

# Materials Research Express



## PAPER

# Magnetic and resonant properties of ferrite compositions $(1 - x)$ $\text{MeFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$ prepared by plasma spraying

RECEIVED  
25 January 2017

REVISED  
29 March 2017

ACCEPTED FOR PUBLICATION  
11 April 2017

PUBLISHED  
3 May 2017

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**Keywords:** ferrites, plasma spraying, magnetic properties

## Abstract

It was found that after plasma spraying and rapid quenching of ferrite compositions  $(1 - x)$   $\text{MeFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$  within the range of  $0.35 < x < 0.4$  for any considered compounds, transition to the x-ray-amorphous state is observed (so called concentration depended transition (CDT)). This transition occurs along with modification of x-ray and Mössbauer spectra (quadrupole splitting into isomer shift) and changing their magnetic parameters: a saturation magnetization  $\sigma_s$ , a coercive field  $H_c$  and a line width of ferromagnetic resonance (FMR)  $\Delta H$ . The abnormal increase of saturation magnetization  $\sigma_s$  is revealed at  $T < 100$  K. Particularities of changing the Curie temperature  $T_c$  and a saturation magnetization  $\sigma_s$  at different rates of quenching ferrites from the liquid state are discussed.

## Highlights

- We studied the influence of the ferrite compositions inclusion on their magnetic state;
- We studied the behavior of the magnetic parameters ( $T_c$ ,  $\sigma_s$ ,  $H_c$ ) of spinel ferrites under different conditions of rapid quenching;
- We studied the  $\gamma$ -spectra of ferrite compositions;
- We studied the behavior of the magnetic parameters  $H_c$  and  $\Delta H$  at concentration transitions (CDT).

## 1. Introduction

Magnetic ordering and corresponding magnetic properties of ferrites are significantly determined by their structural condition and the distribution of cations on sublattice points. Therefore, searching new ferromagnetic materials has been mainly carried out for the purpose of creating multi-component compounds with complex chemical composition [1–4]. This approach is aimed at the possibility of selective injection of various ions into the sublattice, with the consequent changes of the character and the ratio of intra- and inter-sublattice exchange interactions, and as a result, changes of the magnetic properties of these compounds. In this respect, the investigation was focused on defining of the composition—property regularities.

An alternative approach in the production of new materials appeared because of the possibility to influence the oxide compounds by concentrated energy flows (plasma, laser, etc). This enables rapid heating and subsequent ultra-fast quenching of the melt [5–11]. After corresponding treatment, a number of changes are observed in the structure and properties of the substances. They are as follows: decrease of the crystallite size, increase of material disorder, increase of solubility limit for the components in multiphase systems, formation of metastable crystalline phases, redistribution of cations at sublattice points, as well as partial or complete amorphization of the compound. Due to strong correlation between the structural state, the type of spin ordering and the set of magnetic characteristics of the oxides, determining the correlation Structure—Disorder state—Property would provide direct control of the ferrite properties.

Compared to metal alloys, ferrites with spinel structure are prone to amorphization significantly less and are prone to thermal conductivity much less (by several orders). Therefore the production of iron oxides in the x-ray amorphous state is quite difficult because of achieving rather high rate of quenching the melt.

Widely known amorphizing additions such as  $B_2O_3$ ,  $SiO_2$ ,  $P_2O_5$  and  $Bi_2O_3$  can be used in some cases to facilitate the amorphization process [3]. The additives significantly reduce the critical rate of ferrite compositions quenching, and in most cases are not included in the ferrite composition.

The abovementioned approaches were used to achieve non-equilibrium state (up to amorphization) of ferrite compositions  $(1 - x)MeFe_2O_4 \cdot xP_2O_5$  during plasma spraying [11, 12].

This paper shows the results of new studies of the magnetic state, resonance and magnetic properties of ferrite compositions based on spinel ferrites.

## 2. Materials and methods

The initial crystalline spinel ferrite powders  $MeFe_2O_4$  (where Me is Fe, Ni, Co, etc) were synthesized according to the traditional ceramic technology. The final sintering of ferrites was carried out in the opened air at the temperature of  $T = 1400$  K during 4 h followed by slow cooling.

