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Mechanical and Electric Properties of $Ni_xCo_{1-x}Fe_2O_4$ Ferrites

V.S. Bushkova*, I.P. Yaremiy, R.V. Ilnitsky, B.S. Dzundza, O.M. Matkivsky

Vasyl Stefanyk Precarpathian National University, 57, Shevchenko Str., 76025 Ivano-Frankivsk, Ukraine

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The Ni-Co ferrites were prepared by the technology sol-gel with participation of auto-combustion and sintered at temperature 1573 K in air. The effects of Ni^{2+} ions addition on the microstructure and electric properties of $Ni_xCo_{1-x}Fe_2O_4$ ferrites were systematically studied. The added of Ni^{2+} ions significantly affects the formation of pores and grain size of ferrites. By doping with nickel ions the pores decreased and a dense material are obtained. The micro-hardness values increases from 5,16 GPa to 8,59 GPa with increasing nickel contents. The Hall coefficient, conductivity type, concentration of charge carriers and specific conductivity were found. In Ni-Co ferrites Hall mobility is within the limits from $7.04 \cdot 10^{-1}$ cm²/V·s to 4.38 cm²/V·s.

Keywords: Ferrite, Microstructure, Micro-hardness, Hall effect, Electrical conductivity.

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1. INTRODUCTION

Ferrites are semiconductors in nature having a wide range of resistivity from 10^{-3} to 10^{10} Ohm·cm at room temperature [1]. The high resistivity of ferrites is explained on the basis of location of cations in the spinel structure and also the hopping mechanism [2]. Most of the high frequency applications related to electric properties are concerned with dielectrics. The advantages of the ferrites, which are ceramic in nature, over the other available dielectric materials are elastic properties, and greater resistance to environmental changes, particularly at higher temperature.

Mechanism of charge transport can be understood from the measurement of electrical conductivity and Hall coefficient. Concentration and mobility of charge carriers are the key quantities for obtaining information of the ferrite conductivity. Therefore the knowledge of these parameters and the mechanical properties are highly important for their use. The micro- hardness of material is an important mechanical property because it relates how much the material will inelastic deformed when a surface load is applied. Sometimes, hardness is increased when the grain size is decreased in the ultrafine range [3].

Hence, the present paper is devoted to studying the effect of Ni^{2+} ions substitution on the microstructure, mechanical and electric properties of cobalt ferrite.

2. EXPERIMENTS

Ferrites with the general formula $Ni_xCo_{1-x}Fe_2O_4$ ($x = 0.0$, 0.1, 0.2, 0.3, 0.4 and 0.5) have been prepared by SGA technique [4]. The X-ray diffraction (XRD) patterns were recorded at room temperature on Dron 3 X-ray diffractometer using CuKα radiation. The scanning was done in the 2θ range from 15° to 60°. After completing the process auto-combustion was obtained one phase of ferrite powders which corresponded to the cubic structure of spinel space group $Fd3m$.

The average size of coherent scattering regions of powders was in the range 39-62 nm. The resulting powders were pressed under a pressure of $3{,}3 \cdot 10^8$ Pa die to make pallets. The pressed samples were then finally sintered at 1573 K for 5 h in air and cooled in the furnace.

The microstructure of samples after etching was analyzed by optical microscopy. Grain sizes were determined with the aid of a digital image processor program, hooked to the microscope. The mechanical properties of pallets were obtained from Vickers microhardness measurements using a NEXUS 412A tester at the transverse surface of the sintered samples at a time of 15 s, with a minimum of 3 indentations per sample. A 3 N load was used to measure the microhardness values. A computer program was used to analyze the image and calculate the micro-hardness.

The measurement of Holl parameters of samples was carried out in air at a temperature of 300 K in permanent magnetic fields by using automated installation, which provides processes for measuring electrical parameters, recording and primary data processing. The measured sample had two Hall and two current contacts. The current through the samples was about 100 μA. The magnetic field at an induction of 1.6 T was directed perpendicularly to the pellets base.

