mössbauer spectra (Fig. 9) indicate that after deposition samples contain mainly one iron phase — α -Fe. The paramagnetic iron components can be seen in spectra also. Singlet isomer shift coincides with isomer shift of α -Fe and is

most probably caused by iron nanoprecipitates. Doublet spectra with Mössbauer parameters: the isomer shift¹ $\delta = 0.338 \pm 0.035$ mm/s and the quadrupole splitting $\Delta = 0.203 \pm 0.072$ mm/s assignable to Fe(III) compounds.

The ratio of intensities of lines in the Mössbauer sextet is given as:

$$3 : \frac{4\sin^2\theta}{1 + \cos^2\theta} : 1 : 1 : \frac{4\sin^2\theta}{1 + \cos^2\theta} : 3 \quad (1)$$

where θ is the angle between the direction of γ -rays and magnetic moments in an iron atom. We have found that for all cases the relative intensities of the second and fifth lines in Mössbauer spectra of as-grown iron nanowires are close to zero, implying that the magnetic moments of the nanowires are along their growth direction.

Area of the subspectrum is proportional to the amount of iron in respective compounds. Based on this the concentration of each species of iron was evaluated. In addition the average length of iron nanowires was determined by evaluating the average thickness of α -Fe in the sample and recalculating it according to the relative area occupied by the pores (Fig. 10).

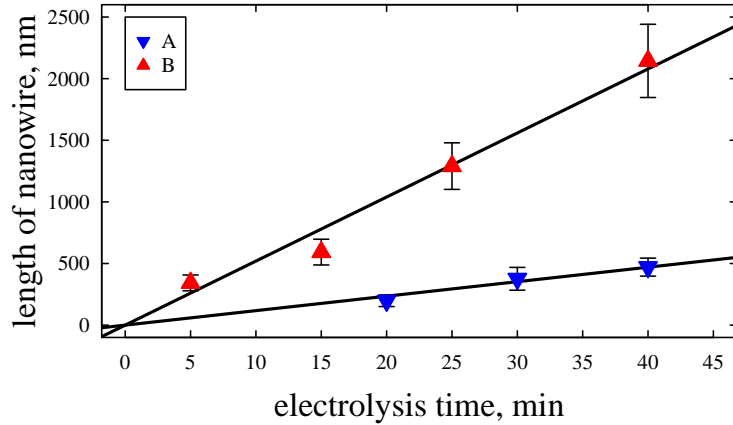


Figure 10 Time-variations in the average height of iron nanowires deposited within the alumina pores

A — FeSO_4 , H_3BO_3 and ascorbic acid solution, B — FeSO_4 , MgSO_4 and citric acid solution

The amount of paramagnetic Fe(III) is the same in all samples and it increases in time (Fig. 11). This behavior is clearly a sign of the corrosion of the upper part

¹relative to α -Fe

of nanowires, i.e. the areas exposed to air. The TEM studies showed on observable differences in the shapes of iron nanowires liberated from the template before and after their storage inside the matrix for 5 months. However TEM images produced 5 months after the storage of the free iron nanowires in the air revealed the strong corrosion in only some places of the nanowired surface (Fig. 12b). The corroded regions, seen as sphere balls, were randomly distributed along all the nanowire length and some of them looked like catkin-twigs. Such kind of corrosion can be explained by the localization of positive and negative charges in separate places of nanowire [8–10].

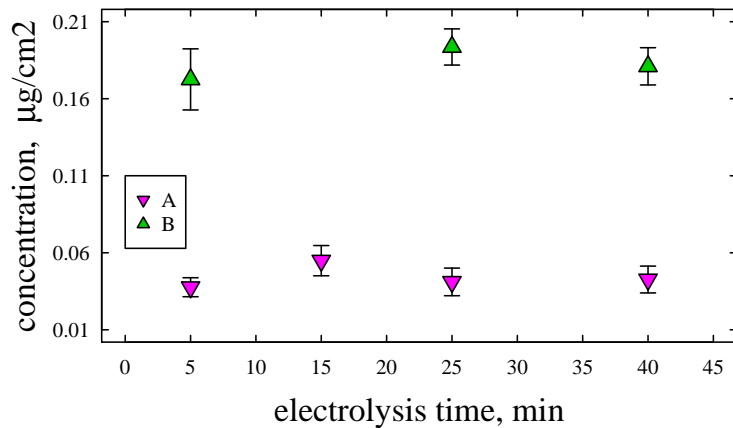


Figure 11 Variations in paramagnetic iron contents deposited within the alumina template

