

Spatial Spin-Modulated Structure and Hyperfine Interactions of ^{57}Fe Nuclei in Multiferroics $\text{BiFe}_{1-x}\text{T}_x\text{O}_3$ ($T = \text{Sc, Mn}; x = 0, 0.05$)

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Abstract—The results of the Mössbauer studies on ^{57}Fe nuclei in multiferroics $\text{BiFe}_{1-x}\text{T}_x\text{O}_3$ ($T = \text{Sc, Mn}; x = 0, 0.05$) in the temperature range of 5.2–300 K have been presented. The Mössbauer spectra have been analyzed in terms of the model of an incommensurate spatial spin-modulated structure of cycloid type. Information has been obtained about the effect of the substitution of Sc and Mn atoms for Fe atoms on the hyperfine parameters of the spectrum: the shift and the quadrupole shift of the Mössbauer line, the isotropic and anisotropic contributions to the hyperfine magnetic field, and also the parameter of anharmonicity of the spatial spin-modulated structure.

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1. INTRODUCTION

Bismuth ferrite (BiFeO_3) occupies a unique position among multiferroics which simultaneously exhibit magnetic and ferroelectric ordering. High temperatures of the magnetic ($T_N = 643$ K) and ferroelectric ($T_C = 1103$ K) transitions allow their wide application based on the magnetoelectric effects [1]. BiFeO_3 has an incommensurate spatial spin-modulated structure (SSMS) of cycloid type [2] that determines its magnetic properties. Recently, it was found that a partial substitution of atoms of transition $3d$ elements for Fe atoms does not substantially decrease the Néel temperature T_N and the Curie temperature T_C , but markedly improves its electrical and magnetic properties (e.g., [3–11]).

These results show that multiferroic BiFeO_3 with insignificant containment of the transition metal impurity can be promising for applying in magnetoelectric devices. That is the reason why the studies of bismuth ferrites doped with transition-metal atoms attract high interest.

It is well known that the Mössbauer spectroscopy is effective in studies of local atomic state and hyperfine interactions of their nuclei. In recent works [12–14], it was shown that the Mössbauer spectroscopy is also an effective method of diagnostics and studying SSMS, along with nuclear magnetic resonance. The methods of Mössbauer spectroscopy sensitive to the hyperfine quadrupole interaction of nuclei in excited state makes

it possible to obtain information on features of SSMS and to fairly accurately determine the parameter of anharmonicity of SSMS of the cycloid type [12].

The aim of this work is to study the influence of the substitution of Sc and Mn atoms for Fe atoms on the spatial spin-modulated structure and the hyperfine interactions of ^{57}Fe nuclei in multiferroic BiFeO_3 using Mössbauer spectroscopy.

2. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUE

Polycrystalline samples of ferrites $\text{BiFe}_{1-x}\text{T}_x\text{O}_3$ ($T = \text{Sc, Mn}; x = 0, 0.05$) were prepared using the solid-state ceramic technology described in detail in [15]. The phase composition of the samples was determined using a PANalytical Empyrean X-ray diffractometer with CuK_α ($\lambda = 1.5405$ Å) radiation in the Bragg–Brentano geometry. The measurements were performed using a Pixel3D x – y detector with a Ni-filter on the diffracted beam. The X-ray diffraction patterns were processed using the HighScore Plus program and the ICDD PDF-4 database.

The Mössbauer spectra were measured on an MS1104Em spectrometer operating in the constant acceleration mode with a triangular form of varying the Doppler velocity of moving the source with respect to the absorber. The source was ^{57}Co nuclei in the Rh matrix. The Mössbauer spectrometer was calibrated at room temperature using an α -Fe standard absorber.

The measurements were performed in the temperature range of 5.2–300 K in an SHI-850-5 JANIS RESEARCH close-type helium cryostat and a small-size cryogenic complex produced by VNIIFTRI. The Mössbauer spectra were processed and analyzed using a method of interpreting the spectra in terms of the model of SSMS of cycloid type [12] that is realized in the SpectrRelax program [16, 17].

3. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns of the samples of synthesized substituted ferrites $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$.