The conducting characteristics of the investigated samples were determined by the parameters of complex impedance, the measurement of which was carried out using the Autolab PGSTAT 12/FRA-2 spectrometer in the frequency range of 10^{-2}-10^6 Hz.

3. RESULTS AND DISCUSSION

3.1 Micro-structural Study

The microstructure and compositional analysis of all the ferrite samples were studied systematically by optical microscopy. The samples for optical microscopy were prepared by polished in three stages of 6, 3, and 1 μm on a polishing wheel using diamond spray. All the samples were etched firstly with 2 % nitric acid solution for few seconds to reveal the microstructure of the affected areas.

* bushkovavira@gmail.com

According to analysis of X-ray diffraction patterns of $Ni_xCo_{1-x}Fe_2O_4$ pallets crystallite size was lying in the range of 42 nm to 94 nm. Low magnification (× 400) optical micrographs were attained to determine the grain size. The results from optical micrograph show the chemical homogeneities and porosity along with grains formation. All the samples of $Ni_xCo_{1-x}Fe_2O_4$, except $x = 0.0$, are homogeneous and free from any sort of macro-porosity (Fig. 1).

The decrease of porosity by increasing Ni^{2+} concentration in this series of ferrite is again clear from optical electron micrographs. The grain size of the ferrite pellets is about 0,5-1,4 μm. It is to be noted that for all the compositions of the studied ferrites the addition of Ni ions suppressed the grain growth and causes the grain size to decrease.

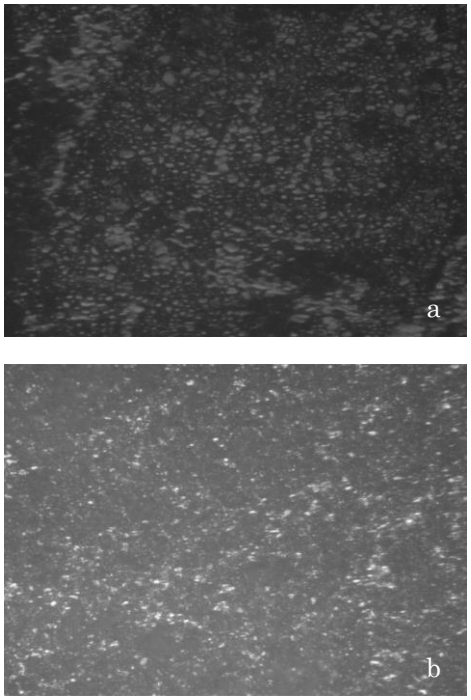

Fig. 1 – Optical micrograph of Ni-Co ferrites: a – $CoFe_2O_4$, b – $Ni_{0.5}Co_{0.5}Fe_2O_4$

3.2 Micro-hardness Study

Micro-hardness tests of all the compositions of ferrite were performed on the same samples prepared for the optical microscopy. The Vickers micro-hardness in [MPa] depends on the size of the indentation and the applied load, and is determined by the formula:

$$H_V = \frac{F}{S}, \qquad (1)$$

where F is load in [N], S is indentation area in [mm^2], from here we have:

$$H_V = \frac{2F \cdot \sin\left(\alpha/2\right)}{d^2}, \qquad (2)$$

where a is a dihedral angle of a diamond pyramid at its top ($a = 136°$), d is the arithmetic mean of the both diagonal lengths of the indentation ($d = (d_1 + d_2)/2$).

Then

$$H_V = 1,854 \cdot \frac{F}{d^2}. \qquad (3)$$

On the other hand, the Vickers micro-hardness in [kg·mm^{-2}] is determined by the formula:

$$H_V = 0,102 \cdot \frac{2F \cdot \sin\left(\alpha/2\right)}{d^2} = 0,1891 \cdot \frac{F}{d^2}. \qquad (4)$$

It is well known that microstructure greatly affecting the material properties such as micro-hardness. It is revealed that the hardness measured on all the samples of Ni-Co ferrite compositions showed values, in the range of 5,16-8,59 GPa (Fig. 2). It is clear that the hardness of samples increased with increasing Ni^{2+} content. This is because NiO form double bonding due to some ionic character with O_2, which further increases its polarity and generate strong interaction among ions. The micro-hardness has a maximum value at $x = 0.5$ which will result in a material being more resistance to the indentation at a given load, which will signify that the material will be able to plastically deform more so than the $CoFe_2O_4$ ferrite. The effect of Ni addition on hardness of different compositions of ferrites has also been studied by other researchers [5, 6] and observed that the micro-hardness was increased with Ni addition.