A — fresh samples, B — storage under ambient conditions for 5 months

The alumina matrix was encased with iron nanowires and stored under ambient conditions for several days. Afterwards the conversion electron Mössbauer spectra (CEMS) of both side of alumina matrix were recorded. As can be seen in Fig. 13, these spectra are quite different. A back-side alumina Mössbauer spectrum (Fig. 13a) can be simply fitted using the sextet assignable to ferromagnetic iron α -Fe. Nonetheless, the topside Mössbauer spectrum (Fig. 13b) demonstrates an additional typical doublet with Mössbauer parameters: the isomer shift $\delta = 0.361 \pm 0.002$ mm/s and the quadrupole splitting $\Delta = 0.74 \pm 0.01$ mm/s assignable to Fe(III). The doublet could arise due to the formation of $\text{Fe}(\text{OH})_3$ or $\text{Fe}(\text{OH})_3 \cdot n\text{H}_2\text{O}$ hydroxides.

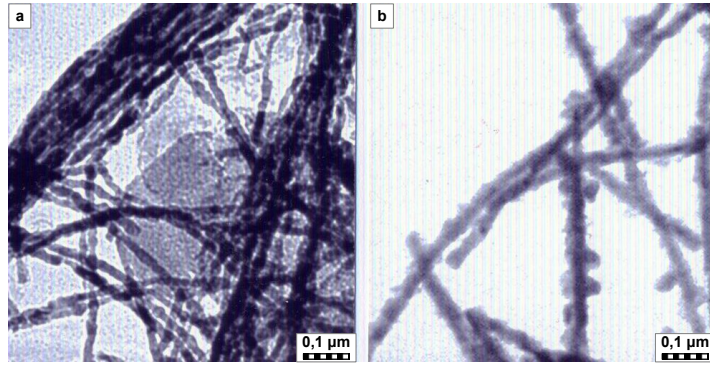


Figure 12 TEM image of iron nws liberated from the template.

a — freshly liberated nanowires, *b* — same nanowires after their storage in the air for 5 months

