

Phase Composition and Magnetoresistive Properties of Film Systems Based on Ferum and Vanadium or Nikel and Vanadium

T.M. Grychanovs'ka, V.V. Bibyk, I.P. Buryk, O.S. Gryschuk, L.A. Sheshenya

Sumy State University, 2, Rymsky-Korsakov Str., 40007 Sumy, Ukraine

(Received 02 November 2012; revised manuscript received 14 February 2013; published online 28 March 2013)

The paper describes methods of forming three-layer structure based on thin films Ni, Fe and V and research of the structure and phase composition of the obtained samples. The results of investigation magnetoresistive properties of film systems Ni/V/Ni, Fe/V/Fe and their dependence on annealing temperature and thickness of the nonmagnetic layers are presented. Correlation of magnetoresistance properties and structural-phase state of films and its components are established.

Keywords: Phase composition, Three-layer film systems, Magnetoresistance, Size dependence.

PACS numbers: 68.55.Jk, 72.15.Eb, 72.15.Lh, 72.15.Qm

1. INTRODUCTION

The development of modern nanoelectronics is closely associated with the new technologies of memory magnetic devices, intelligent sensors, etc. From the standpoint of practical use of giant magnetoresistance effect [1-5] it is important to find materials that will provide the greatest possible value of magnetoresistance ratio $(R - R_s) / R$, (where R – resistance in an external magnetic field, R_s - resistance at saturation). The high level of minimizing film element requires consideration of the manifestations of these different kinds of size effects [6, 7]. This is due to the fact that the size-limited cells, along with bulk electron scattering mechanisms have a significant role in the scattering of the sample surface, on the edge of the crystallite, the interphase boundaries of impurity atoms. Thus, the limitations of the geometric dimensions of the film and/or crystalline grains lead to decreasing in the mean free path of carrier's electric current. In addition, magnetic non-uniform film materials appear spin-dependent scattering of electrons in antiparallel magnetic moments of individual ferromagnetic layers or granules, or magnetic moments of impurity atoms. In the heating process of film samples or the influence of the magnetic field appear additional changes of the mean free path of the carriers of electric current and conductivity [8, 9]. The above shown phenomena are manifested in the size dependence of many properties and including magnetoresistance.

Nano-structures with spin valve and tunnel magnetoresistive effects that may occur in weak magnetic fields are of considerable practical interest.

The purpose of this paper was the investigation of phase composition and the magnetoresistive properties of three-layer film systems Ni (50) / V (dV) / Ni (10) / S and Fe (40) / V (dV) / Fe (10) / S and determination of the size dependence of magnetoresistance (MR).

2. EXPERIMENTAL

Thin metal films Ni, Fe, and three-layer film systems Ni / V / Ni, Fe / V / Fe were obtained by thermal

evaporation in a vacuum of 10^{-3} - 10^{-4} Pa. The amorphous plates were used as substrate for investigation magnetoresistive properties. The evaporation three-layer film systems were carried out layer by layer from two evaporators on heated substrate (350-400 K). Thicknesses of some layers were controlled in situ by method of quartz resonator. The total thickness of film systems was controlled by the interferometric method. The samples were annealed in the temperature range from 320 to 900 K.

Phase structural sample's composition was investigated with the help of TEM-125K.

The study of magnetoresistive properties was carried at the room temperature in an alternating external magnetic field induction from 0 to 600 mT. All measurements were carried out at the three geometries: parallel (the magnetic induction vector B and direction of the current collinear); transverse (vector B lies in the plane of the sample and perpendicular to the direction of current); perpendicular (vector B perpendicular to the plane of the sample and the direction of the current).