The structural disordering of the prepared samples was carried out by means of:

- plasma spraying and subsequent rapid quenching of melt droplets in water and copper disc;
- diluting the initial ferrite with amorphizing addition of  $P_2O_5$ .

Ferrite compositions  $(1 - x)MeFe_2O_4 \cdot xP_2O_5$  were prepared from the initial ferrites by adding a required amount of phosphorus pentoxide and sintering the mixture at the temperature  $T = 1100$  K.

The plasma spraying of ferrite composite powders with a dispersion of  $50 < d < 80 \mu m$  was carried out in the plasma of (Ar +  $CO_2$ ) with the Ar/ $CO_2$  ratio by volume equal to 1/3, at the arc discharge power, not exceeding 10 kW. Spraying was carried out by plasmatron with coaxial powder feed into the cathode zone [14]. The melt drops were quenched on the polished external surface of the fast-rotating copper disk ( $\sim 12 \cdot 10^3$  rpm), placed in the reaction chamber. In order to avoid thermal exposure of the disk, the process nozzle was put on the plasmatron. In some cases, in order to avoid the dependence of material properties on the conditions of its preparing, the spraying process parameters remained constant.

The  $\gamma$ -resonance measurements were performed by the spectrometer MC 1104E with source of  $Co_{57}$  in a chrome matrix. The spectra were recorded at room temperature ( $T = 22$  °C) in a constant acceleration mode. The test samples were placed into containers made of a thin aluminum foil.

The resonant magnetic properties were investigated by means of the FMR spectrometer at 9.2 Hz frequency and the magnetic fields reaching 20 kOe. The magnetic measurements were performed by the magnetometer with a vibrating sample in the range of magnetic field of  $-25 \div 25$  kOe.

The phase composition of the obtained samples was performed using Advance D8 x-ray diffractometer in  $CoK\alpha$  monochromatic radiation.

## 3. Results and discussion

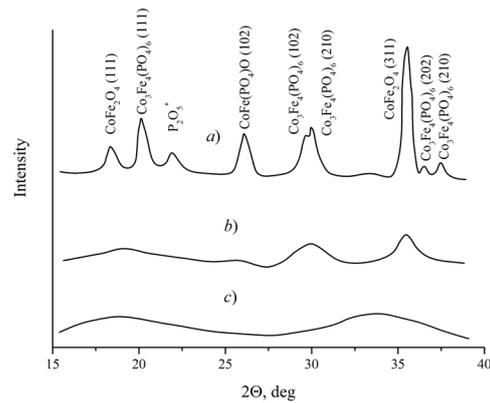
X-ray diffraction pattern of ferrite compositions has significantly changed after plasma spraying and quenching on the disk. Broadening and disappearance of sharp diffraction lines, characteristic of long-crystalline order, and emergence of clearly defined diffuse scattering, characteristic of the amorphous state, were observed.

Figure 1 shows an example of such a change in the form of an x-ray image of quenched powder compositions based on cobalt ferrite  $(1 - x)CoFe_2O_4 \cdot xP_2O_5$ . The diffraction pattern of the samples with a low content of  $P_2O_5$  (about 10–35%) appears as a group of broadened reflexes corresponding to a spinel structure. Since phosphorus-containing compounds are the first to become amorphous while quenching, samples are likely to have amorphocrystalline condition. The x-ray spectra of the ferrite compositions with  $P_2O_5$  content exceeding 35%, do not exhibit Bragg reflections, which indicates the amorphous state of the samples.

