



Magnetic Ordering of Perovskite-Like La-, Nd-, and Gd-Doped Bismuth Ferrite

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ABSTRACT

Bismuth ferrite (BiFeO_3) and La-, Nd- and Gd-substituted bismuth ferrite of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ types with the atomic part of the substitution element x equal up to 0.20 were synthesized by the solid-state reaction method using powders of oxides Bi_2O_3 , Fe_2O_3 , and La_2O_3 , or Nd_2O_3 , or Gd_2O_3 of pure grade quality and investigated using X-ray diffraction analysis. The magnetization was measured in the magnetic field up to $6.5 \cdot 10^6$ A/m at 5 and 300 K. It was found that the total substitution up to 0.20 atomic part of Bi by La, Nd, and Gd leads to the paramagnetic behavior of the doped bismuth ferrite at low temperatures in a wide range of magnetic field. Strong nonlinear dependence of magnetization on the magnetic field was detected and a ferromagnetic-like dependence of magnetization was observed for small magnetic fields. This can be explained by the exchange interaction between doping magnetic ions, as well as by the exchange interaction of these ions with ions of iron. The enhancement of magnetic properties with the increase of the content of the substitution is monotone and is more pronounced for the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics.

INTRODUCTION

Bismuth ferrite (BiFeO_3) is a promising magnetoelectric material due to coexistence of both ferroelectric and antiferromagnetic orders in this material at room temperature [1]. It is desirable to transform the antiferromagnetic spin configuration of BiFeO_3 into the ferromagnetic one by an action on the spiral modulated spin structure. Chemical doping can modify both the electric conductivity and the spiral spin structure to intensify the macroscopic properties. In particular, the use of rare-earth elements Nd and Gd for the substitution of Bi was proposed [2, 3]. The effect of substitution of Bi by Gd on structural, magnetic, and dielectric properties was studied in [4, 5]. There was evidence of weak magnetization in the Gd-containing samples, and this weak magnetization increases with the Gd content for $x \leq 0.25$, and then decreases gradually with increasing x for $x \geq 0.30$ [4]. The preparation and physical properties of substituted multiferroics were discussed in detail [6]. Polycrystalline samples of $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$ ($x = 0, 0.175, 0.20$) were prepared by chemical co-precipitation method and the effect of doping in the BiFeO_3 compound on its structural and ferroelectric properties was studied [7]. The crystal structure and magnetic properties of the Nd-doped BiFeO_3 materials were investigated by X-ray diffraction, Raman scattering, vibrating sample magnetometer, and complex impedance spectroscopy [8]. Samples exhibited weak ferromagnetism at room temperature, and the magnetization was enhanced by the presence of Nd^{3+} ions. Electron spin resonance and magnetization versus magnetic field for $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ samples were investigated [9], and the improved magnetic behavior indicated weak ferromagnetism in the samples with remnant magnetization ($2 M_r$) of 0.145 emu/g for $x=0.15$. The magnetic susceptibility, thermal expansion, electrical conductivity, and thermo-EMF of $\text{Bi}_{0.85}\text{Nd}_{0.15}\text{FeO}_3$ were studied [10], and the value of Neel temperature (T_N) for $\text{Bi}_{0.85}\text{Nd}_{0.15}\text{FeO}_3$ solid solution was 624 K, which is close to the Neel temperature of BiFeO_3 (640 K). Ferroelectric and ferromagnetic loops were observed in the $\text{Bi}_{0.80}\text{Nd}_{0.20}\text{Fe}_3$ ceramics, indicating that ferroelectric and ferromagnetic ordering coexist in the ceramics at room temperature [11].

The main goal of this paper is to prepare the substituted ceramics based on BiFeO_3 of the types $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ with the atomic part of the substitution element x equal up to 0.20 and to determine the influence of the bismuth substitution by rare earth elements La, Nd, and Gd on magnetic ordering in bismuth ferrite at 5 and 300 K. Both magnetic parameters—a double remnant magnetization $2 M_r$ and a double coercive force $2 H_c$ —will be determined and ceramics with improved magnetic properties will be found. The magnetization for the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ at 5, 10, 20, 50, 100, and 300 K will be calculated and compared with the experimental results in the magnetic field up to $5.0 \cdot 10^6$ A/m.