3. RESULTS AND DISCUSSION

3.1 Film systems based on Ni and V

Phase diagram of Ni-V in the range of high temperatures is rather complex, and it is characterized by the presence of intermediate phases. In conducted experiments double-layer films based on Ni and V in the non-annealed state had phase bcc-V + fcc-Ni (Fig. 1). The lattice parameter $a = 0,303$ nm (film V) and $a = 0,353$ nm (film Ni), which is close to the lattice parameters of single-layer films and bulk samples of V and Ni [10]. Note that grain size of film in the unannealed state is 15-20 nm. Annealing at a temperature $T < 700$ K does not change the phase composition of samples (Table 1), and does not cause recrystallization process. There is a slight parallel in the lattice parameter of Ni to the value $a = 0,354$ nm and a perpendicular in the lattice parameter of V to the value $a = 0,305$ nm in the annealed films state at a temperature of

* The material in the article was reported at the 2nd International Conference «Nanomaterials: Applications & Properties-2012»

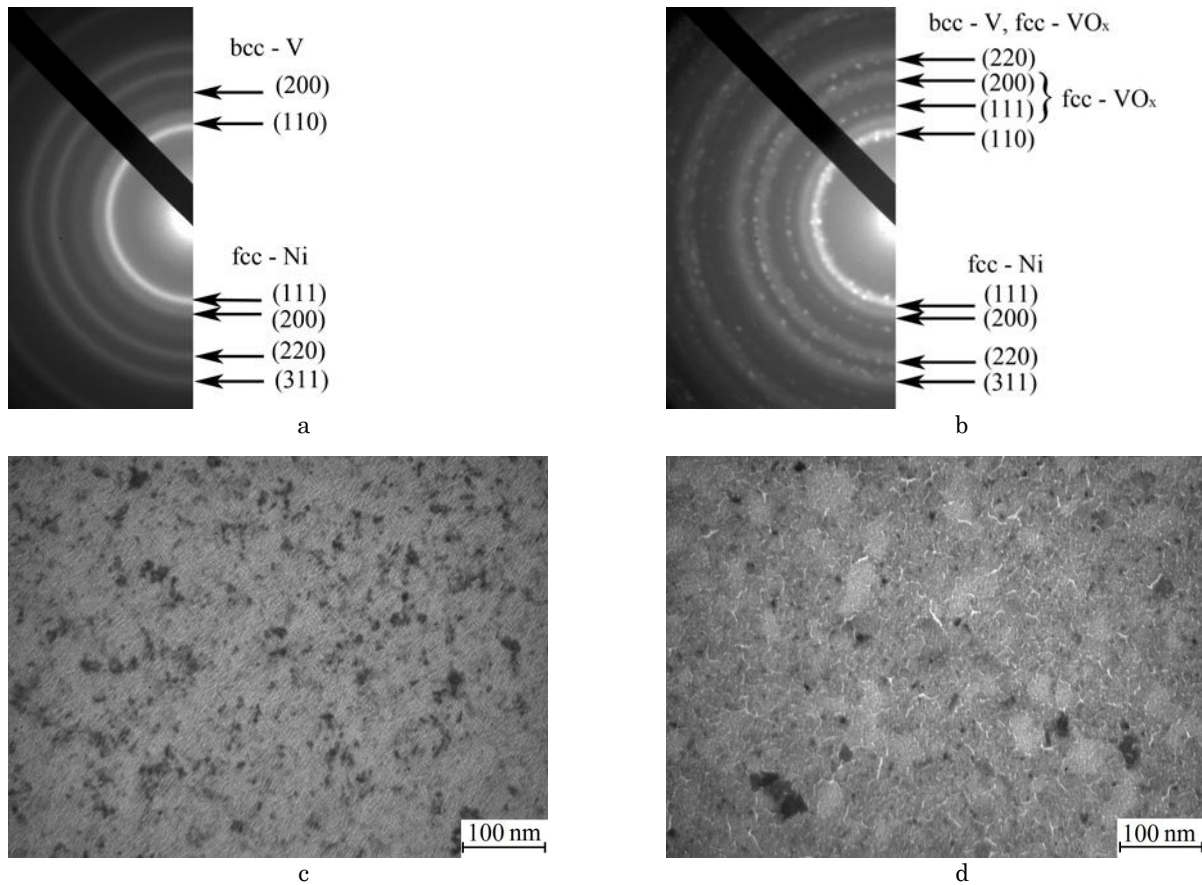


Fig.1 – Diffraction pattern (a, b) and crystalline structure (c, d) of film system Ni(45) / V(10) / Ni(20) / S annealed to 320 K (a, c) and 720 K (b, d). S – substrate. The value of thickness is in nm