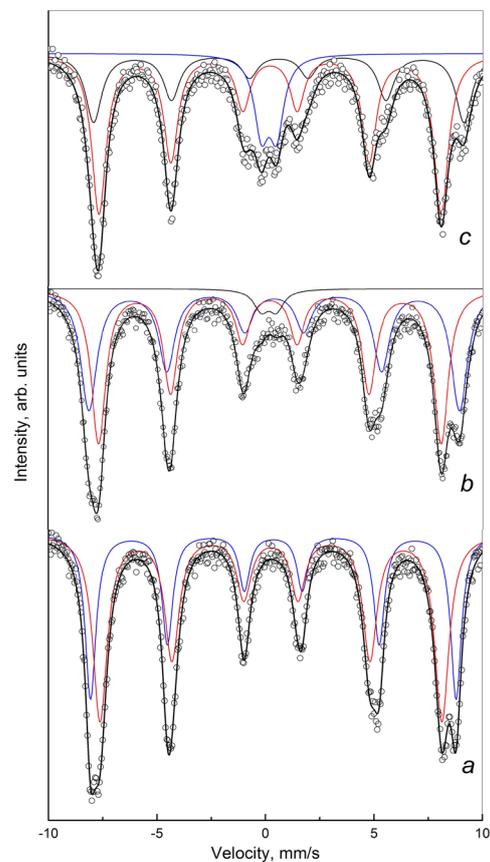
Under the fixed quenching conditions, the minimum concentration of  $P_2O_5$  at which a transition in the amorphous state is observed, reflects propensity of ferrite compositions to become amorphous. Ferrite compositions from the studied composition can be arranged in order of increasing propensity to amorphisation:  $Fe_3O_4 < CoFe_2O_4 < Mn-Zn < Ni-Zn < NiFe_2O_4$ .

Let's discuss the results of the Mossbauer studies of ferrite powders quenched from a liquid state, showing change of their magnetic state [4]. Figure 2 shows Mossbauer spectra of nickel ferrite samples ( $x = 0$ ) in the initial state and after plasma spraying into water and on the disk surface.

Nickel ferrite has a structure of completely inverted spinel  $Fe^{3+}[Ni^{2+}Fe^{5+}]O_4$ . Nickel ions occupy octahedral positions and iron ions are distributed equally on the octahedral and tetrahedral positions. The lines of  $\gamma$ -spectrum are differentiated, that allow defining the state of the  $Fe^{3+}$  cations in octahedral and tetrahedral positions. The external lines correspond to the positions of the iron ions in octahedral positions, and the internal lines refer to the tetrahedral positions.



**Figure 1.** X-ray of the quenched ferrite compositions  $(1 - x)\text{CoFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$ . (a) Initial,  $x = 0.35$ ; (b) quenched,  $x = 0.35$ ; (c) quenched,  $x = 0.45$ .

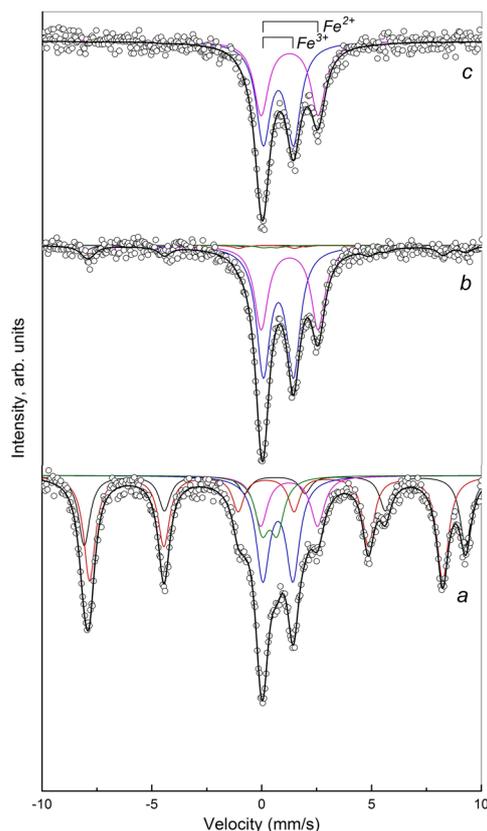


**Figure 2.** Mössbauer spectra of nickel ferrite: (a) ferrite at the initial stage; (b) ferrite after quenching on water; (c) ferrite after quenching on the disk.