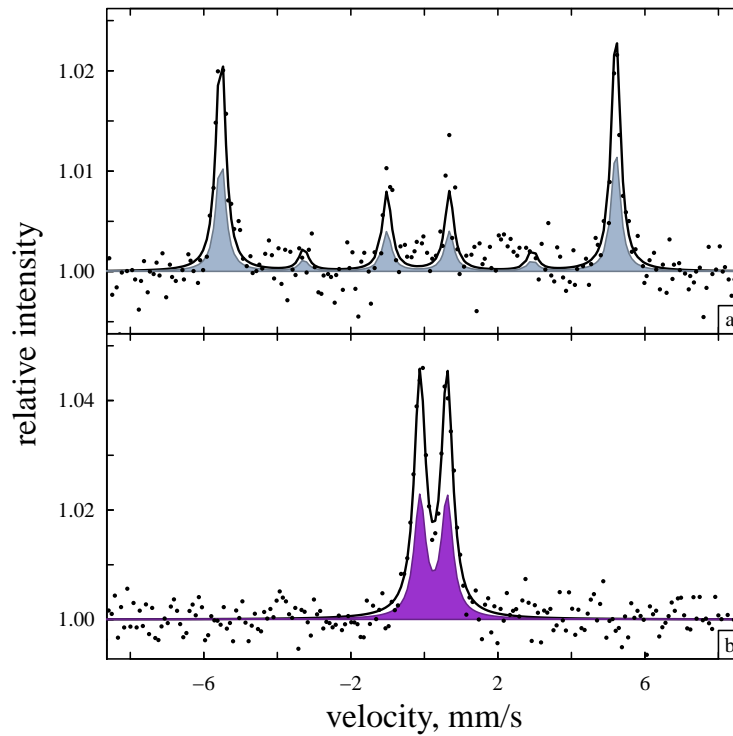


Figure 13 The Mössbauer conversion electron spectra recorded for back-side (a) and top-side (b) non-sealed alumina film filled with metallic iron nanowires

Section conclusions:

- it is shown that at low *ac* current densities, metallic iron α -Fe nanowires in AAO templates are deposited with the preferred $[1\ 1\ 0]$ growth direction;
- it is shown that iron nanowires in solution B grow faster than in solution A —

52 ± 2 nm/min. and $11,8 \pm 0,6$ nm/min. respectively;

- CEMS studies have revealed that only the upper part of nanowires in the AAO template have been affected by corrosion.

Annealing effects on the transformations of Fe nanowires encapsulated in the alumina template pores

As before, porous alumina templates with the average pore diameter of 8, 13, 15 and 30 nm were prepared via two-step anodization process [4]. A solution composed of FeSO_4 , MgSO_4 and citric acid was used in iron electrodeposition to grow iron nanowires inside AAO pores. After deposition, all the samples were placed in boiling deionized water for 30 min. to seal the pores encapsulating the deposited iron nanowires and to prevent unwanted/spontaneous oxidation of nanowires. The oxidation and reaction of iron nanowires with the surrounding Al_2O_3 matrix were studied by heating the samples in the air and keeping them for 1.5 h at the temperature of up to 600 °C.

The Mössbauer spectra (Fig. 14) indicate that after deposition samples contain mainly one iron phase — α -Fe. The direction of magnetic moment is mostly perpendicular to the sample plane; therefore the 2nd and 5th sextet lines disappear. Also there is no considerable variation in the hyperfine parameters for different diameter nanowires in the 8–30nm range. In samples with the pore diameter of 13 nm and 15 nm there is a small fraction of Fe(III) compound. The Mössbauer spectrum of the as-prepared sample with the average pore diameter of 8 nm was found to be more complex and composed of three components similar to those following annealing of specimens (Fig. 14d). The quantities of paramagnetic Fe(III) and Fe(II) components were found to be larger, together making up more than 40% of the total amount of deposited iron. It may be assumed that it indicates initial corrosion of the surface of iron nanowires during sealing in boiling water.

Annealing for 1.5 hour in the air caused partial or full oxidation of α -Fe phase, depending on annealing temperature and the average pore diameter. With the larger diameter of nanowires the temperature, above which no α -Fe was found, identified to be higher and it was around 600 °C for iron nanowires of the 30 nm diameter (Fig. 15).