Table 1 – The interpretation of diffraction pattern for film system Ni(45)/V(10)/ Ni(20)/S

№	After annealing to 320 K					After annealing to 720 K				
	I, a.u.	d_{hkl} , nm	hkl	phase	a , nm	I, a.u.	d_{hkl} , nm	hkl	phase	α , nm
1	V H	0,213	110	bcc-V	0,301	V H	0,213	110	bcc-V	0,301
2	V H	0,203	111	fcc-Ni	0,352	V H	0,204	111	fcc-Ni	0,353
3	m	0,177	200	fcc-Ni	0,354	m	0,177	200	fcc-Ni	0,354
4	l	0,152	200	bcc-V	0,304	m	0,160	-	VO _x	-
5	H	0,125	220	fcc-Ni	0,352	m	0,131	-	VO _x	-
6	H	0,107	311	fcc-Ni	0,355	H	0,125	220	fcc-Ni	0,352
7	m	0,081	331	fcc-Ni	0,353	m	0,109	220	bcc-V	0,308
8						m	0,107	311	fcc-Ni	0,355
$\bar{a}(\text{bcc-V}) = 0,303 \text{ nm}$; $\bar{a}(\text{fcc-Ni}) = 0,353 \text{ nm}$ $a_0(\text{bcc-V}) = 0,304 \text{ nm}$; $a_0(\text{fcc-Ni}) = 0,352 \text{ nm}$ [10]						$\bar{a}(\text{bcc-V}) = 0,305 \text{ nm}$; $\bar{a}(\text{fcc-Ni}) = 0,354 \text{ nm}$ $a_0(\text{bcc-V}) = 0,304 \text{ nm}$; $a_0(\text{fcc-Ni}) = 0,352 \text{ nm}$ [10]				

H – high, m – medium, l – low.

$700 \leq T < 800 \text{ K}$. For annealed samples based on Ni and V, with increase in temperature to $T = 800\text{-}830 \text{ K}$, the most part of the volume will occupy solid solution (Ni-V), and fcc phase VO_x is formed instead of Vanadium layer [11, 12].

Further annealing in the temperature range of 850-900 K stimulates the increase of termodiffusion of atoms V in grain boundaries with their accumulation on the outer surface Ni and in the bulk grains. Lattice parameter solid solution (Ni-V) increases and strongly depends on the concentration of vanadium. For the samples with the concentration of = 20-25 at. % $a = 0,350 \text{ nm}$ [12].

For research of magnetoresistive effect all samples of Ni (50 nm) / V(dv) / Ni (10 nm) / S were obtained

under identical technological conditions. The results of electron diffraction and electron microscopy confirm that all samples have the phase composition of bcc-V + fcc-Ni and the polycrystalline structure.

Experimental studies of magnetoresistive properties of thin-film systems have shown that the non-annealed samples, with a layer of vanadium < 3 nm have anisotropic magnetoresistance (AMR), and magnetoresistive hysteresis loops similar to the corresponding single-layer films of ferromagnetic metals (Ni, Fe). The value of the longitudinal and transverse magnetoresistance (MR) does not exceed 0.01-0.05 %. Annealing of these samples at a temperature $T = 600\text{-}650 \text{ K}$ is accompanied by a slight increase of MR.

The absolute values of MR unannealed samples with a thickness of the nonmagnetic layer = 3-11 nm, an order of magnitude greater than previous, and largely depend on the phase composition and concentration of the components of the system (Fig. 2). In samples where it was possible to avoid the formation of solid solution, MR increases with increasing annealing temperature and for films annealed at 650 K is 0.2-0.6 % of the longitudinal and 0.1-0.5 % of the perpendicular MR.

Isotropy of the magnetoresistive properties (Fig. 2a, b, c) is observed for film systems with the atomic concentration = 12-14 at. %. It appears to reduce the electrical resistance in a magnetic field regardless of the measurements geometry, which can be considered one of the signs of giant magnetoresistance [1-5].