The Mössbauer spectra of nickel ferrite in the initial state is characterized by the symmetry of lateral lines, which indicates a uniform distribution of  $\text{Fe}^{3+}$  cations on the octahedral and tetrahedral positions (figure 2(a)). Plasma spraying and quenching of ferrite results in some changes of the spectra, while the lines broadening is observed, and appearance of the doublet in the central part and the asymmetric distribution of the lateral lines is viewed. The degree of these changes increases while the quenching rate is increased (figures 2(b) and (c)).

The observed asymmetry of lateral lines proves the change in the distribution of  $\text{Fe}^{3+}$  ions on the sublattice points, therefore the relative amount of the cations in octahedral positions decreases. Probably the process goes with redistribution of  $\text{Ni}^{2+}$  ions in tetrahedral positions. The doublet in the central part of the spectrum seems to appear due to the formation of amorphous phase in the ferrite.

This indicates to the presence of iron ions both with the increased magnetic field compared to the initial one, and with hyperfine zero magnetic field. This case can occur because of a non-equilibrium distribution of cations, characterized by two states of iron ions: magnetically ordered and disordered, leading to both increasing and decreasing an exchange interaction degree.



**Figure 3.** Mössbauer spectra of nickel composition  $(1 - x)\text{NiFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$  quenched on copper disk: (a)  $x = 0.2$ ; (b)  $x = 0.35$ ; (c)  $x = 0.4$ .

We should also note that the amorphous state throughout the whole volume was not reached even at high quenching rates (on a copper disk) of molten droplets of ferrite spinels. This is also confirmed by electron beam diffuse scattering, characteristic to the amorphous state, which was observed only at the edges of the thinnest parts ( $\sim 100$  nm) of the flakes.

Let's consider the behavior of the  $\gamma$ -spectra of ferrite composition  $(1 - x)\text{NiFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$ , containing various percentage of amorphizing addition  $\text{P}_2\text{O}_5$ . Mössbauer spectra of quenched nickel composition at low concentrations of  $\text{P}_2\text{O}_5$  ( $x = 0.2$ ) represents a superposition of the magnetic hyperfine structure, characteristic of a magnetically ordered state, and situated in the central part of the spectrum of the asymmetric doublet (figure 3). It should be noted, that the observed hyperfine structure is characterized by the same features (lateral lines and their asymmetry) as the original nickel ferrite.

Increasing the content of phosphorus pentoxide in the sample causes the broadening and decrease in the intensity of the lines, which are characteristic for hyperfine structure of the Mossbauer spectrum, until they completely disappear at the concentration depended transition (CDT) at  $x = 0.35$ . Simultaneously, an asymmetric triplet is formed in the central part of the spectrum. As a result, the spectra of the samples, being in the amorphous state ( $x = 0.4$ ), represents the intensive triplet while a magnetically ordered phase is not indicated at all.

The appearance of the doublet in the central part of the Mossbauer spectrum is probably due to the formation of an amorphous phase, and the presence of some ions of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  in it. Since ferrites include at least two times more ferrous irons in comparison with the ferric ions, then the doublet, corresponding to  $\text{Fe}^{2+}$  ions, does not appear in the initial stages of amorphization. Increasing the percentage of the amorphous phase leads to the superposition of two doublets corresponding to  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  ions with different degree of the isomer shift and quadrupole splitting, and to formation of an intense triplet.

Corresponding changes of Mössbauer spectra are observed for other ferrite compositions. Table 1 shows the parameters of isomer shift, quadrupole splitting and the ratio of their areas corresponding to the ions of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  in the amorphous state. It is obvious, that the amorphous compositions, in spite of the differences in the initial ferrites, are characterized by similar values of the Mössbauer parameters. This indicates that amorphized ferrite compositions have very similar magnetic states.

Let's analyze the magnetic properties of spinel ferrites in correlation with the quenching conditions after plasma spraying. Table 2 shows that the magnetic parameters ( $\sigma_s$ ,  $T_c$ ,  $H_c$ ) mostly change during spraying ferrites on a rotating disk. In this case (while spraying the substance on the disk), the highest rate of quenching is reached, and the highest structural disorder of ferrites is observed.

**Table 1.** Basic parameters of the Mössbauer spectra of ferrite compositions  $0.6\text{MeFe}_2\text{O}_4 \cdot 0.4\text{P}_2\text{O}_5$  at  $T = 300\text{ K}$ .