The X-ray phase analysis confirms the formation of the ferrites with a rhombohedral structure (space group $R3c$). In this case, the X-ray diffraction patterns demonstrate the formation of impurity phase $\text{Bi}_{25}\text{FeO}_{39}$ in small amounts, whose main reflections are denoted by asterisks in Fig. 1.

Figure 2 shows the Mössbauer spectra of ^{57}Fe nuclei in studied ferrites $\text{BiFe}_{1-x}\text{T}_x\text{O}_3$ ($T = \text{Sc}, \text{Mn}$;

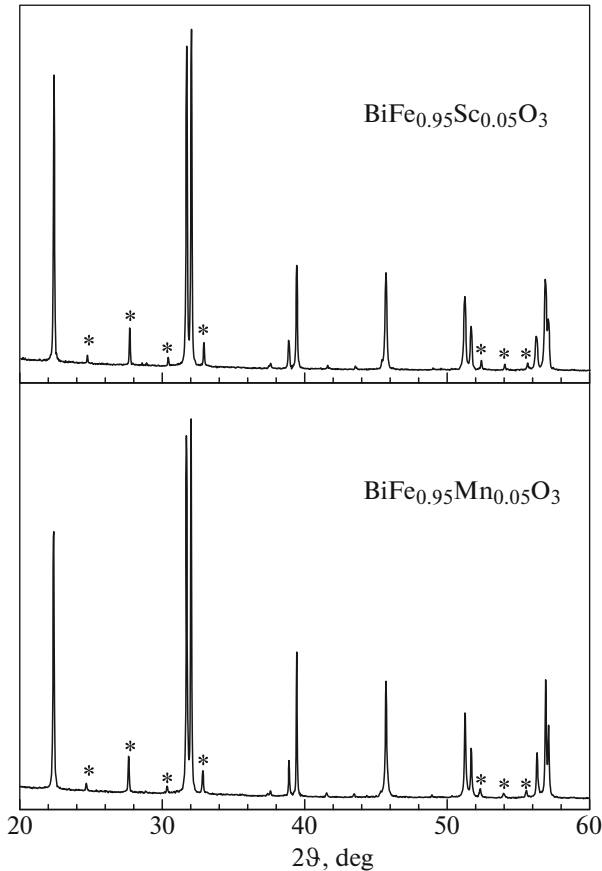


Fig. 1. X-ray diffraction patterns of the samples of synthesized ferrites $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$. The asterisks indicate the main reflections of the impurity phase $\text{Bi}_{25}\text{FeO}_{39}$.

$x = 0, 0.05$) measured at temperatures of 5.2 and 300 K. At $T = 300$ K, the spectrum contains the main partial spectrum of the ferrite in the magnetically ordered state ($\sim 96\text{--}92$ at % Fe) along with a small ($\sim 2\text{--}3$ at % Fe) paramagnetic contribution (quadrupole doublet) from ^{57}Fe nuclei in $\text{Bi}_{25}\text{FeO}_{39}$ and a small contribution of magnetically ordered type (Zeeman sextet) from ^{57}Fe nuclei in $\alpha\text{-Fe}_2\text{O}_3$ ($\sim 2\text{--}5$ at % Fe). In all the cases, the main partial spectrum of the ferrite exhibits the features related to the existence of a positive correlation of the hyperfine magnetic field and the quadrupole shift of the resonance lines due to the rotation of the Fe atom spin in SSMS of the cycloid type that is considered in detail in [12]. In this case, the resonance lines of the spectra of substituted ferrites $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ are slightly more broadened than those in the spectrum of pure ferrite BiFeO_3 (Fig. 2).

The Mössbauer spectra were interpreted in terms of the model of SSMS of the cycloid type over entire temperature range (5.2–300 K) lower than that the magnetic phase transition temperature of the studied ferrites [12]. In this model, according to [18, 19], the dependence of angle $\vartheta(x)$ between the antiferromagnetism vector and the axis of symmetry in the structure of bismuth ferrite BiFeO_3 on coordinate x along the direction of the spin modulation for the positive sign of the coefficient of uniaxial magnetic anisotropy K_u (the sign of K_u is discussed in more detail in [12]) is represented by the equation

$$\cos \vartheta(x) = \text{sn} \left(\frac{4K(m)}{\lambda} x, m \right) \quad \text{at } K_u > 0, \quad (1)$$

where λ is the wavelength of the anharmonic spin modulation; $0 \leq m \leq 1$ is the parameter of the Jacobi elliptic function $\text{sn}(x, m)$; $K(m)$ is the total first-order elliptic integral.