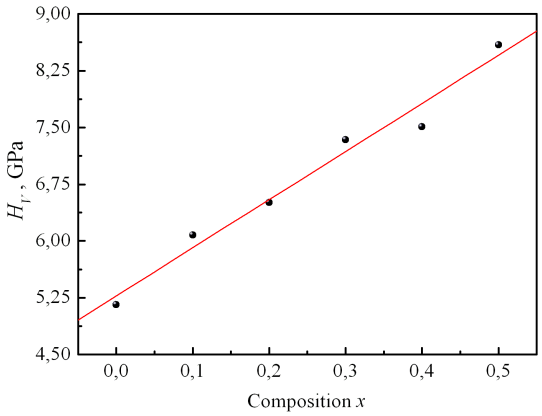

Fig. 2 – The microhardness of $Ni_xCo_{1-x}Fe_2O_4$ ferrites as a function of Ni content

The micro-hardness values obtained are better than those reported in the literature for different compositions of ferrites [7]. In addition, the method of material synthesis also influence on the micro-hardness of ferrites. For example, in the work [8] $Mn_{0.8}Zn_{0.2}Fe_2O_4$ ferrites were obtained in two ways. At first, Mg-Zn ferrite was obtained by ball milling method with oxides MnO, ZnO and Fe_2O_3. The sample was annealed at 973 K for 4 h. The second method was to prepare solutions $Mn(NO_3)_2 \cdot 4H_2O$, $Zn(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ with 65 % HNO_3. The sample was then dried in an over at 383 K and annealed at 973 K in argon for 4 h. In the first case the micro-hardness values is 293,2 MPa, but in the other – 758,3 MPa. Therefore, self combustion method leads to higher micro-hardness and better mechanical properties. This is due to the small size of the grains.

It is well know that relation between yield stress and grain size is described mathematically by the Hall-Petch equation:

$$\sigma_y = \sigma_0 + \frac{K_y}{\sqrt{d}}, \quad (5)$$

where σ_y is the yield stress, σ_0 is a materials constant for the starting stress for dislocation movement (or the resistance of the lattice to dislocation motion), K_y is the strengthening coefficient (a constant unique to each material), and d is the grain diameter.

The Hall-Petch law gives a quantitative description of an increase in the yield stress of a polycrystalline material as its grain size decreases. This relationship is based on dislocation mechanisms of plastic deformation: grain boundaries hinder the movement of dislocations. It is important to note that for nanomaterials with grain sizes of several tens of nanometres this law, to a certain extent, is not observed, giving way to the so-called inverse Hall-Petch effect, whose mechanisms are not well understood yet.

In work [9] it was proposed that the hardness dependence on grain size might follow directly from the Hall-Petch relation. Thus the hardness-grain size relation is described by

$$H_V = H_0 + \frac{K_H}{\sqrt{d}}, \quad (6)$$

where H_0, and K_H are constants. In this case hardness is proportional to $d^{-0,5}$.

Fig. 3 shows the Vickers micro-hardness as a function of the inverse square root of grain size for the Ni-Co ferrites. The dependence is approximated by a linear function $y = A + B \cdot x$:

$$H_V = 0,61856 + 5,50619 \cdot d^{-0,5}, \quad (7)$$

where $H_0 = A$, $K_H = B$.