Two new species of iron — Fe(II) and Fe(III) — are observed in Mössbauer spectra

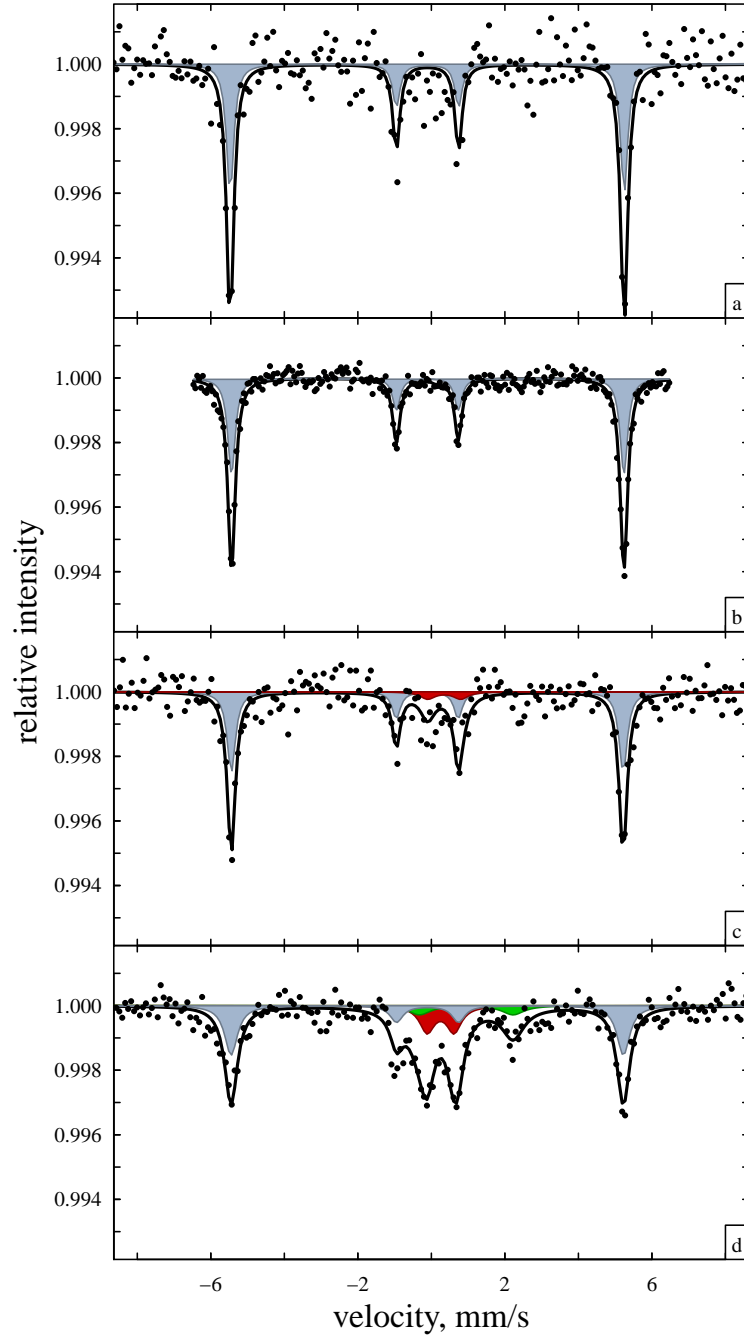


Figure 14 Mössbauer spectra of iron nanowires in AAO templates after deposition for the nanowires
average diameter of pores: a — 30 nm, b — 15 nm, c — 13 nm, d — 8 nm

after annealing. According to the parameters of the description of Mössbauer spectra ($\delta=0.9 - 1.0\pm 0.2$ mm/s $\Delta=1.9 - 2.1\pm 0.2$ mm/s), Fe(II) doublet is caused by the formation of hercynite FeAl_2O_4 . Fe(III) doublet ($\delta=0.3 - 0.4\pm 0.1$ mm/s $\Delta=0.9 - 1.0\pm 0.2$ mm/s) is found due to the dissolution of Fe atoms in aluminium oxide

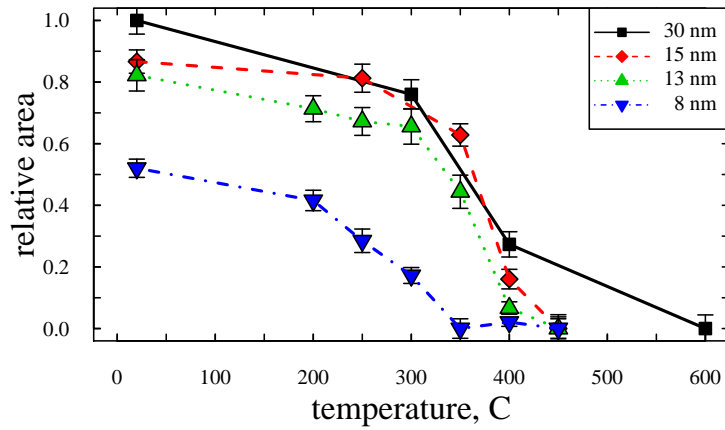


Figure 15 *Dependence of contents of α -Fe iron on annealing temperature*

matrix producing $(\text{Fe}_x\text{Al}_{1-x})_2\text{O}_3$ ($x \leq 0.15$). However, the X-ray diffraction spectra did not indicate formation of these compounds in annealed samples of Fe nanowires in AAO [11]. The absence of peaks could be caused either by extremely small grains or the amorphous nature of compounds.