For every value of angle $\vartheta(x)$ from the range of variation in the coordinate $x \in [0, \lambda]$ there is a resonance absorption line in the form of a Zeeman sextet with shift δ , quadrupole shifts of the first order $\varepsilon(\vartheta)$ and the second order $a_{\pm}(\vartheta)$ of smallness in the expansion in terms of the energy of quadrupole interaction and hyperfine magnetic field $H_n(\vartheta)$. Shift δ of the Zeeman sextet that is determined by the degree of covalency of bonds and the dynamic properties of Mössbauer atoms was taken to be independent of angle ϑ . The quadrupole shifts of the resonance line in an inhomogeneous electric field with the axial symmetry in the region of disposition of ^{57}Fe nuclei in the structure of the studied ferrites in the first $\varepsilon(\vartheta)$ and the second $a_{\pm}(\vartheta)$ smallness orders were taken to be [20]

$$\varepsilon(\vartheta) = \varepsilon_{\text{lat}} \frac{3 \cos^2 \vartheta - 1}{2}, \quad (2)$$

$$a_{\pm}(\vartheta) = \varepsilon_{\text{lat}} \frac{3\varepsilon_{\text{lat}}}{g_{\text{ex}} \mu_n H_n} \left(\cos^2 \vartheta \pm \frac{1}{8} \sin^2 \vartheta \right) \sin^2 \vartheta, \quad (3)$$