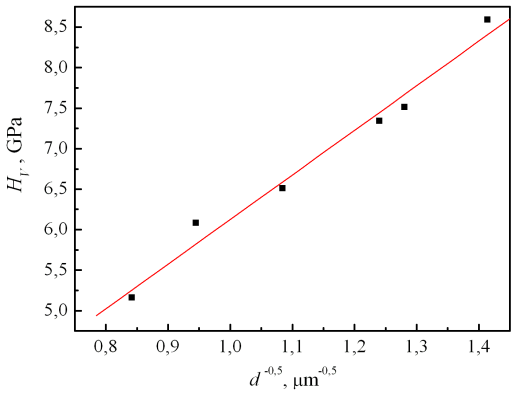

Fig. 3 – Hall-Petch plot for the Ni-Co ferrites

In accordance with the Hall-Petch relation, the hardness decreases linearly for ferrites sintered in higher temperatures, because the grain size increases. In work [10] it was found that for Ni-Zn ferrite samples sintered at 1473 K the densification process in the absence of grain growth enhances the hardness of the samples. The important grain growth is observed at temperatures higher than 1473 K. It reduces the hardness, independently of the densification attained. On the other hand, in work [11] it was found that the micro-hardness increases linearly with sintering temperature for both $NiFe_2O_4$ and $MgFe_2O_4$. The increase in micro-hardness with sintering temperature in these ferrites may be mainly due to the reduction in the porosity of the samples.

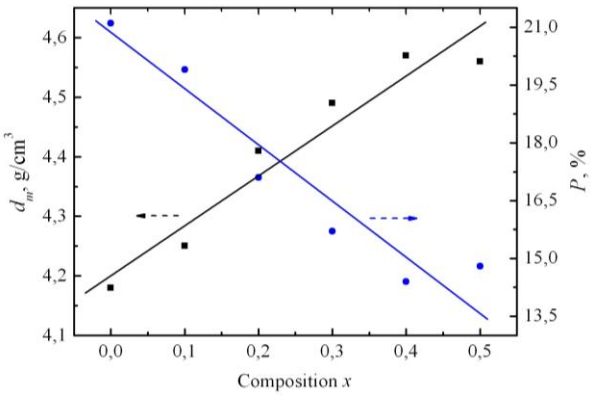

Fig. 4 – Dependences of relative density and porosity from content of Ni^{2+} ions

Fig. 4 shows the composition dependence of relative density and porosity. Because the micro-hardness values are highly correlated with the relative density and porosity, so reducing the number of defects in sample is a common way of increasing its micro-hardness [7].

3.3 Hall Effect

The Hall effect in ferromagnets has been mainly studied in metals. Meanwhile the study of the spontaneous Hall effect in semiconductors is of great interest in at least two respects. First, there exist at present two interpretations of the temperature variation of the spontaneous Hall coefficient R_H in ferromagnets, one of which associates R_H with the resistivity ρ, and the other with the square of the magnetization M_s: $R_H(T) \sim \rho_n(T)$, where $n = 1, 2$; $R_H(T) \sim Ms^2(T)$. The temperature variation of the magnetization has the same character both in metallic and in semiconducting ferromagnets, whereas the temperature variation of the electrical resistance is markedly different. Second, the conduction mechanism in ferrites is not yet entirely clear. The study of the Hall effect, together with the electrical conductivity, can give valuable information on this question.

In the present paper the variation of R_H in nickel-cobalt ferrites is calculated using formula [12]:

$$R_H = \frac{U_H}{IB} \cdot h, \quad (8)$$

where U_H – the potential difference arising between the pallet bases, I – current strength, B – induction of magnetic field, h – height of the pallet. On the other hand, Hall coefficient is defined by expression:

$$R_H = \frac{A}{en}, \quad (9)$$

where e – electron charge, n – concentration of charge

carriers, A – constant (for semiconductors is equal $3\pi/8$). Thus Hall coefficient is inversely proportional to the concentration of charge carriers. Using the formulas (8) and (9) we obtain the following expression for the calculation n:

$$n = \frac{3\pi}{8e} \cdot \frac{IB}{U_H h}, \quad (10)$$

where do we have:

$$n = 7{,}36 \cdot 10^{18} \cdot \frac{IB}{U_H h}. \quad (11)$$

In semiconductors, electrons and holes can participate in the formation of electric current. According to Verwey the conduction mechanism in ferrites is due to exchange of electrons between cations in the same site in the lattice. In cobalt ferrite, the conduction can possibly be attributed to hopping of electrons (e^-) between Fe^{2+} and Fe^{3+} at the octahedral (B) sites of the spinel. At the tetrahedral (A) sites, hole (e^+) is involved in hopping process between Co^{2+} and Co^{3+} [13]. In nickel ferrite, the conduction can possibly be attributed to hopping of electrons (e^-) between Fe^{2+} and Fe^{3+}, and hole (e^+) is involved in hopping process between Ni^{2+} and Ni^{3+} at the octahedral sites.

Thus the probable conduction mechanisms in the Ni-Co system are $Fe^{2+} \leftrightarrow Fe^{3+} + e^-$ (n-type) and $Ni^{3+} \leftrightarrow Ni^{2+} + e^+$ (p-type) at the B sites, and $Co^{3+} \leftrightarrow Co^{2+} + e^+$ (p-type) at the A site of the spinel ferrite. Assuming that two hopping mechanisms are involved, the predominance of one over the other depends upon the concentration of substituted cations. If the electron exchange mechanism dominates to the hole exchange mechanism the ferrite composition might conductas n-type semiconductor (or vice versa).

In Table 1 are listed Hall coefficient, conductivity type and concentration of charge carriers. For samples with $x < 0.4$ the electronic conductivity type prevails, and for $Ni_{0.4}Co_{0.6}Fe_2O_4$ and $Ni_{0.5}Co_{0.5}Fe_2O_4$ ferrites holes conductivity type is predominates. The carrier concentration decreases with increasing Ni content up to $x = 0.2$ inclusive, after which it begins to increase.

The conduction mechanism in $Ni_{0.4}Co_{0.6}Fe_2O_4$ and $Ni_{0.5}Co_{0.5}Fe_2O_4$ ferrites is hopping of electrons between Fe^{3+} and Fe^{2+} ions and hopping of holes between Ni^{+2} and Ni^{+3}, which is the dominant one. The number of holes hopping between Ni^{+2} and Ni^{+3} ions increases with nickel doping. This is because of Fe^{3+} ions migration from the octahedral to the tetrahedral sites.

Table 1 – Hall parameters for $Ni_xCo_{1-x}Fe_2O_4$ system

x	R_H, $cm^3 \cdot C^{-1}$	type	n, cm^{-3}
0.0	$-2.34 \cdot 10^8$	n	$8.66 \cdot 10^{11}$
0.1	$-5.48 \cdot 10^8$	n	$3.74 \cdot 10^{11}$
0.2	$-6.05 \cdot 10^8$	n	$3.45 \cdot 10^{11}$
0.3	$-4.10 \cdot 10^7$	n	$5.09 \cdot 10^{12}$
0.4	$+2.34 \cdot 10^7$	p	$8.93 \cdot 10^{12}$
0.5	$+6.47 \cdot 10^6$	p	$3.17 \cdot 10^{13}$

3.4 Specific Electrical Conductivity Study

For nickel-cobalt ferrites the specific conductivity of the direct current σ_{dc} is estimated using the diagrams $\sigma''(\sigma')$ by extrapolation of the relations between σ'' and σ', which in the region of low frequencies have the form of straight lines (Fig. 5). It is obvious that up to $x = 0.2$ the specific electrical conductivity on a direct current decreases, after which it begins to increase monotonously. The high specific resistivity of samples $Ni_{0.1}Co_{0.9}Fe_2O_4$ and $Ni_{0.2}Co_{0.8}Fe_2O_4$ is likely to indicate their high stoichiometry and the presence in the octahedral sites of a small amount of Fe^{2+} ions, as compared to other ferrite compounds.