Me	Isomer shift, $\text{mm s}^{-1}$		Quadrupole splitting, $\text{mm s}^{-1}$		Ratio $\text{Fe}^{3+}/\text{Fe}^{2+}$
	$\text{Fe}^{3+}$	$\text{Fe}^{2+}$	$\text{Fe}^{3+}$	$\text{Fe}^{2+}$	
Fe	0.65	1.27	0.95	2.34	1.18
Co	0.71	1.29	0.99	2.4	1.15
Ni	0.67	1.24	0.97	2.33	1.06
Mn–Zn	0.7	1.28	0.98	2.37	1.22
Ni–Zn	0.66	1.22	1.00	2.41	1.19

**Table 2.** Magnetic properties of ferrite powders after plasma treatment and quenching.

Ferrite	Cooling conditions	$\sigma_s$ , $\text{emu g}^{-1}$	$H_c$ , Oe	$T_c$ , K
$\text{Fe}_3\text{O}_4$	<i>a</i>	82.3	43	858
	<i>b</i>	73.5	96	855
	<i>c</i>	52.2	185	808
$\text{NiFe}_2\text{O}_4$	<i>a</i>	43.5	25	863
	<i>b</i>	39.0	75	859
	<i>c</i>	28.8	140	833
$\text{CoFe}_2\text{O}_4$	<i>a</i>	71.6	315	793
	<i>b</i>	63.2	354	793
	<i>c</i>	47.9	578	768
$\text{MnFe}_2\text{O}_4$	<i>a</i>	74.5	8	783
	<i>b</i>	64.3	27	780
	<i>c</i>	49.2	94	770
Ni–Zn ferrite (600 HH)	<i>a</i>	61.8	2	413
	<i>b</i>	50.4	24	453
	<i>c</i>	41.1	69	513
Mn–Zn ferrite	<i>a</i>	62.5	4	400
	<i>b</i>	58	23	426
	<i>c</i>	46	51	510

Note: Quenching: (a) in inert gas; (b) in liquid nitrogen; (c) on a rotating disk. Values  $\sigma_s$  and  $H_c$  are measured at the temperature of 300 K.

One of the particularities of the investigated ferrites ( $x = 0$ ), regardless of their chemical composition, was a sharp decrease in the saturation magnetization  $\sigma_s$  following a rapid quenching process (figure 2). Compared with the bulk polycrystalline ferrites, the range of  $\sigma_s$  change was more than 30%. It is hardly possible to explain the observed significant change of the resulting magnetization  $\sigma_s$  by non-equilibrium distribution of cations on sublattices. Evidently, rapid quenching of the melt results in the deformation of backbone oxygen atoms simultaneously with the redistribution of cations, which leads to changing the crystal fields symmetry, the value and the type of the exchange bonds under study.

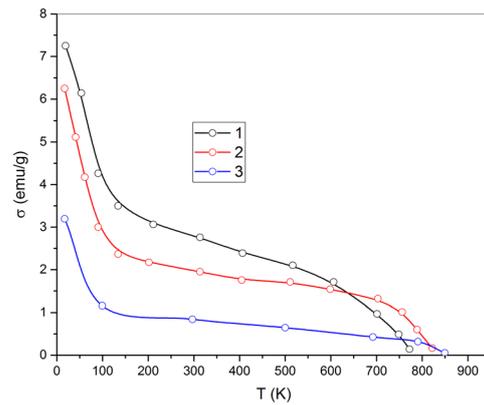
Let's pay attention to the behavior of Curie temperature for the quenched ferrites of different composition. While  $\text{MeFe}_2\text{O}_4$  compounds (where Me is a Fe, Ni, Co or Mn atoms) show decrease of  $T_c$  with increasing the quenching rate, then Ni–Zn and Mn–Zn ferrites are prone to greatly increase the Curie temperature.