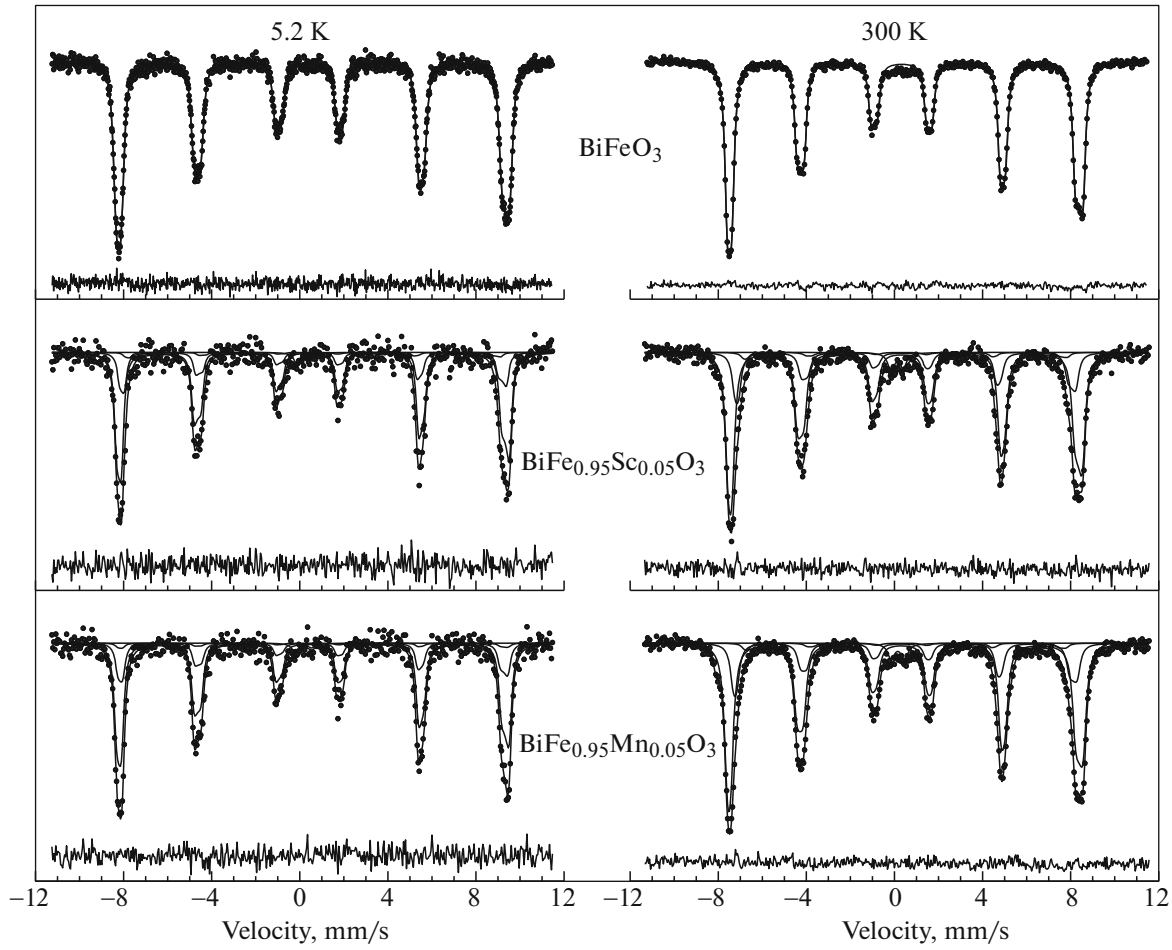


Fig. 2. Results of the mathematical treatment in terms of the model of anharmonic spin modulation of the Mössbauer spectra of ^{57}Fe nuclei measured at 5.2 and 300 K in ferrites BiFeO_3 , $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$, and $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$.

where ε_{lat} is the quadrupole shift caused by the electric field gradient induced by atoms surrounded the nucleus; μ_n is the nuclear magneton; g_{ex} is g factor of the excited state of a ^{57}Fe Mössbauer nucleus. In formula (2), we did not take into account additional possible shift $\varepsilon_{\text{magn}}$ that is due to a local lattice distortion because of strong magnetoelectric interaction [21], since, as is shown in [12], this additional shift can be neglected. In the case of the uniaxial anisotropy of the hyperfine interaction when it is fairly small, as is the case with ^{57}Fe nuclei in BiFeO_3 , the hyperfine magnetic field at ^{57}Fe $H_n(\vartheta)$ can be represented as

$$H_n(\vartheta) = H_{\text{is}} + H_{\text{an}}(3 \cos^2(\vartheta) - 1)/2, \quad (4)$$

where H_{is} is the isotropic contribution to the hyperfine magnetic field H_n that is mainly determined by the contact Fermi interaction with s electrons polarized by atomic spin and localized at a nucleus; H_{an} is anisotropic contribution due to the magnetic dipole–dipole interaction with localized magnetic moment of atoms and the anisotropy of the hyperfine magnetic interaction of the nucleus with electrons of the ionic core of

the proper atom. It follows from Eq. (4) that the values of hyperfine magnetic fields in the case when the magnetic moment of a Fe atom is parallel (H_{\parallel}) and perpendicular (H_{\perp}) to the axis of symmetry of the crystal which were indicated in [12–14, 21] are related to the isotropic and anisotropic contributions by simple relationships: $H_{\parallel} = H_{\text{is}} + H_{\text{an}}$ and $H_{\perp} = H_{\text{is}} - H_{\text{an}}/2$. When interpreting the Mössbauer spectra in terms of the model of SSMS of the cycloid type, we searched for optimum values of all parameters of the hyperfine interaction (δ , ε_{lat} , H_{is} , H_{an}) and the parameter of anharmonicity of the spin modulation (m).

Since bismuth ferrite BiFeO_3 has only one crystallographically nonequivalent position of Fe atoms with the same nearest cation environment from six Fe atoms for all positions of a Fe atom, only one partial spectrum in the model of SSMS of the cycloid type corresponded to the Mössbauer spectrum of the ferrite upon its interpreting. In the case of the substituted ferrites $\text{BiFe}_{0.95}\text{T}_{0.05}\text{O}_3$ ($T = \text{Sc}, \text{Mn}$), their Mössbauer spectra were interpreted, assuming that there are three partial spectra in the model of SSMS of the cycloid