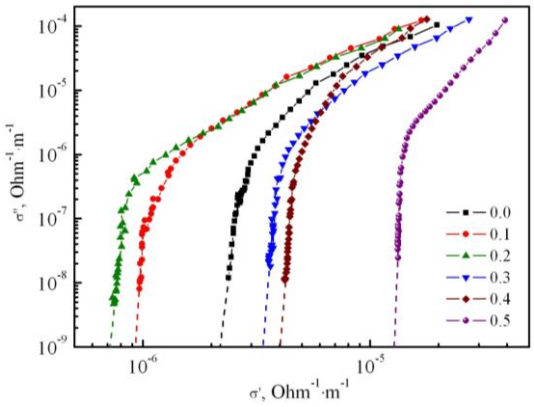

Fig. 5 – The diagrams $\sigma''(\sigma')$ for $Ni_xCo_{1-x}Fe_2O_4$ ferrites at $T = 298$ K

The temperature dependence of specific conductivity for nickel-cobalt ferrites is investigated in work [2]. It was found that the clearly pronounced fracture of dependence $\ln \sigma_{dc}(10^3/T)$ in the range of temperatures 425-445 K is inherent by all substituted of Ni^{2+} ions ferrites. This is due to the change in the conduction mechanism.

W. Chen [14] has observed that in cobalt ferrites the transport properties differ considerably from those of normal semiconductors, as the charge carriers are not free to move through the crystal lattice but jump from one ion to the other ion. It was further observed that in this type of materials the possibility of a change in the valency of a considerable fraction of metal ions and especially in that of Fe ions. In the jump mechanism of conductivity, the activation energy is associated with change in the mobility of carriers, and not with their formation. Thus the temperature dependence of conductivity arises only due to increase of mobility and not due to the number of charge carriers in the $CoFe_2O_4$ sample. The linear dependence of $\ln \sigma_{dc}(10^3/T)$ is by confirmation of this result, which we found also in the work [15].

It is known that in semiconductor materials Hall mobility is within the limits from 10^{-5} $cm^2/V \cdot s$ to 10^5 $cm^2/V \cdot s$. In oxides of transition elements the Hall mobility is small and is in range 10^{-5} $cm^2/V \cdot s$ – 10^{-1} $cm^2/V \cdot s$ [16]. Low mobility values are characteristic from the mechanism of conductivity of these substances [17].

To determine the mobility of current carriers, we write the expression for the semiconductor specific

electrical conductivity σ[18]:

$$\sigma = en\mu_H, \tag{12}$$

where μ_H – Holl mobility of charge carriers, which is determined by the average ratio of the speed of the charge ordered movement to the intensity of the electric field, which caused this movement.

Using the expression (12), we obtain:

$$\mu_H = 0{,}85 \cdot \sigma R_H. \tag{13}$$

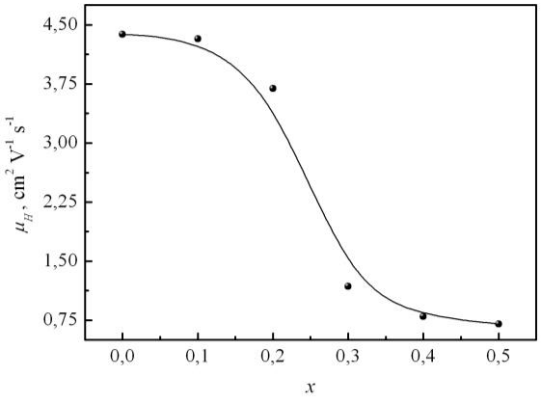

Fig. 6 – The dependence of Holl mobility from content of Ni^{2+} ions

Fig. 6 shows the composition dependence of Holl mobility. The sharp decrease in Hall mobility with Ni addition can be attributed to the increase in the carrier concentration.