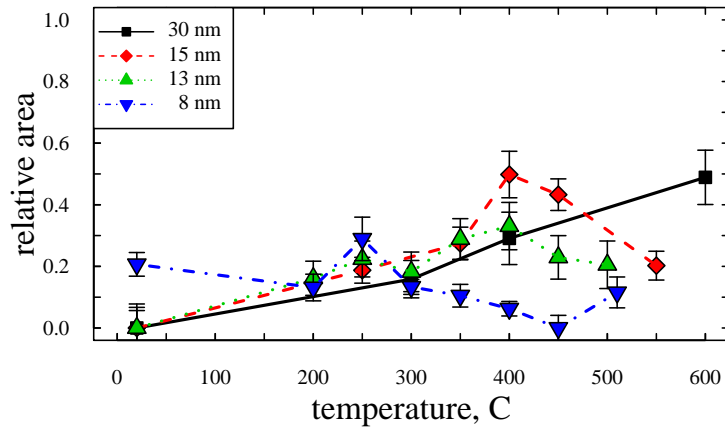


Figure 16 *Dependence of contents of FeAl_2O_4 in AAO template on annealing temperature*

The increase in annealing temperature changes the ratio between the components Fe(III) (Fig. 16) and Fe(II) (Fig. 17) significantly. With the smaller nanowire diameter the annealing temperature above which Fe(III) becomes dominant compound decreases.

After this research additional samples with nanowires of 15 nm diameter, were annealed at 450 °C for different durations up to 140 min. No α -Fe was found for 15 nm

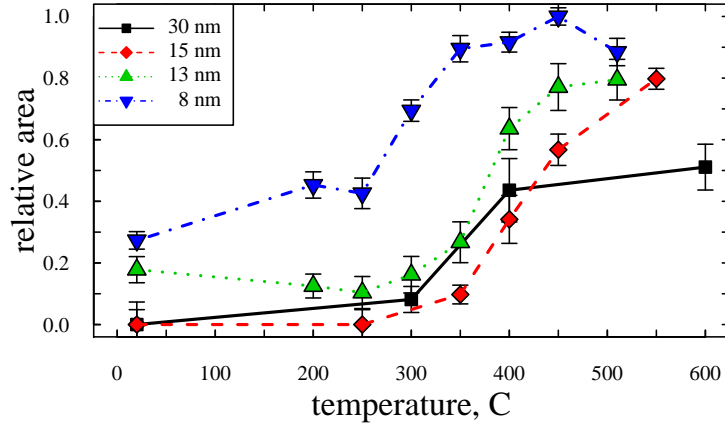


Figure 17 Dependence of contents of $(Fe_xAl_{1-x})_2O_3$ in AAO template on annealing temperature

samples above this annealing temperature (Fig. 15). The dependence of the phase composition on annealing time (Fig. 18) showed a very sharp decrease in the α -Fe content during first 20 min. Afterwards it decreased almost linearly. A decrease in α -Fe content within first 20 min. is followed by a rapid formation of Fe(II) compound. The dependence of content of all phases is nearly linear for annealing durations above 20 min. (Fig. 18).