EXPERIMENTAL DETAILS

The substituted ceramics of BiFeO_3 of the types $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ with the atomic part of the substitution element x equal up to 0.20 were synthesized by the solid-state reaction method using powders of oxides Bi_2O_3 , Fe_2O_3 , La_2O_3 , or Nd_2O_3 , or Gd_2O_3 of pure grade quality. The X-ray diffraction method was applied using the diffractometer Dron-3 on monochromatic Cu K_α -radiation (1.5406 Å). The experimental data were collected during scanning repeated ten times in the 2Θ range from 20° to 80° at the scanning rate of $10^\circ/6$ min. The magnetization was measured using an automated vibrating sample magnetometer (VSM, Oxford instruments) in magnetic fields up to $6.5 \cdot 10^6$ A/m. Temperature magnetic measurements were carried out using

Physical Property Measurement System equipped with a 9 T superconducting magnet (PPMS: Model 6000, Quantum design).

DISCUSSION

1. X-ray powder diffraction

It was found that the crystal structure for the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics is the rhombohedrally distorted structure of perovskite, $R3c$ space group. There is a shift of X-ray reflexes that clearly shows the formation of solid solutions in the $\text{BiFeO}_3 - \text{LaFeO}_3$, and $\text{BiFeO}_3 - \text{NdFeO}_3$, and $\text{BiFeO}_3 - \text{GdFeO}_3$ systems. A more detailed description of the XRD studies was provided by us earlier [12].

Additional reflexes are detected for ceramics of all series and the amount of the impurity phases is larger for the Bi (Gd) - Fe - O system and can reach 10 molar %. These reflexes can be attributed to the phases which are based on ternary compounds $\text{Bi}_{25}\text{FeO}_{39}$ and $\text{Bi}_2\text{Fe}_4\text{O}_9$ formed in the $\text{Bi}_2\text{O}_3 - \text{Fe}_2\text{O}_3$ quasibinary system for the molar ratio of Bi_2O_3 and Fe_2O_3 being 25:1 and 1:2, respectively. BiFeO_3 exists in the $\text{Bi}_2\text{O}_3 - \text{Fe}_2\text{O}_3$ quasibinary system for the molar ratio of Bi_2O_3 and Fe_2O_3 being 1:1 [13, 14]. Its melting occurs in accordance with the peritectoid reaction. This means that this compound in principle cannot be prepared by preparation methods from the melt. Additionally, the high volatility of bismuth oxide Bi_2O_3 has to be taken into account. All of this leads to a thermodynamic instability of BiFeO_3 and the impossibility to synthesize single-phase BiFeO_3 by the solid-state reaction. Synthesis in accordance with the ceramic technology produces the pseudomonophasic composition with accuracy of up to 3% by weight of impurity of $\text{Bi}_{25}\text{FeO}_{39}$ [15].

It should be noted that the presence of impurity phases affects physical properties and partially changes quantitative values. Nevertheless, the impurity phases cannot be the cause of the observed phenomena since their content is relatively small to sufficiently affect physical properties of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics.

2. Magnetization

2.1. Magnetization of the BiFeO_3 ceramics

The dependencies of magnetization of the BiFeO_3 ceramics on magnetic field were measured under a magnetic field up to $6.0 \cdot 10^6$ A/m at 5, 30, 100, and 300 K (figure 1). The magnetization of pure BiFeO_3 shows a near linear relationship that points to the antiferromagnetic spin order. This experimental result can be explained by the existence of spin cycloid structure with long period wavelength of ~ 62 nm [16].

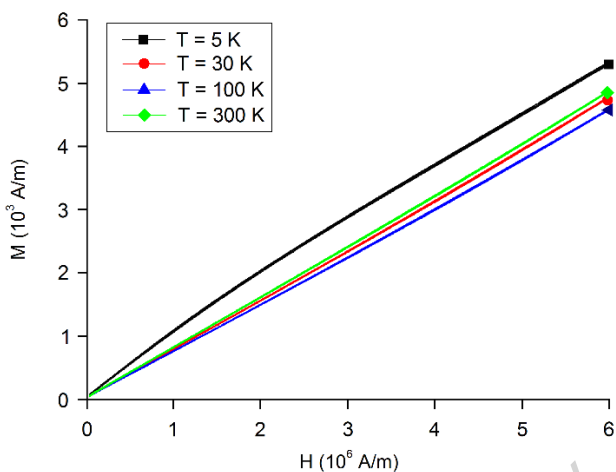


Figure 1. Field dependence of magnetization of the BiFeO_3 ceramics at 5, 30, 100, and 300 K.

