Although no consistent microscopic theory for describing the mechanisms of the appearance of ferroelectricity and dynamic coupling in the multiferroics studied has yet been developed, it is already clear that the electric polarization vector appears as a result of breaking the centrosymmetricity of the system due to the appearance (in a specific temperature interval) of cycloidal magnetic structures incommensurate with the crystal lattice. At present, the most widely applied mechanism is the exchange-relativistic inhomogeneous magnetoelectric interaction which leads to the appearance of a local polarization vector  $\mathbf{p} \sim [\mathbf{e}_{ij}[\mathbf{S}_i\mathbf{S}_j]]$  with a noncollinear arrangement of the adjacent spins, where  $\mathbf{e}_{ij}$  is the vector that connects spins  $S_i$  and  $S_i$  [23, 25, 26] (Dzyaloshinskii–Moriya exchange interaction). The total polarization vector for this cycloidal structure proves to be proportional to the vector product of the wave vector **k** of the magnetic structure and the normal **e** to the plane of the spin rotation:  $\mathbf{P} \sim [\mathbf{ek}]$  [23]. In the continuum approximation, as follows from a group-theoretical analysis, the observed ferroelectric properties of these multiferroics are caused by a Lifshitz type inhomogeneous magnetoelectric interaction:

$$\Phi_{\rm me} = -a_x P_x \left( A_x \frac{\partial A_y}{\partial y} - A_y \frac{\partial A_x}{\partial y} \right) - a_z P_z \left( A_z \frac{\partial A_y}{\partial y} - A_y \frac{\partial A_z}{\partial y} \right),$$

where A is the antiferromagnetic vector [12, 20], which both leads to a spontaneous polarization and determines the contribution to the dielectric constant and the coupling of the homogeneous electric field with spin excitations. With this type of magnetoelectric interaction, the change in the plane of rotation of  $Mn^{3+}$  spins in the cycloid from *bc* to *ab* should lead to a change in the excitation condition of electromagnons  $(\mathbf{e} \| a \to \mathbf{e} \| c)$ ; however, as follows from our and other investigations, this is not the case, and the electromagnons are observed only at the  $\mathbf{e} \parallel a$  polarization. This means that another type of magnetoelectric coupling can also exist, which has been suggested recently in Ref. [22]. This type of magnetoelectric coupling, which has an exchange origin, is related to specific features of the crystal structure and determines the contribution to the dielectric constant along the *a*-axis irrespective of the cycloid orientation. However, as was revealed, this contribution comes only from high-lying spin excitations corresponding to the Brillouin zone boundary. Therefore, there is no full understanding of what role is played by the magnetoelectric interactions of this type in the formation of the electromagnon response, and the problem calls for additional investigations.

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# Heteroepitaxial films of a bismuth ferrite multiferroic doped with neodymium

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The possibility of the coexistence of spontaneous magnetic moments and electric polarization does not contradict the general criteria for the formation of ferromagnetism and ferroelectricity as separate phenomena. The magnetic ordering is determined by the exchange interaction of electron spins, while ferroelectric ordering by a redistribution of charge density in the crystal lattice. Bismuth ferrite (BiFeO<sub>3</sub>) is one of the first ferroelectromagnets in which there were discovered ferroelectric [1, 2] and antiferromagnetic [3, 4] ordering with uniquely high temperatures of electric ( $T_c = 1083$  K) and magnetic ( $T_N = 643$  K) ordering, but the high electrical conductivity of the samples hampered their study and practical implementation [5].

At room temperature, the crystal structure of a bismuth ferrite single crystal is described by the space group R3c. Its rhombohedral unit cell (a = 0.562 nm,  $\alpha = 59.35^{\circ}$ ) contains two formula units. The spontaneous polarization is oriented in the direction [111] of the pseudocubic perovskite unit cell. G type antiferromagnetic ordering appears when the magnetic moments of iron ions, preserving a locally antiparallel orientation, are turned in a spiral oriented along the direction  $[10\overline{1}]$ . According to neutron-diffraction data [6], the period of this cycloid equals 62 nm. The interaction of spin waves with optical phonons in BiFeO<sub>3</sub> single crystals was recently investigated by light scattering methods [7].