Oscillation of the exchange coupling between the ferromagnetic layers is an important feature of magnetic nanostructures. It depends on the thickness of the nonmagnetic layer, which leads to oscillations of the magnetic and magnetoresistive properties of the film structures. The strength of coercivity (H_c) and the saturation field (H_s) are the important characteristics that give qualitative information on the behavior of the

multilayer film systems in the process of magnetization reversal. The magnetic field is required to complete demagnetization of the sample (B_c) by analogy with H_c and H_s , and the saturation induction (B_s) can be used [9]. The results of the influence of the indirect exchange between ferromagnetic layers of Ni on the value of the coercive force of Ni (50 nm) / V (dv) / Ni (20 nm) are shown in Fig. 3. Maximum induction demagnetization B_c is observed at ≈ 3 nm and ≈ 11 nm, which corresponds to the maxim of the saturation induction B_s , i.e. there is the antiferromagnetic coupling of ferromagnetic Ni layers in these film systems [1].

3.2 Film systems based on Fe and V

The system Fe-V is characterized by continuous solubility in the liquid and solid states therefore it is difficult to get the Fe (40 nm) / V (dv) / Fe (10 nm) / S system. The phase diagram Fe-V analysis showed that there is a continuous series of solid solutions between α -Fe and V at high temperatures. The formation of an intermediate phase σ (Fe-V) is possible at 1219 °C and concentration of 48 % at. V. The existence region of the phase expands when the temperature drops to 600 °C,

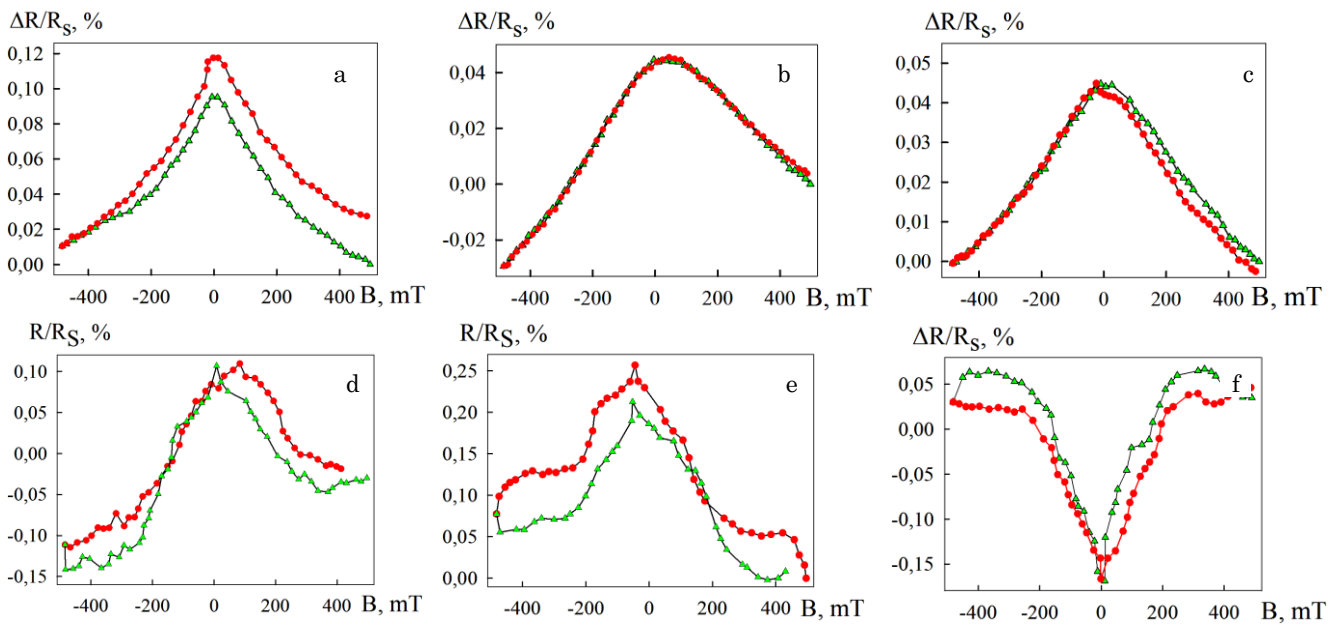


Fig. 2 – Field dependence of MR at $T = 320$ K for the film system of Ni (45) / V (10) / Ni (10) / S (a, b, c) and Ni (35) / V (5) / Ni (25) / S (d, e, f). Measurement geometries: parallel (a, d), perpendicular (c, e) and transverse (d, f)