2.2. Magnetization of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ ceramics

The dependencies of magnetization of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ ceramics on magnetic field (M - H plots) were measured under a magnetic field up to $6.5 \cdot 10^6$ A/m at room temperature (figure 2).

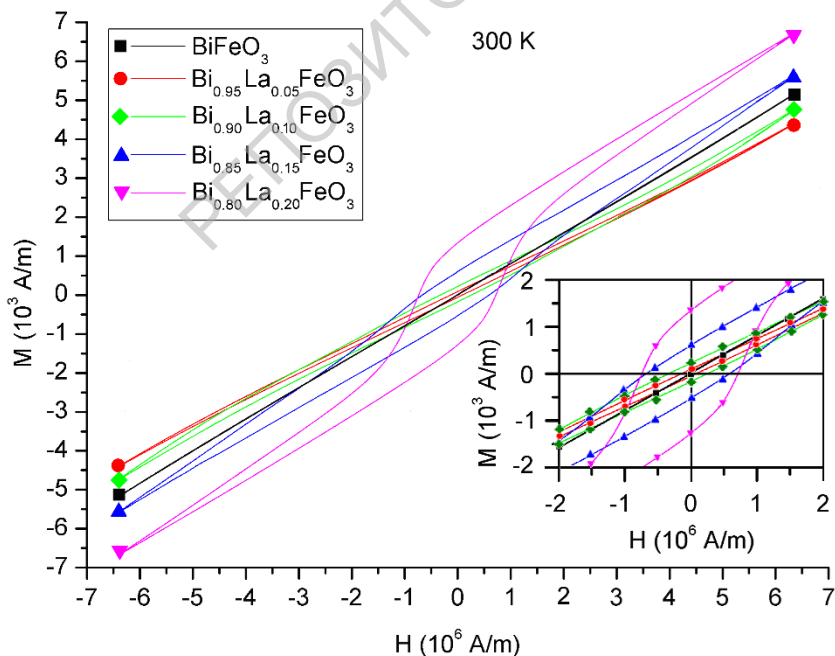


Figure 2. Field dependence of magnetization of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ ceramics with $x = 0, 0.05, 0.10, 0.15,$ and 0.20 at 300 K (Inset shows M - H plot with enlarged view near origin).

The magnetic hysteresis loops of ceramics were detected but no saturated magnetization was observed under the experimental conditions. The macroscopic double remnant magnetization ($2 M_r$) increases with the increase of content of La and is equal to $0.17 \cdot 10^3$ A/m, $0.41 \cdot 10^3$ A/m, $1.12 \cdot 10^3$ A/m, and $2.62 \cdot 10^3$ A/m for $x = 0.05, 0.10, 0.15,$ and 0.20 , respectively. This indicates weak ferromagnetism in the system. The double coercive field ($2 H_c$) increased monotonically and was $0.23 \cdot 10^6, 0.58 \cdot 10^6, 1.27 \cdot 10^6,$ and $1.47 \cdot 10^6$ A/m for $x = 0.05, 0.10, 0.15,$ and 0.20 , respectively. This coincides with the result of the paper [11] where the magnetization-magnetic field curve of $\text{Bi}_{0.80}\text{La}_{0.20}\text{FeO}_3$ showed a very narrow magnetic hysteresis loop, no saturation magnetization, with a small but nonzero remnant magnetization (M_r) of ~ 0.005 emu/g and coercive field of ~ 454.6 Oe. The monotone increase of magnetic parameters for the La-containing alloys was also detected in paper [17] for the samples of $\text{Bi}_{0.90}\text{La}_{0.10}\text{FeO}_3$ with the substitution of Fe by Gd. The double remnant magnetization ($2 M_r$) and coercive force ($2 H_c$) were 0.035 and 7,141, 0.109 and 9,293, and 0.184 emu/g and 14,283 Oe for $\text{Bi}_{0.90}\text{La}_{0.10}\text{Fe}_{0.90}\text{Gd}_{0.10}\text{O}_3,$ $\text{Bi}_{0.90}\text{La}_{0.10}\text{Fe}_{0.70}\text{Gd}_{0.30}\text{O}_3,$ and $\text{Bi}_{0.90}\text{La}_{0.10}\text{Fe}_{0.50}\text{Gd}_{0.50}\text{O}_3,$ respectively.