The Curie temperature can be represented in the following generalized view according to the molecular field theory [15]:

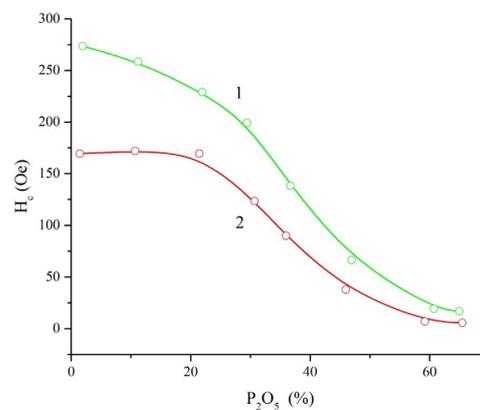
$$T_c = -\frac{2}{3}S(S+1)Z_{BA}J_{AB}^{\text{eff}}/K, \quad (1)$$

where  $J_{AB}^{\text{eff}}$  is an effective exchange integral measurable in kelvin,  $Z_{BA}$  is a number of the nearest to A neighbors of  $\text{Fe}^{3+}$  ion.

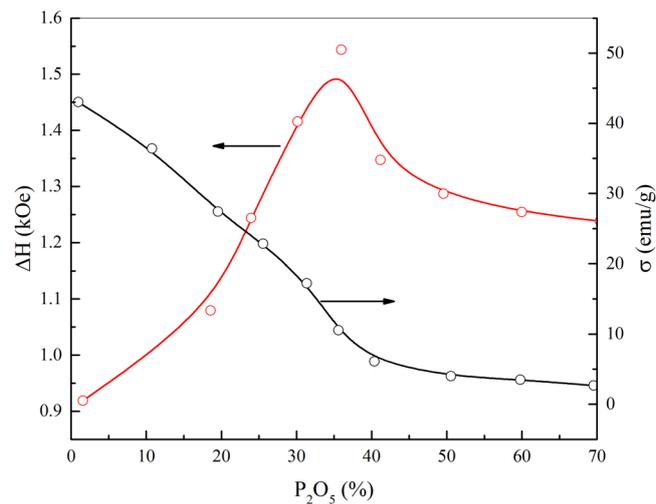
It is known [12–14] that fast quenching of ferrites causes a formation of the amorphous–crystalline state with disordered micro-zones. The magnetic state of corresponding micro-zones considerably depends on their size. So, decrease of the disordered micro-zones causes increase of the percentage of magnetically active ions, located near the border and having less number of exchange couplings. These changes probably imply the Curie temperature decrease and the specific magnetization reduction.



**Figure 4.** The temperature dependence of magnetization for ferrite compositions  $0.6\text{MeFe}_2\text{O}_4 \cdot 0.4\text{P}_2\text{O}_5$ ; (1) Me–Mn; (2) Me–Fe; (3) Me–Ni.



**Figure 5.** Concentration dependence of the coercive force: (1) samples at the initial stage; (2) samples after annealing at  $700^\circ\text{C}$ .



**Figure 6.** FMR line width and the saturation magnetization of the nickel compositions.

The situation changes significantly when the initial ferrite contains diamagnetic ions in tetrahedral positions. In this case, ferrites quenching causes cations redistribution, and a certain amount of  $\text{Fe}^{3+}$  ions transfer into these tetrahedral positions. While the value of the exchange integral  $J_{AB}$  is considerably determined by strong  $A$ - $B$  interaction of magnetically active  $\text{Fe}^{3+}$  ions and the number of interlattice exchange couplings of  $\text{Fe}^{3+}$ - $\text{O}$ - $\text{Fe}^{3+}$  increases, as a result, the increases of Curie temperature is observed.

As it could be expected, dilution of ferrites with phosphorous pentoxide resulted in further decrease of saturation magnetization  $\sigma_s$ . The specific features of temperature behavior of  $\sigma_s$  at the temperatures of  $T = 100\text{ K}$  and the Curie temperature of the amorphous samples  $T_c$  remained approximately the same as that of the initial stage

ferrites. The abrupt increase in the magnetization at low temperatures should be referred to the magnetic properties changes for ferrite compositions. The abrupt changes of  $\sigma_s$  were observed in the temperature range of 4.2–80 K, and their intensity depended on the composition of the samples (figure 4).