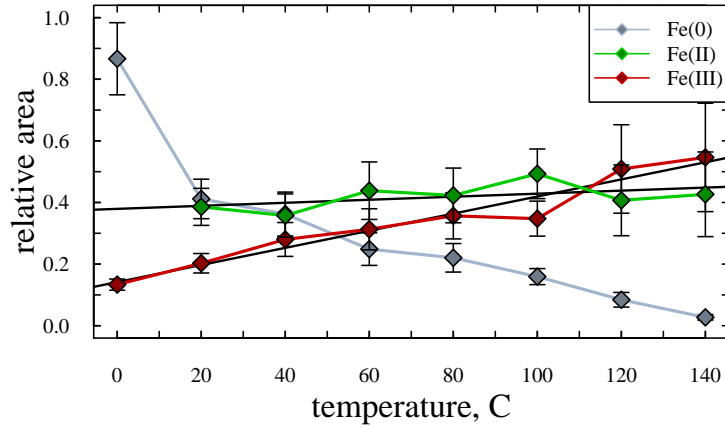


Figure 18 Dependence of contents of iron species in AAO template annealed at $450^\circ C$ on annealing time

Section conclusions:

- sealing the alumina matrix just after deposition of iron protects iron nanowires from spontaneous corrosion under ambient conditions;

- annealing temperature, at which metallic nanowires encapsulated inside AAO template disappears, is lower for templates with smaller pore diameter;
- Fe atoms diffuse into the alumina lattice during annealing and they form paramagnetic compounds: hercynite FeAl_2O_4 and $(\text{Fe}_x\text{Al}_{1-x})_2\text{O}_3$;
- metallic iron reacts rapidly at critical annealing temperature. Amount of α -Fe decreases from 100 % to 40 % in just 20 min.

Conclusions

1. RSMR spectrometer is equipped with high resolution detector, which allowed to decrease observed linewidth from 1.3 keV to 0.3 keV and increase sensitivity from 1 % to 40 %.
2. A new type RSMR spectrometer was developed, which due to favorable time scale of scattering process is suitable for the studies of complicated dynamics of biomolecules (not containing mossbauer nucleus).
3. Iron nanowires get deposited faster into AAO templates, in new aqueous electrolyte, composed of FeSO_4 , MgSO_4 ir citric acid. 52 ± 2 nm/min and $11,8 \pm 0,6$ nm/min respectively.
4. Metallic iron nanowires encapsulated inside AAO template (pore diameter ranges from 8 nm to 30 nm) transforms to Fe(II) and Fe(III) compounds, forming hercynite FeAl_2O_4 and $(\text{Fe}_x\text{Al}_{1-x})_2\text{O}_3$ ($x \leq 0.15$) when annealing inside alumina for 1.5 h at temperatures up to 600 °C.
5. The annealing temperature, at which metallic nanowires encapsualted inside AAO template disappears, is lower for the templates with smaller pore diameter;

List of publications by the author

Articles in journals:

1. J. Reklaitis, D.A. Baltrūnas, V. Remeikis and K. Mažeika, Application of semiconductor detector for recording the Rayleigh scattering of Mössbauer radiation, Lithuanian journal of physics **50**(4), 419-426 (2010).
2. A. Jagminas, K. Mažeika, J. Reklaitis, V. Pakštas and D.A. Baltrūnas, Annealing effects on the transformations of Fe nanowires encapsulated in the alumina template pores, Materials chemistry and physics **115**(1), 217-222 (2009).
3. K. Mažeika, J. Reklaitis, A. Jagminas and D.A. Baltrūnas, Studies of oxidation of iron nanowires encased in porous aluminium oxide template, Hyperfine interactions **189**, 137-142 (2009).
4. A. Jagminas, K. Mažeika, J. Reklaitis, M. Kurtinaitienė and D.A. Baltrūnas, Template synthesis, characterization and transformations of iron nanowires while aging, Materials Chemistry and Physics **109**(1), 82–86 (2008).

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1. J. Reklaitis, D.A. Baltrūnas and V. Remeikis, Detektoriaus parinkimas Mesbauerio spinduliuotės Relėjaus sklaidos spektrometrui. 38-oji Lietuvos nacionalinė fizikos konferencija: programa ir pranešimų tezės, 2009 m. birželio 8 – 10, Vilnius, p. 303.
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