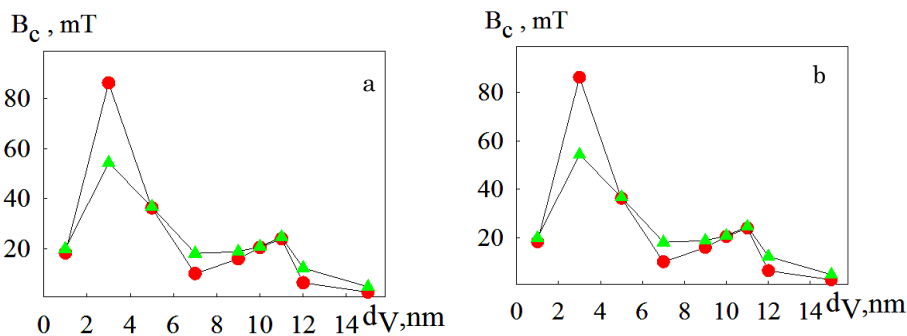


Fig. 3 - The dependence of the induction demagnetization (a) and saturation induction (B) from the thickness of the layer V of films Ni (50) / V (dv) / Ni (10) unannealed (●) and annealed (▲) at 650 K

but there is no information about the phase stability in the range of lower temperatures. Calculation of electron diffractions of film systems Fe / V / Fe showed that in nonannealed state the film systems have the composition of bcc-Fe + bcc - V (Fig. 4) and lattice parameters of $a(\text{Fe}) = 0,287 \text{ nm}$ and $a(\text{V}) = 0,301 \text{ nm}$. In annealed samples at 720-650 K one blurred line Fe_3O_4 is graphically recorded by electron, and phase composition corresponds to bcc-Fe + bcc - V + Fe_3O_4 (Table 2). The crystal structure analysis of the samples shows its nano dispersion in nonannealed state: average crystallite size less than 5 nm. Annealing at 650-720 K leads to minor recrystallization processes and average crystallite size increases to 10 nm. Annealing at 650-720 K leads to the formation of the bcc of solid solution the lattice parameter of which depends on the concentration of vanadium [13, 14].

In samples with < 10 nm the calculation pattern diffraction did not show the presence of vanadium, lattice

parameter $a(\text{Fe}) = 0,289 \text{ nm}$. In samples with = 10-12 nm the calculation pattern diffraction showed that the Fe lattice parameter did not change $a(\text{Fe}) = 0,287 \text{ nm}$, and vanadium lattice parameter $a(\text{V}) = 0.301 \text{ nm}$. Annealing at 650 K did not change the phase state and lattice parameters. The crystal structure analysis showed that all samples in the unannealed state nano-dispersed with an average crystallite size of less than 5 nm. Annealing at a temperature of 650-700 K leads to a slight recrystallization processes: it increases the average crystallite size of 5-10 nm and $a(\text{Fe}) = 0,287 \text{ nm}$, $a(\text{V}) = 0.304 \text{ nm}$.

Isotropy of the magnetoresistive properties, which appears to reduce the electrical resistance in a magnetic field, regardless of the geometry of the measurements, is observed for three-layer film systems with the atomic concentration = 22-30 % at. In systems with layers < 2 nm field dependences of anisotropic (AMR)