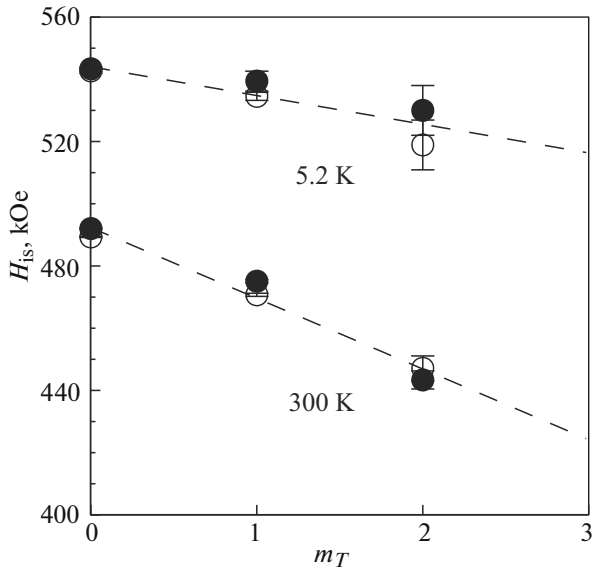


Fig. 3. Isotropic contribution to the hyperfine magnetic field on ^{57}Fe nuclei in (bright circles) $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and (dark circles) $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ in the dependence on number m_T of Sc(Mn) atoms in the nearest cation coordination shell of an Fe atom at 5.2 and 300 K.

type which correspond to iron atoms the nearest cation environment of which contains different numbers of atoms of the T impurity $m_T = 0, 1$, and 2 . These partial spectra corresponded to the model of unique anharmonic spin modulation, in which spins of all iron atoms take part. In this case, line shifts δ , quadrupole shift ε_{lat} , anisotropic contributions H_{an} to the hyperfine magnetic field, and anharmonicity parameter m for these partial spectra were taken to be the same, and the isotropic contributions H_{is} were taken to be different. Upon the model interpretation, the ratios of the intensities of the partial spectra of the substituted ferrites corresponded to binominal distribution $P_6(m_T)$ of the T impurity atoms according to a random distribution of the T impurity atoms over positions of iron atom in the bismuth ferrite structure:

$$\begin{aligned} I(m_T = 0) : I(m_T = 1) : I(m_T = 2) \\ = P_6(m_T = 0) : P_6(m_T = 1) : P_6(m_T = 2) \\ = 73.5 : 23.2 : 3.1. \end{aligned}$$

As is seen in Fig. 2, the description of the experimental spectra in terms of the chosen model of SSMS of the cycloid type with allowance for two low-intensity partial spectra of impurity phases ($\text{Bi}_{25}\text{FeO}_{39}$ and $\alpha\text{-Fe}_2\text{O}_3$) is good (with normalized $\chi^2 = 1.0\text{--}1.2$) over entire temperature range under study. The results of searching for optimal values of the parameters of hyperfine interactions and the parameter of anharmonicity of the spin modulation upon the model interpretation in terms of the model of SSMS of the cycloid type are presented in Figs. 3–6.

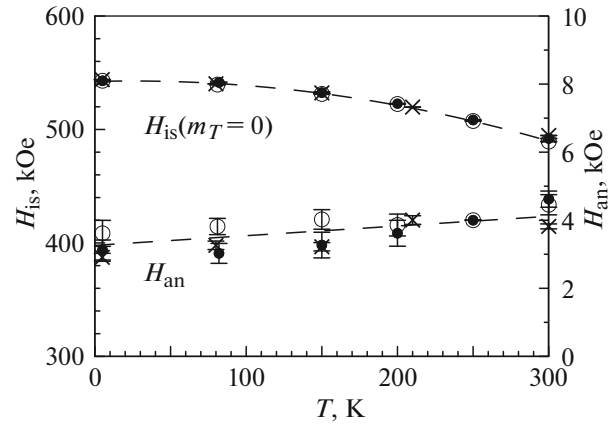


Fig. 4. Temperature dependences of isotropic H_{is} and anisotropic H_{an} contributions to the hyperfine magnetic field on ^{57}Fe nuclei in (crosses) BiFeO_3 , and also in (bright circles) $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and (dark circles) $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ for iron atoms in the cation environment of six Fe atoms ($m_T = 0$).

Figure 3 shows the dependences of the isotropic contribution to the hyperfine magnetic field on the number of impurity atoms m_T in the nearest cation environment at temperatures 5.2 and 300 K. It is seen that these contributions decrease almost linearly as m_T increases, and this decrease is observed most clearly at room temperature. The substitution of one Mn or Sc atom for one Fe atom in the nearest cation environment of a Fe atom decreases the isotropic contribution to the hyperfine magnetic field by 22 ± 2 kOe at 300 K and by 10 ± 3 kOe at 5.2 K. Isotropic contribution H_{is} to the hyperfine magnetic field on nuclei of all Fe atoms, like as the average value of the spin, decreases as temperature increases; in this case, contribution $H_{\text{is}}(m_T)$ for a Fe atom, whose nearest cation environment contains only Fe atoms, is almost the same as that in the case of pure bismuth ferrite (Fig. 4).