It can be concluded that the La-substitution of Bi leads to the destruction of the space-modulated cycloid structure in BiFeO_3 and gives a monotone enhancement in the magnetic properties upon La-substitution of Bi in the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ ceramics in the range of composition up to $x = 0.20$.

2.3. Magnetization of the $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$ ceramics

The dependencies of magnetization of the $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$ ceramics on magnetic field were measured under a magnetic field up to $6.5 \cdot 10^6$ A/m at 5 and 300 K (figure 3). S-shaped magnetization is observed for all samples which indicates a weak ferromagnetism order. The magnetic parameters, such as a double remnant magnetization ($2 M_r$) and a double coercive force ($2 H_c$), provided with hysteresis loops are listed in table I.

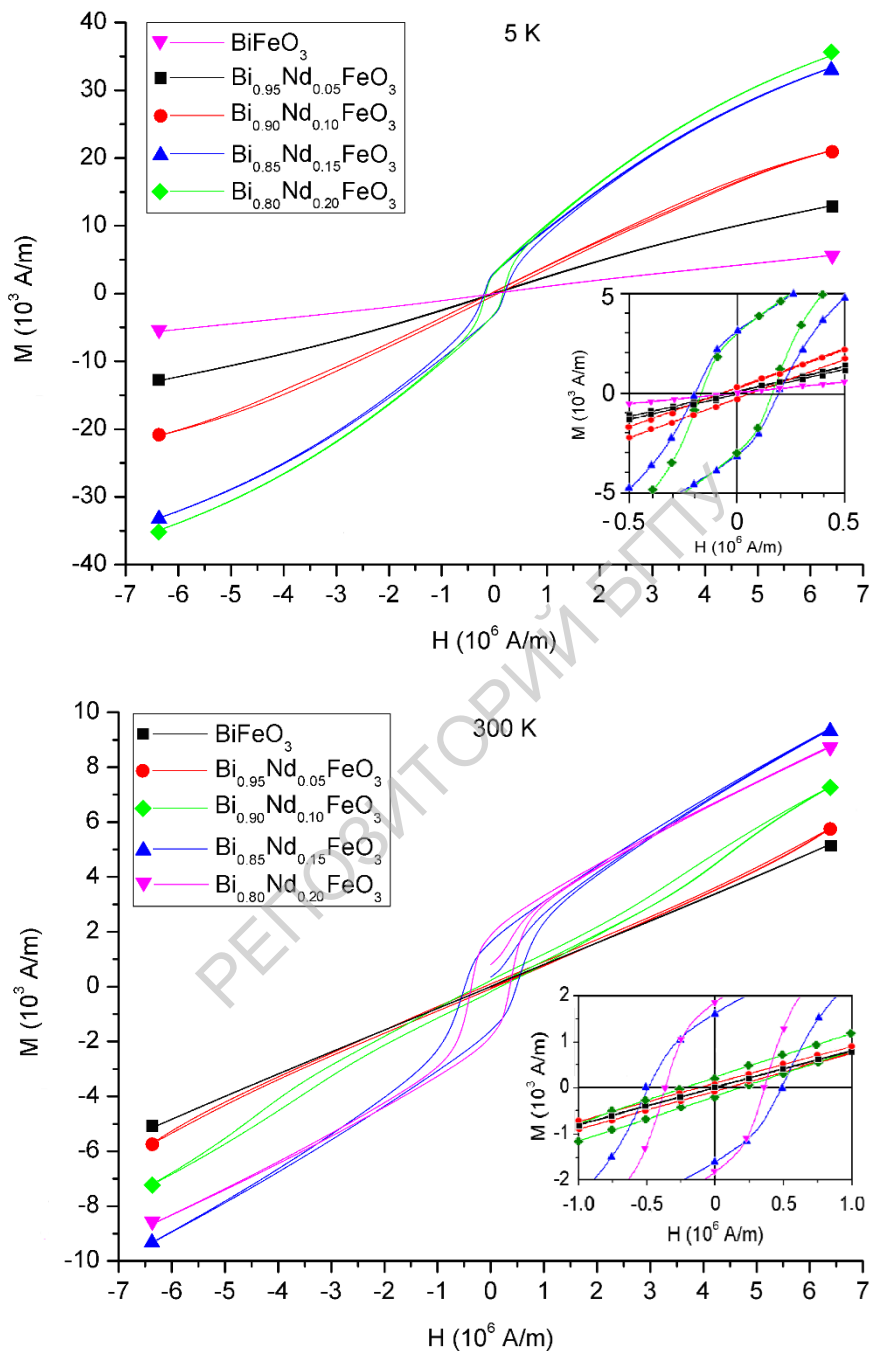


Figure 3. Field dependence of magnetization of the $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$ ceramics with $x = 0, 0.05, 0.10, 0.15,$ and 0.20 at 5 (top) and 300 K (bottom) (Insets show M - H plots with enlarged view near origin).

Both magnetic parameters—a double remnant magnetization $2M_r$, and a double coercive force $2H_c$ —increase as the concentration of Nd increases from $x = 0$ to $x =$