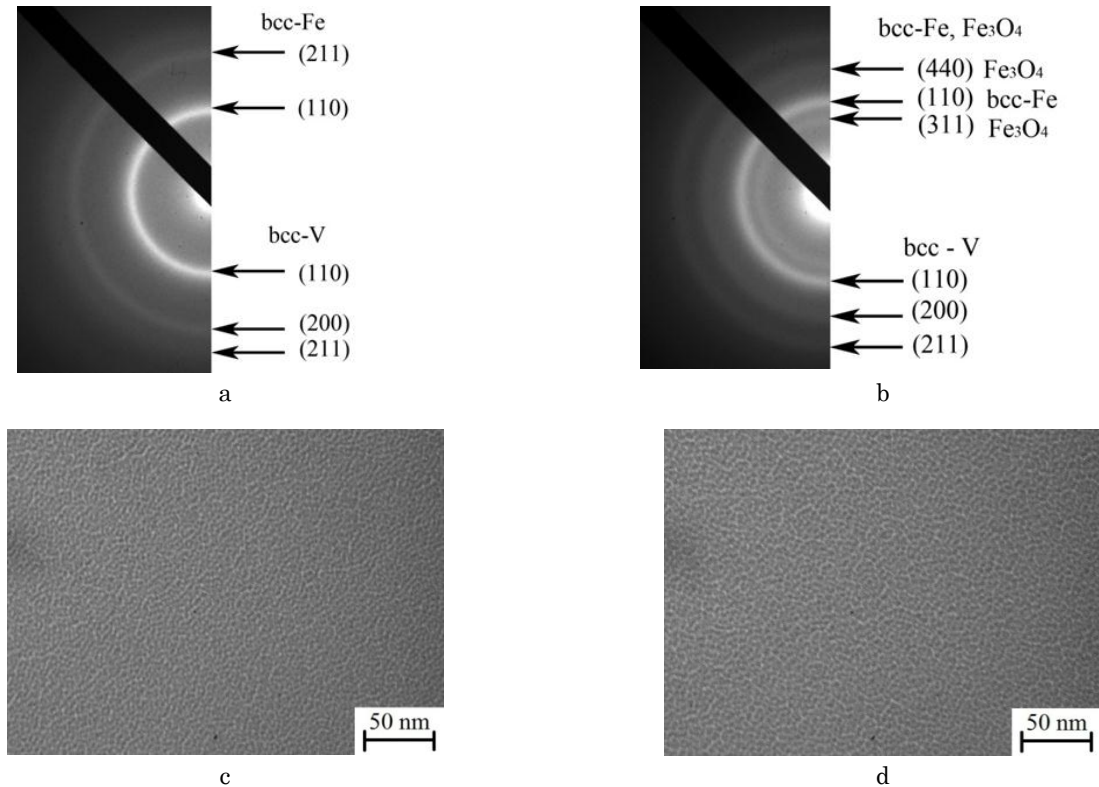


Fig. 4 – Diffraction pattern (a, b) and crystalline structure (c, d) of film system Fe(40) / V (10) / Fe (20) / S after condensation at $T_s = 320 \text{ K}$ (T_s – temperature of substrate) (a, c) and after annealing to 720 K (b, d)

Table 2 – The interpretation of diffraction pattern for film system Fe(40)/V(10)/Fe(20)/S

№	After condensation to 320 K					After annealing to 720 K				
	<i>I</i> , a.u.	<i>d_{hkl}</i> , nm	hkl	phase	<i>a</i> , nm	<i>I</i> , a.u.	<i>d_{hkl}</i> , nm	hkl	phase	<i>a</i> , nm
1	-	-	-	-	-	H	0,251	-	Fe_3O_4	-
2	V H	0,213	110	bcc-V	0,301	V H	0,214	110	bcc-V	0,302
3	V H	0,201	110	bcc-Fe	0,284	V H	0,203	110	bcc-Fe	0,287
4	l	0,150	200	bcc-V	0,300	l	0,152	200	bcc-V	0,305
5	l	0,145	200	bcc-Fe	0,290	m	0,152	-	Fe_3O_4	-
6	l	0,123	211	bcc-V	0,301	m	0,124	211	bcc-V	0,304
$\bar{a}(\text{bcc-V}) = 0,301 \text{ nm}$; $\bar{a}(\text{bcc-Fe}) = 0,287 \text{ nm}$ $a_0(\text{bcc-V}) = 0,304 \text{ nm}$; $a_0(\text{bcc-Fe}) = 0,287 \text{ nm}$ [10]					$\bar{a}(\text{bcc-V}) = 0,304 \text{ nm}$; $\bar{a}(\text{bcc-Fe}) = 0,287 \text{ nm}$ $a_0(\text{bcc-V}) = 0,304 \text{ nm}$; $a_0(\text{bcc-Fe}) = 0,287 \text{ nm}$ [10]					

H – high, m – medium, l – low.

are similar to the films of Fe. In the sample with dv of 2 to 10 nm, the $\Delta R/R_s$ (B) dependences for the longitudinal and transversal MR are almost coincident, and for some films, the longitudinal MR exceeds insignificantly size of the magnetoresistance in the transversal geometry. The magnetoresistance value attains 0,11 % for the sample Fe (40) / V (10) / Fe (20) / S at room temperature in transverse geometry (Fig. 5), while AMR not exceeding 0,01 % is observed for unannealed films.