The values of anisotropic contribution $H_{\text{an}} \sim 3\text{--}4$ kOe to hyperfine field H_n at the ^{57}Fe nuclei (Fig. 4) obtained as a result of the interpretation demonstrate strong anisotropy of hyperfine magnetic interactions in the studied ferrites. The anisotropy of the hyperfine magnetic field is usually associated with the dipole contribution H_{dip} that can be estimated in an approximation of localized magnetic moments of Fe. Using the low-temperature structural data for ferrite BiFeO_3 [22] and taking into account the features of its magnetic structure, we calculated dipole contribution $H_{\text{dip}} \sim 0.04$ kOe that is significantly smaller than the anisotropic contribution determined experimentally. Note that anisotropic contribution H_{an} to the hyperfine field H_n at ^{57}Fe nuclei in ferrites $\text{BiFe}_{1-x}\text{T}_x\text{O}_3$ ($T = \text{Sc, Mn, } x = 0, 0.05$) slightly increases from ~ 3 to ~ 4 kOe (Fig. 4) with increasing temperature in the

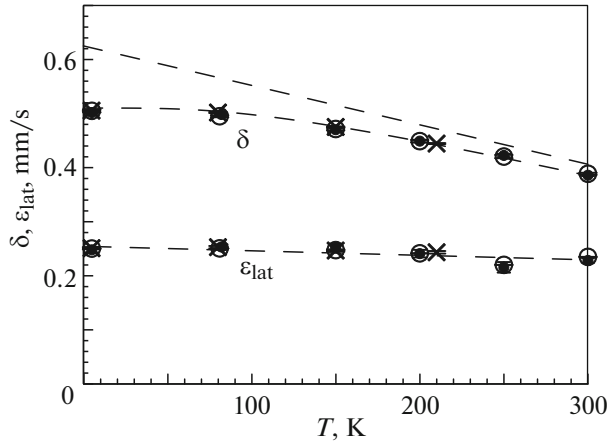


Fig. 5. Temperature dependences of shift δ and quadrupole shift ε_{lat} of the components of the Mössbauer spectra of ^{57}Fe nuclei in (bright circles) $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$, (dark circles) $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$, and (crosses) BiFeO_3 . The dashed lines in dependence $\delta(T)$ correspond to the Debye approximation in the description of the vibration spectrum of ^{57}Fe nuclei at $\vartheta_D = 420$ K and to the classical limit.

range under study (5.2–300 K), which also cannot be due to the dipole contribution, since the average values of the magnetic moments of the Fe atoms decrease as temperature increases. Possible causes of the observed anisotropy of the magnetic hyperfine interactions in the studied ferrites can be related to an internal electron contribution that forms due to covalency effects.

The Mössbauer line shifts δ , as well as the quadrupole shifts ε_{lat} of the spectral components, for all the substituted ferrites $\text{BiFe}_{0.95}T_{0.05}\text{O}_3$ ($T = \text{Sc}, \text{Mn}$) and pure BiFeO_3 almost completely coincide with each other over the entire temperature range studied (Fig. 5). As temperature increases, Mössbauer line shift δ decreases, according to the temperature contribution δ_T due to the dynamic properties of the Fe atoms. This behavior of the shift is described well in a Debye approximation of the vibration spectrum of the Fe atoms at the Debye temperature $\vartheta_D = 420$ K (Fig. 5). As is shown in [14], the observed high quadrupole shift (~ 0.23 mm/s) in BiFeO_3 and, therefore, in $\text{BiFe}_{0.95}T_{0.05}\text{O}_3$ ($T = \text{Sc}, \text{Mn}$) can be obtained when calculating the electric field gradient tensor with allowance for contribution of not only localized charges. The insignificant decrease in quadrupole shift ε_{lat} with decreasing temperature observed experimentally (Fig. 5) seems to be caused by the thermal expansion of the ferrite lattice.