4. CONCLUSION

The $Ni_xCo_{1-x}Fe_2O_4$ ferrites were synthesized by the technology sol-gel with participation of auto-combustion at low temperature. The optical micrograph shows the chemical homogeneities and porosity along with grains formation. The nickel addition increase had major effect on the surface morphology of the ferrites. The addition of Ni ions suppressed the grain growth and causes the grain size to decrease from 1,4 μm to 0,5 μm.

The micro-hardness of all the samples was consistent from top to bottom of the ferrite matrix. The hardness values obtained were comparable with those reported in the literature and found better results due to using of sol-gel auto-combustion method for synthesis of the Ni-Co composition, which suppressed the ceramic grain growth and enhanced the micro-structural and hardness properties. Microstructure and microhardness properties were correlated. Porosity decreases and hardness value increases with increasing Ni concentration of Ni-Co ferrite systems

The Hall coefficient demonstrates that for samples with $x < 0.4$ the majority of charge carriers of n-type, suggesting that the mechanism of conduction is predominantly caused by hopping of electrons between Fe^{2+} and Fe^{3+} ions. Hopping of holes between Ni^{+2} and Ni^{+3} ions is the dominant one for $x \geq 0.4$, which indicates the advantage of the conduction *p*-type in these ferrites. The value of *dc*-conductivity decreases when increases in Ni contents up to $x = 0.2$, after which it begins to increase. In the $CoFe_2O_4$ sample Hall mobility is the highest and is equal 4.38 cm²/V·s.

Механічні та електричні властивості феритів $Ni_xCo_{1-x}Fe_2O_4$

В.С. Бушкова, І.П. Яремій, Р.В. Ільницький, Б.С. Дзундза, О.М. Матківський

ДВНЗ «Прикарпатський національний університет імені Василя Стефаника», вул. Шевченка, 57, 76025 Івано-Франківськ, Україна

Ni-Co ферити одержано за технологією золь-гель за участі автогоріння та відпалено за температури 1573 К в атмосфері повітря. Вивчено вплив додавання йонів Ni^{2+} на мікроструктуру та електричні властивості феритів $Ni_xCo_{1-x}Fe_2O_4$. Присутність йонів Ni^{2+} значно впливає на формування пор та розмір зерен феритів. За допомогою легування йонами нікелю пористість зменшується і утворюється щільний матеріал. Значення мікротвердості збільшується зі збільшенням вмісту нікелю з 5,16 ГПа до 8,59 ГПа. Встановлено коефіцієнт Холла, тип провідності, концентрацію носіїв заряду та питому провідність. У Ni-Co феритах холлівська рухливість знаходиться в межах від $7.04 \cdot 10^{-1}$ см²/В·с до 4.38 см²/В·с.

Ключові слова: Ферит, Мікроструктура, Мікротвердість, Ефект Холла, Електрична провідність.

Механические и электрические свойства ферритов $Ni_xCo_{1-x}Fe_2O_4$

В.С. Бушкова, И.П. Яремий, Р.В. Ильницкий, Б.С. Дзундза, О.Н. Маткивский

ГВНЗ «Прикарпатский национальный университет имени Василия Стефаника», ул. Шевченко, 57, 76025 Ивано-Франковск, Украина

Ni-Co ферриты получено по технологии золь-гель с участием автогорения и отожженно при температуре 1573 К в атмосфере воздуха. Изучено эффекты добавления ионов Ni^{2+} на микроструктуру и электрические свойства ферритов $Ni_xCo_{1-x}Fe_2O_4$. Присутствие ионов Ni^{2+} существенно влияет на образование пор и размер зерен ферритов. При легировании ионами никеля пористость уменьшается и получается плотный материал. Значение микротвердости увеличивается с увеличением содержания

никеля с 5,16 ГПа до 8,59 ГПа. Установлено коэффициент Холла, тип проводимости, концентрацию носителей заряда и удельную проводимость. В Ni-Co ферритах холловская подвижность находится в пределах от $7.04 \cdot 10^{-1}$ см²/В·с до 4.38 см²/В·с.

Ключевые слова: Феррит, Микроструктура, Микротвердость, Эффект Холла, Электрическая проводимость.

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