Thus, the GMR effect seems to be realized in the three-layer systems Fe (40) / V (dv) / Fe (10) / S and Fe (40) / V (dv) / Fe (20) / S (= 2-14 nm). The presence of

a horizontal section on the dependence of $\Delta R/R_s$ of vector \mathbf{B} shows the independent movement of the magnetic moments for the upper and lower layers [15]. The GMR results in various scattering of two electron groups different in spin orientation relative to the magnetization direction of the magnetic structure that scatters the electrons. [1]. Feature samples Fe (40) / V (dv) / Fe (10) / S have the highest value of MR corresponding transverse and perpendicular geometry (0,12-0,14 %), whereas for the longitudinal magnetoresistance geometry is 0,03-0,06 %, or even less.

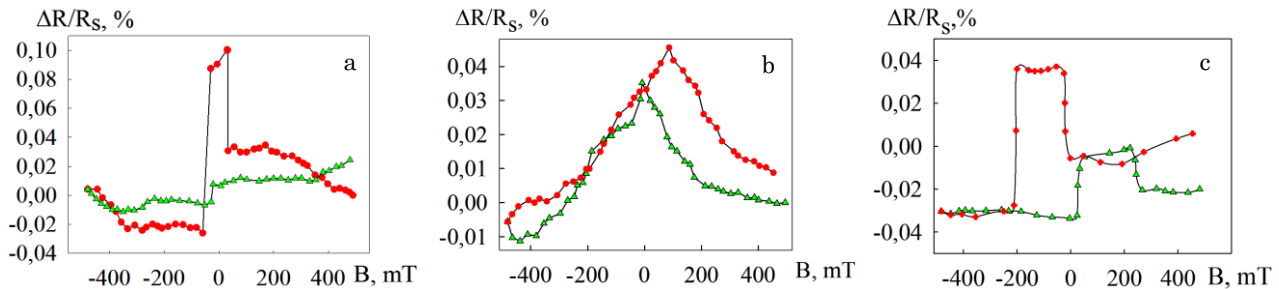


Fig. 5 – Field dependence of MR at $T = 320$ K for the film systems Fe (40) / V (10) / Fe (20) / S (a), Fe (40) / V (14) / Fe (20) / S (b) and Fe (40) / V (10) / Fe (10) / S (c). Transverse geometry

4. CONCLUSIONS

We have studied the structure-phase state and the magnetoresistive properties of Ni (50) / V (dv) / Ni (10) / S and Fe (40) / V (dv) / Fe (10) / S three-layer film systems obtained by the condensation method. The carried out investigations showed that in the unannealed Fe (40) / V (10) / Fe (10) / S and Ni (45) / V (10) / Ni (10) / S films the GMR effect is realized. Systems have isotropic magnetoresistance, they promise to create a spin-valve structure. It is shown that annealing systems in the 300-600 K temperature range retain their structural and phase stability.

For the unannealed films Fe (40) / V (dv) / Fe (10) / S (= 2-14 nm) and the longitudinal magnetoresistive effect is not observed. The unannealed samples Ni (40) / V (dv) / Ni (10) / S with a layer of vanadium < 3 nm have anisotropic magnetoresistance

The transition from the GMR to the AMR takes place after three-layer film Ni (50) / V (dv) / Ni (10) / S and Fe (40) / V (dv) / Fe (10) / S annealing at 720 K.

This work is done in the framework of the state project №0112U001381.