The interpretation of the spectra in terms of the SSMS model of the cycloid type made it possible to obtain the value of the anharmonicity parameter of spin modulation m in the studied ferrites. As a result, it was found that the anharmonicity parameter

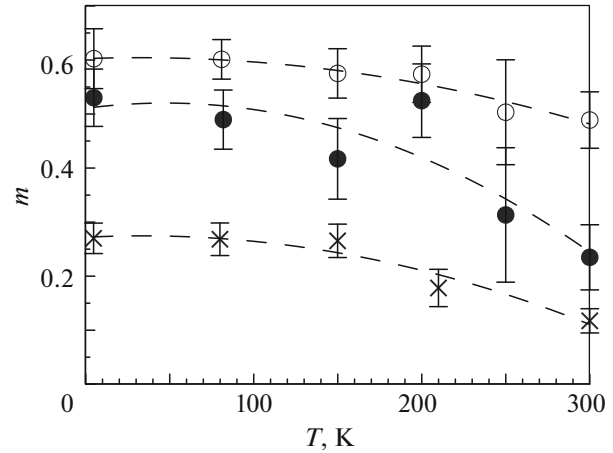


Fig. 6. Temperature dependences of anharmonicity parameter m of the spin modulation in (bright circles) $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$, (dark circles) $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$, and (crosses) BiFeO_3 .

increases (at $T = 5.2$ K, from $m = 0.27 \pm 0.03$ to 0.5–0.6) as impurity atoms (Mn or Sc) are substituted for Fe atoms in the BiFeO_3 structure (Fig. 6). In this case, the anharmonicity parameter of substituted ferrites $\text{BiFe}_{0.95}T_{0.05}\text{O}_3$ ($T = \text{Sc}, \text{Mn}$), as is the case with BiFeO_3 [13], decreases by ~ 0.1 – 0.2 as temperature increases. Such behavior of anharmonicity parameter m is due to the change in the constant of uniaxial magnetic anisotropy K_u and the energy of nonuniform exchange interaction $E_{\text{exch}} = Aq^2$, where A is the constant of inhomogeneous exchange (exchange hardness), and $q = 2\pi/\lambda$ is the wave number. According to [23], parameter m is related to coefficient K_u and energy E_{exch} by the relationship

$$mK^2(m) = \frac{\pi^2 K_u}{4 E_{\text{exch}}}. \quad (5)$$

As follows from Eq. (5), the increase in the anharmonicity parameter of spin modulation m as impurity atoms (Mn or Sc) are substituted for Fe atoms in the BiFeO_3 structure can be caused by both the increase in the anisotropy coefficient K_u because of the formation of local structural distortions and the decrease in the energy of exchange interaction E_{exch} . As for the decrease in m with increasing temperature, it seems to be due to, first, the decrease in anisotropy coefficient K_u .

4. CONCLUSIONS

The influence of the substitution of Sc and Mn atoms for Fe atoms on the spatial spin-modulated structure and also hyperfine electric and magnetic interactions of ^{57}Fe nuclei in multiferroic BiFeO_3 has been studied using Mössbauer spectroscopy. As a result, the following was stated. The structure of the

substituted ferrites contains the iron atom positions in the first cation coordination shell of which there are one or two impurity atoms. The substituted ferrites $\text{BiFe}_{0.95}\text{Sc}_{0.05}\text{O}_3$ and $\text{BiFe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ have the anharmonically modulated spin structure of the cycloid type in which iron atoms with various cation environments take part. As Sc or Mn atoms are substituted for Fe atoms in the BiFeO_3 structure, the anharmonicity parameter of the spin modulation increases (at $T = 5.2$ K, from 0.27 ± 0.03 to $0.5\text{--}0.6$). As temperature increases from 5.2 to 300 K, the anharmonicity parameter decreases by $0.1\text{--}0.2$. As one Mn atom or one Sc atom are substituted for one Fe atom in the nearest cation environment of a Fe atom, the isotropic contribution to the hyperfine magnetic field decreases (by 22 ± 2 kOe at 300 K and by 10 ± 3 kOe at 5.2 K), but, in this case, the anisotropic contribution, the quadrupole shift, and the Mössbauer line shift are not noticeably changed.

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