The results of investigating the concentration dependence of a saturation magnetization  $\sigma_s$ , a coercive force  $H_c$  and a width of ferromagnetic resonance (FMR) line  $\Delta H$  are shown in figures 5 and 6. It is easy to notice, that  $H_c$  and  $\Delta H$  correlate most strongly with the change in the structural state (the CDT at  $x = 0.35$ ).

The coercive force of nickel composition is reduced by nearly one order while the substance becomes x-ray-amorphous. In the concentration range of  $x < 0.35$  the quenched samples possess disordered amorphous crystal microstructure and a high coercive force ( $H_c \sim 250$  Oe). In the x-ray-amorphous state at  $x > 0.35$  the value of  $H_c$  is reduced down to 5 Oe, what indicates the presence of more homogeneous material structure. After annealing the sample at 973 K, the coercive force is reduced by approximately two times (at  $x < 0.35$ ), but it remains sensitive to the CDT.

The measurement of the FMR line width  $\Delta H$  was carried out on flakes with thickness of one micron order. As this value is significantly less than the depth of the skin layer, it can be assumed that the total volume of the sample undergo the resonance conditions. At the range of low concentrations of  $P_2O_5$ , the width of FMR line was approximately equal to 0.9 kOe and correlates with  $\Delta H$  value of the quenched nickel ferrite. While increasing the phosphorus pentoxide concentration, the width of FMR line is increased and reached its maximum at  $x = 0.35$ , and then decreased to the value of  $H = 1.24$  kOe, when  $x = 0.6$  (figure 6).

As the width of the line is significantly determined by the local anisotropy of the material, the observed dependence of  $\Delta H$  in the value interval of the CDT towards the amorphous state is consequently explained. Indeed, the decrease in  $P_2O_5$  percentage in the samples should cause the appearance and growth of crystal nuclei. Naturally, it causes various types of defects, inhomogeneities and other stresses, that finally determine the loss, and as a result, the increase of the FMR line width in the CDT interval.

It should be noted that the FMR line width in the amorphous state is more than that of the crystal one. Although it could seem to the viewer, that on the contrary, it should be reduced, while amorphous materials are isotropic. Most probably, the reason for this disagreement is the following: in spite of the integrated isotropic characteristics, amorphous materials possess a sufficiently large local anisotropy, which influences the FMR line width.

It has been shown that the saturation magnetization  $\sigma_s$  monotonically decreases while the percentage of  $P_2O_5$  is increased in nickel composition. Therefore the saturation magnetization does not significantly change in the range of critical concentrations  $x = 0.35$ – $0.37$  (figure 6).

## 4. Conclusion

Structural features, magnetic and resonant properties of rapidly quenched ferrite compositions  $(1 - x)\text{MeFe}_2\text{O}_4 \cdot x\text{P}_2\text{O}_5$  (where Me is Fe, Co, Ni, Zn, Mn) were studied. It was shown that after plasma spraying and fast quenching of ferrites ( $x = 0$ ) a value of saturation magnetization  $\sigma_s$  considerably changes ( $>30\%$ ). This effect can hardly be explained only by redistribution of Fe cations on sublattices. Dilution of ferrites with phosphorous pentoxide  $P_2O_5$  resulted in further decrease of saturation magnetization  $\sigma_s$ . It was found that after plasma spraying and rapid quenching in the range of  $0.35 < x < 0.4$  a transition to the x-ray-amorphous state was observed for all the studied compositions. The CDT is accompanied by modification of x-ray and Mössbauer (quadrupole splitting and isomeric shift) spectra and by the change in magnetic parameters: a coercive field value and a FMR line width. Configuration, size and number of exchange couplings changed because of the structural disorder. This leads to modification of spin ordering and changes in the fundamental magnetic characteristics of ferrites ( $\sigma_s, T_c, H_c, \Delta H$ ).

## Acknowledgments

The reported study was funded by Russian Foundation for Basic Research, Government of Krasnoyarsk Territory, Krasnoyarsk Region Science and Technology Support Fund to the research projects No. 16-48-242101/16 and No. 03/16.

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