0.15. This is contradictory to the results of paper [8] where the magnetization decreases as the concentration of Nd increases from $x = 0$ to $x = 0.05$ but increases as the concentration of Nd increases from $x = 0.075$ to $x = 0.15$. The increase in macroscopic magnetization is explained by the formation of Fe^{2+} ions, by the suppression in inhomogeneous spin structure, and by the change in canting angle [18 – 20].

Table I. Magnetization- Magnetic Field hysteresis loop parameters of the $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$ ceramics at 5 and 300 K

Sample	$2 M_r, 10^3 \text{ A/m}$		$2 H_c, 10^6 \text{ A/m}$	
	5 K	300 K	5 K	300 K
$\text{Bi}_{0.95}\text{Nd}_{0.05}\text{FeO}_3$	0.18	0.22	0.02	0.18
$\text{Bi}_{0.90}\text{Nd}_{0.10}\text{FeO}_3$	0.62	0.44	0.12	0.40
$\text{Bi}_{0.85}\text{Nd}_{0.15}\text{FeO}_3$	6.22	3.19	0.39	0.96
$\text{Bi}_{0.80}\text{Nd}_{0.20}\text{FeO}_3$	6.04	3.70	0.32	0.71

2.4. Magnetization of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics

2.4.1. Experimental Studies of Magnetization of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics

The dependencies of magnetization of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics on magnetic field were measured under a magnetic field up to $6.5 \cdot 10^6 \text{ A/m}$ at 5 and 300 K (figure 4). The magnetic parameters, such as a double remnant magnetization ($2 M_r$) and a double coercive force ($2 H_c$), provided with hysteresis loops are listed in table II.