Recently, interest in bismuth ferrite has grown sharply, which is connected with the detection in the films of this compound of a giant magnetoelectric (ME) effect (the appearance of magnetization under the action of an electric field [8]) and of a giant magnetocapacitance (the appearance of electric polarization under the action of a magnetic field [9]). Under normal conditions, these effects are not observed because of the presence of a cycloid — a spatially modulated structure in which the magnetization vectors of the sublattices periodically change direction from point to point. On account of the presence of a cycloid, the average-over-the-volume linear ME effect and spontaneous magnetization are equal to zero [10]. The magnetoelectric effect observed in BiFeO<sub>3</sub> films with a nanoscale thickness points to the destruction of the spatially modulated structure in films, which is caused by high internal deformation fields arising due to the misfit of the lattice parameters of the film and substrate and related deformation of the unit cell [11, 12]. As a result of electrostriction, these stresses in nanoscale films lead to the appearance of an additional contribution to the electric polarization. A similar effect occurs in ferroelastics. Therefore, the presence of magnetic ordering, a redistribution of charge density, and electrostriction lead to the appearance of new properties in heterostructures based on ferroelectromagnets (multiferroics, according to the contemporary terminology), which are quite important from the viewpoint of practical application.

Thus, the development of the methods of synthesis and crystallization of multiferroic heterostructures on different substrates has become an urgent task. For the real application of multiferroic films, it is necessary that the ferroelectric polarization, the piezoelectric coefficient, and the magnetization have sufficiently high values, just like the magnetoelectric interaction.

In recent years, new multiferroic materials with high values of physical characteristics have been obtained by doping traditional single-phase multiferroic  $BiFeO_3$  with various ions of rare-earth metals [12, 13]. Doping with neodymium ions favors the suppression of the cycloidal structure and can lead to an increase in the remanent polarization and saturation magnetization [13], as well as to a decrease in the ferroelectric coercive field and leakage currents [12].

In this report, we present results on the preparation and investigation of heteroepitaxial films of BiFeO<sub>3</sub> multiferroic doped with neodymium and deposited on single-crystal MgO substrates.

To obtain the ( $Bi_{0.98}Nd_{0.02}$ )FeO<sub>3</sub> (BNFO) films on (001) cuts of MgO single crystals, we used the technique of high-frequency (HF) sputtering of stoichiometric ceramic targets of the same composition at elevated pressures of oxygen, which earlier was successfully applied to obtaining epitaxial films of barium strontium titanate (BST) [14]. The basic distinction between our procedure of deposition and the known ones consisted in our using a heavy-current HF discharge to bring about film growth. The supplied high-frequency power (73 W cm<sup>-2</sup>), a special geometry of the electrodes, and a high pressure of oxygen (0.6 Torr) made it

possible to pulverize the oxide at a cluster level and to create in the plasma a dispersed phase of the initial oxide, which serves as a vapor phase for the growing film. The basic difference between the procedure of obtaining BST heterostructures and that of obtaining BNFO is in the effect of the substrate temperature on the synthesis and crystallization of the film. In the case of the BST structure, an increase in the temperature from 300 °C to 700 °C led to a progressive increase in the degree of structural perfection of the films in the following sequence: X-ray amorphouspolycrystalline-textured-single-crystalline, whereas in the structures on the base of BNFO the reverse sequence was observed as the substrate temperature increased from 340 °C to 550 °C: single-crystal films-textured filmspolycrystalline films-films consisting of a mixture of oxides.

Measurements of the composition of the target and the films was performed using a Comebax-micro analyzer. As the standard, we used a BiFeO<sub>3</sub> single crystal. The neodymium concentration in the films coincided with the concentration in the target to an accuracy of 1%.

The structural perfection of the films, the lattice parameters along the normal to the substrate plane and in the substrate plane, and the orientation relationships between the film and the substrate at room temperature were established by X-ray diffraction method using a DRON-7 diffractometer ( $\Theta - 2\Theta$  method; taking symmetric and asymmetric Bragg reflections; Cu  $K_{\alpha}$  radiation). The deposition of BNFO films on MgO (100) substrates showed that the threshold temperature of the substrate in the process of deposition for a singlecrystal growth of the film runs to 350 °C, which is lower than the temperatures of both the ferroelectric and antiferromagnetic phase transitions. An increase in the temperature to above 500 °C leads to the film condensation in the form of a mixture of simple oxides.

The X-ray diffraction spectrum ( $\Theta - 2\Theta$  scanning) of a single-crystal BNFO film with a thickness of 35 nm on an MgO (100) substrate in the  $20^{\circ} - 50^{\circ}$  range of angles is given in Fig. 1. The diffractogram contains only (001) and (002) reflections of the perovskite phase of the BNFO, and a (002) reflection from the substrate, which indicates that the [001]-axis of the film is parallel to the [001]-axis of the MgO substrate. The vertical misorientation of the film relative to



**Figure 1.** Typical X-ray diffraction pattern ( $\Theta - 2\Theta$  scanning) of a singlecrystal BNFO film on MgO (001). The inset displays the diffractograms obtained by  $\varphi$  scanning of BNFO (113) and MgO (113) reflections.