Фазовий склад та магніторезистивні властивості плівкових систем на основі Феруму і Ванадію або Нікелю і Ванадію

Т.М. Гричановська, В.В. Бібик, І.П. Бурик, О.С. Гришук, Л.А. Шешеня

Сумський державний університет, Римського-Корсакова, 2, 40007 Суми, Україна

У роботі наведено методику формування тришарових структур шляхом пошарової конденсації нанорозмірних плівок Ni, Fe і V та проведено дослідження структури і фазового складу отриманих зразків. Розглянуто магніторезистивні властивості плівкових систем Ni / V / Ni, Fe / V / Fe і їх залежність від температури відпалювання та товщини немагнітного прошарку. Встановлено кореляцію магніторезистивного ефекту з структурно-фазовим станом плівкової системи та її складових.

Ключові слова: Фазовий склад, Тришарові плівкові системи, Магнітоопір, Розмірні залежності.

Фазовый состав и магниторезистивные свойства пленочных систем на основе Феруму и Ванадия или Никеля и Ванадия

Т.М. Гричановская, В.В. Бирик, И.П. Бурик, А.С. Гришук, Л.А. Шешеня

Сумский государственный университет, Римского-Корсакова, 2, 40007 Сумы, Украина

В работе приведена методика формирования трехслойных структур путем послойной конденсации наноразмерных пленок Ni, Fe и V и проведено исследование структуры и фазового состава полученных образцов. Рассмотрены магниторезистивные свойства пленочных систем Ni/V/Ni, Fe/V/Fe и их зависимость от температуры отжига и толщины немагнитного слоя. Установлена корреляция магниторезистивного эффекта со структурно-фазовым состоянием пленочной системы и ее составляющих.

Ключевые слова: Фазовый состав, Трехслойные пленочные системы, Магнитосопротивление, Размерные зависимости.

REFERENCES

1. U. Hartman, *Magnetic Multilayers and Giant Magnetoresistance: Fundamentals and Industrial Applications* (Berlin: Heidelberg: New York: Springer-Verlag: 2000).
2. T.V. Ustinov, A.B. Rinkevich, L.N. Romashev, D. Perov, *Tech. Phys.* **74**, 94 (2004).
3. A.N. Stetsenko, V. Samofalov, V. Zorchenko, *JETP Lett.* **64**, 346 (1996).
4. F.S. Bergeret, A.F. Volkov, K.B. Efetov, *Rev. Mod. Phys.* **77**, 1321 (2005).
5. A.B. Svalov, P.A. Savin, G. Kurlandskaya, J. Gutiérrez, V.O. Vas'kovskiy, *Tech. Phys.* **72**, 54 (2002).
6. V. Yudyntsev, *Electronics: Science, Technology, Business No 1*, 52 (2008).
7. I.A. Garifulin, N. Garifyanov, R.I. Salikhov, *J. Exp. Theor. Phys.* **105**, 227 (2007).
8. A.P. Boltaev, F.A. Pudonyn, I.A. Sherstnyov, *FTT* **53**, 892 (2011).
9. O. Synashenko, D.N. Kondrahova, I.E. Protsenko, *J. Nano-Electron. Phys.* **2** No4, 153 (2010).
10. S.S. Gorelik, L.N. Rastorguev, Y. Skakov, *X-ray and Electron Graphical Analysis of Metals* (Moscow: GNTI: 1963).
11. I.Yu. Protsenko, V.V. Tokman, A.M. Chornous, I.O. Shpetny, *Metallofiz. Nov. Tekh.* **25** No3, 319 (2003).
12. V.V. Bibik, T.M. Grychanovska, L.V. Odnodvoretz, I.V. Cheshko, I.Yu. Protsenko, *Thin Films in Optics and Nanoelectronics – Book of abstract 18th International Symposium* **2**, 134 (Kharkiv: NNTs KhFTI: 2006).
13. V. Uzdin, K. Westerholt, H. Zabel, B. Hjörvarsson, *Phys. Rev. B* **68**, 214407 (2003).
14. E. Holmström, L. Nordström, L. Bergquist, B. Skubic, B. Hjörvarsson, I.A. Abrikosov, P. Svedlindh and O. Eriksson, *P. Natl. Acad. Sci. USA*, **101** No.14, 4742, (2004).
15. A.V. Slavov, G.V. Kurlyandskaya, H. Hammer, P.A. Savin, O.I. Tutynina, *Tech. Phys.* **49**, 868 (2004).