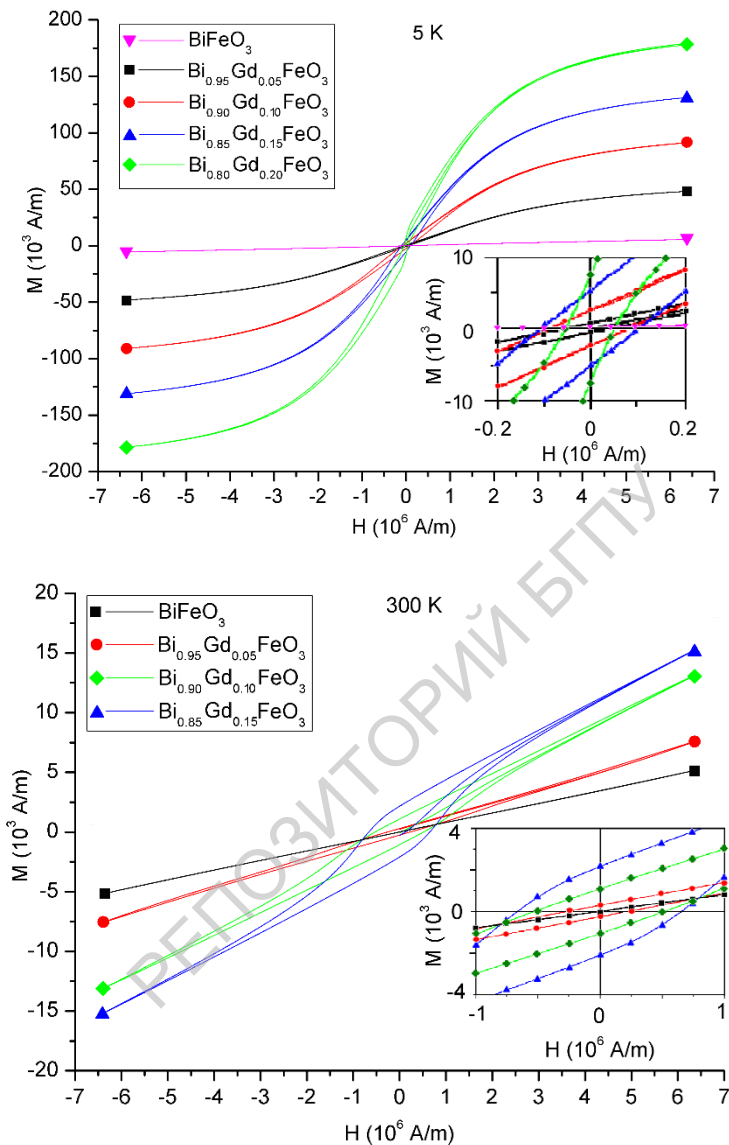


Figure 4. Field dependence of magnetization of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics with $x = 0, 0.05, 0.10, 0.15,$ and 0.20 at 5 (top) and 300 K (bottom) (Insets show M - H plots with enlarged view near origin).

Table II. Magnetization- Magnetic Field hysteresis loop parameters of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics at 5 and 300 K

Sample	$2 M_r, 10^3 \text{ A/m}$		$2 H_c, 10^6 \text{ A/m}$	
	5 K	300 K	5 K	300 K
$\text{Bi}_{0.95}\text{Gd}_{0.05}\text{FeO}_3$	1.72	0.542	0.079	0.472
$\text{Bi}_{0.90}\text{Gd}_{0.10}\text{FeO}_3$	4.90	2.17	0.164	1.03
$\text{Bi}_{0.85}\text{Gd}_{0.15}\text{FeO}_3$	9.80	4.25	0.204	1.30
$\text{Bi}_{0.80}\text{Gd}_{0.20}\text{FeO}_3$	14.95	-	0.100	-

Both magnetic parameters—a double remnant magnetization $2 M_r$ and a double coercive force $2 H_c$ —increase as the concentration of Gd increases from $x = 0$ to $x = 0.15$. This coincides with the results of paper [9] where the double remnant magnetization increased as the concentration of Gd increased from $x = 0$ to 0.03, 0.06, 0.10, 0.12, and 0.15 and was, correspondingly, 0.0006, 0.004, 0.011, 0.040, 0.081, and 0.145 emu/g. The increase of the weak magnetization with the Gd content for $x \leq 0.25$, the maximal value at $x = 0.25$ and then the gradual decrease with increasing x for $x \geq 0.30$ was found for $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ [4].

It can be concluded that the enhancement of the magnetic properties and the appearance of weak ferromagnetism were detected for all types of the substitution of Bi in BiFeO_3 – by La, Nd, and Gd. Comparing the double remnant ($2 M_r$) for the same content of the substitution, it can be observed that this enhancement is more pronounced for the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics. For the substitution with $x = 0.15$, the magnitudes of $2 M_r$ at 300 K are $1.12 \cdot 10^3$, $3.19 \cdot 10^3$, and $4.25 \cdot 10^3$ A/m for $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics, respectively.

2.4.2. Calculation of Magnetization of the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics

The calculated magnetization of $\text{Bi}_{0.80}\text{Gd}_{0.20}\text{FeO}_3$ at 5, 10, 20, 50, 100, and 300 K in dependence on the external field is presented in figure 5. The Brillouin function was applied without additional fitting parameters and the exchange interaction at low magnetic fields was not taken into account. The magnetization M was calculated using the expression

$$M = Ng\mu_B J \cdot B_J,$$

where N is the number of atoms per m^3 , g is the gyromagnetic g -factor, μ_B is the Bohr magneton, J is the total angular momentum quantum number, B_J is the Brillouin function. The used parameters were $3.0 \cdot 10^{27} \text{ m}^{-3}$ for N , 2 for g , and $7/2$ for J .