**Figure 2.** Variation of the lattice parameters along the normal to the film surface and in the film plane as a function of the film thickness.

the normal to the substrate, as determined from the widths of rocking curves, reaches  $\sim 1^{\circ}$ .

To prove the fact of heteroepitaxial growth, reveal the azimuthal misorientation of the film, and determine the orientation relationships between the film and substrate, we used the method of  $\varphi$  scanning of pseudocubic reflections (113) from the film and (113) reflections from the substrate. The corresponding X-ray diffractograms of (113) reflections from the film and the substrate are presented in the inset to Fig. 1. In the film, there is only one azimuthal orientation of the film relative to the substrate: the [001]-axis of the film is parallel to the [001]-axis of the MgO substrate, while the [100]and [010]-axes of the film are oriented along the [110] and [110] directions of the substrate, respectively. Therefore, the diffractogram (see inset to Fig. 1) contains four reflections appearing when the sample is rotated in its plane through an angle of 360°, but the reflections from the film and substrate are shifted by an angle of 45° relative to each other.

To more precisely determine the lattice parameter c of the film in the direction of the normal to the film by the X-ray diffraction method, we used reflections of the (00l) type. For determining lattice parameters of the film in the *ab* plane of the substrate, we used the asymmetric method of taking (024) type reflections. A room-temperature X-ray diffraction studies of BNFO films of different thicknesses on MgO (001) substrates revealed the presence of some peculiarities in the dependences of the film lattice parameters both along the normal to the plane of the substrate and in the plane of the substrate layer. The lattice parameters were calculated in the tetragonal approximation. As is shown in Fig. 2, in the region of thicknesses from 20 to 650 nm the lattice parameter c of the BNFO films along the normal to the plane of the base layer decreases, and the parameters in the substrate plane a = bincrease with increasing thickness of the film. In the region of small thicknesses, the lattice parameter c of the films is greater than the lattice parameter of the perovskite subcell of the rhombohedrally distorted phase of the bulk sample (0.3964 nm), whereas the parameter a is smaller, which testifies to the presence of compressive stresses in the films in the substrate plane. In the very thick BNFO films on MgO (001) substrates, the lattice parameters relax to values characteristic of the bulk state.

It should be noted here that in practice the MgO substrates (NaCl type structure, the lattice parameter a = 0.4212 nm) are mainly used to create tensile stresses in

BST films, which has repeatedly been confirmed in works by various authors, as well as in ours. Taking into account the proximity of lattice parameters of the BST and BNFO films, this behavior of the lattice parameters of the BNFO films with decreasing thickness of the films can hardly be explained by the assumption about the prevailing influence of the misfit in the lattice parameters of the film and the substrate.

It is worthy of note that the epitaxial growth of perovskites on MgO (001) substrates was analyzed theoretically in Ref. [15], where it was shown that, from the viewpoint of the minimization of stresses and a considerable decrease in the density of misfit dislocations in the film, such a conjugation of the unit cells of the film and the substrate is energetically more advantageous where the unit cell of the film of the perovskite PbTiO<sub>3</sub> is rotated through 45° relative to that of MgO in the conjugation plane. However, in the case of films of the ferroelectric perovskite BST on MgO substrates, which were investigated in our previous work [14], the growth of the films always occurred with a parallel conjugation of the axes of the film and the substrate. The epitaxial growth with a turn of the crystallographic axes of the film relative to the MgO substrate is a specific feature of BNFO films, which still had to be clarified. No such conjugation effects were observed upon deposition of bismuth ferrite films on other substrates.

For the investigation of the dielectric characteristics of the films, planar electrodes were applied onto the film surface using contact photolithography and ion-beam sputtering of aluminium with a vanadium underlayer. The planar electrodes represented an interdigitated structure with aluminium electrodes about 0.3 µm thick. The interdigital planar capacitor consisted of 470 fingers with a dielectric clearance between them 0.65–0.75  $\mu$ m wide at a length of 140  $\mu$ m and width of 1.55–1.75 µm of each finger. The real geometrical dimensions of the planar capacitor for determining the dielectric constant after the lithographic process were obtained with a Leitz Latimet microscope. The capacity, conductivity, and leakage currents of the planar capacitors were measured with a Keithley 4200-SCS semiconductor characterization system at a frequency of 1 MHz with the aid of a PM-5 MicroTec probe station. The results of the measurements of the electrode topology and the capacity were used for calculating the dielectric constant according to the formulas given in Ref. [16].