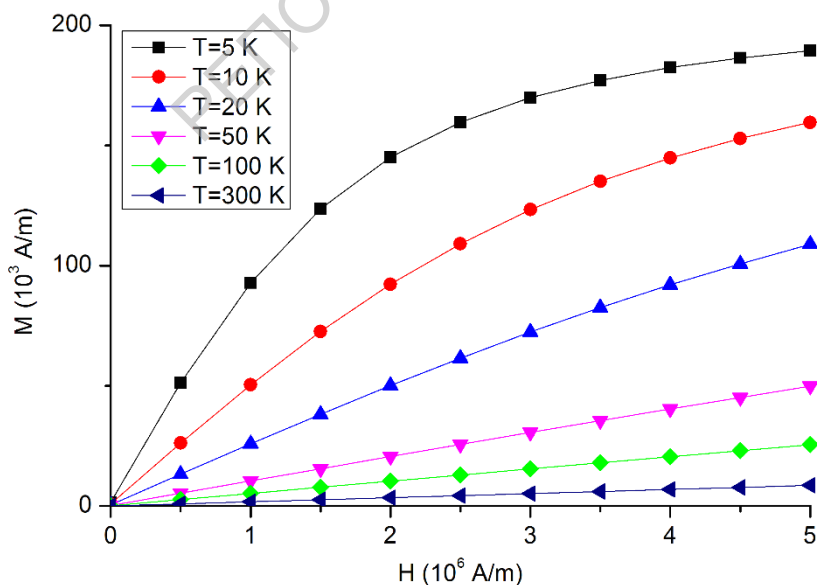


Figure 5. The calculated field dependence of magnetization of $\text{Bi}_{0.80}\text{Gd}_{0.20}\text{FeO}_3$ at 5, 10, 20, 50, 100, and 300 K.

The calculated dependencies show that the paramagnetic behavior is characteristic at temperatures 50, 100, and 300 K in the whole range of magnetic fields (figure 5). The change of slope at the magnetization-magnetic field curves for low temperatures (5, 10, and 20 K) points to the weak ferromagnetism for the substituted BiFeO_3 . The magnitude and dependence of the calculated magnetization of $\text{Bi}_{0.80}\text{Gd}_{0.20}\text{FeO}_3$ at 5 K (figure 5) correlate with the experimental magnetization measured at the same temperature (figure 4, top) except for the region of a small magnetic field. This is explained by the fact that the exchange interaction at low magnetic fields was not taken into account. The weak ferromagnetism can be explained by the exchange interaction between spins of atoms of iron and the substitution element.

SUMMARY

La-, Nd- and Gd-substituted bismuth ferrite (BiFeO_3) of the $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ types with the atomic part of the substitution element x equal up to 0.20 were synthesized by the solid-state reaction method using powders of oxides Bi_2O_3 , Fe_2O_3 , and La_2O_3 , or Nd_2O_3 , or Gd_2O_3 of pure grade quality and tested using X-ray diffraction analysis. The total substitution up to 0.20 atomic part of Bi by La, Nd, and Gd leads to the paramagnetic behavior of the doped bismuth ferrite at low temperatures in a wide range of magnetic field. Strong nonlinear dependence of magnetization on the magnetic field was detected and a ferromagnetic-like dependence of magnetization was observed for small magnetic fields. This can be explained by the exchange interaction between doping magnetic ions, as well as by the exchange interaction of these ions with ions of iron. The enhancement of the magnetic properties and the appearance of weak ferromagnetism were detected for all types of the substitution of Bi in BiFeO_3 – by La, Nd, and Gd. Comparing the double remnant ($2M_r$) for the same content of the substitution, it can be observed that this enhancement is more pronounced for the $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics. For the substitution with $x = 0.15$, the magnitudes of $2M_r$ at 300 K are $1.12 \cdot 10^3$, $3.19 \cdot 10^3$, and $4.25 \cdot 10^3$ A/m for $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$, $\text{Bi}_{1-x}\text{Nd}_x\text{FeO}_3$, and $\text{Bi}_{1-x}\text{Gd}_x\text{FeO}_3$ ceramics, respectively.

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