In none of the samples investigated did the DC leakage currents exceed  $10^{-9}$  A at an external voltage across the planar electrodes equal to 45 V, and the dielectric loss tangent at a frequency of 1 MHz was as low as 0.08. The dependence of the dielectric constant on the film thickness is presented in Fig. 3. In the inset to this figure, dielectric hysteresis loops are shown (at the film thickness of 70 nm), from which it is evident that at a frequency of 0.05 Hz the remanent electric polarization was 7  $\mu$ C cm<sup>-2</sup>; with increasing frequency, it decreased to 2  $\mu$ C cm<sup>-2</sup> at a frequency of 5 Hz and remained constant at frequencies of up to 10<sup>3</sup> Hz. The dielectric hysteresis loops obtained in our samples differ from those presented in Ref. [8] for films of the same composition, which even at high frequencies remain 'rectangular'. The dielectric hysteresis loops given in Ref. [12] occupy an intermediate position with respect to those considered above; however, in the last work the applied electric field was greater by an order of magnitude. It should be emphasized that the shapes of the dielectric hysteresis loops measured in the usual structure of a capacitor (metal-dielectric-metal) and with a planar struc-



**Figure 3.** Effect of the thickness of a BNFO film on its dielectric constant. The inset shows dielectric hysteresis loops.



Figure 4. Polarized Raman spectra of a BNFO film in the geometries of normal backscattering.

ture of the electrodes should differ substantially as a result of the principally different field distribution. A study of the processes of polarization switching in planar capacitors is only beginning in essence, despite the fact that the practical employment of BNFO heterostructures on dielectric substrates is only possible with this topology of the electrodes.

The phonon spectra of the films were investigated by the Raman spectroscopy method. The spectra of micro-Raman scattering (MRS) were excited by polarized radiation from an argon laser ( $\lambda = 514.5$  nm) and were detected by a Renishaw single-pass spectrometer equipped with a NExT (near-excitation tunable) filter for an analysis of the low-frequency region of the spectra. The exciting radiation was focused onto a sample using a Leica optical microscope; the diameter of the focused spot on the sample equaled 2 µm. The polarized Raman spectra were obtained in samples precisely oriented relative to the crystallographic axes of the MgO substrate. The coordinate system used was selected as follows:  $X \parallel [100]$ ,  $Y \parallel [010]$ , and  $Z \parallel [001]$ .

The Raman spectra of the BNFO film (270 nm thick) obtained in the geometry of normal backscattering, in which the incident beam is directed along the normal to the film surface along the Z-axis, are given in Fig. 4. The polarized



Figure 5. Polarized Raman spectra of a BNFO film in the geometries of backscattering from the butt edge of the film.

spectra of this film contain all the lines that were observed earlier in the spectra of bismuth ferrite single crystals at room temperature [17]. The unit cell of a rhombohedral singlecrystal bismuth ferrite contains two formula units. The optical modes  $4A_1 + 9E$  predicted by the factor-group analysis were identified in the spectra of a single crystal in Ref. [17]. Doping with neodymium does not virtually affect the phonon spectra.

No noticeable shifts in optical mode frequencies, indicating the presence of strong mechanical stresses in the film, have been observed in the spectra of a BNFO film. This is apparently explained by the above-described specific type of conjugation of the film with the crystal lattice of the MgO substrate. In the spectra of backscattering from the butt edge of the film given in Fig. 5, a line at 74 cm<sup>-1</sup> is observed in the scattering geometry  $Y(XZ)\overline{Y}$ , originating from the E(TO) component of the soft mode, as are three intense lines at 142, 171, and 225 cm<sup>-1</sup>, which originate from the A<sub>1</sub> symmetry modes of the rhombohedral phase of the BiFeO<sub>3</sub> single crystal [17]. The Raman spectra of the two diagonal orientations  $[Y(XX)\overline{Y}$  and  $Y(ZZ)\overline{Y}]$  are virtually equivalent, which also excludes the tetragonal *c*-domain structure of the film.

An important feature of the spectra of BNFO films on an MgO substrate is their electric polarization dependence, which has not been observed previously for films of pure or neodymium-doped bismuth ferrite. Thus, in the Raman spectra of polycrystalline rhombohedral BNFO films on the  $Pt/TiO_2/SiO_2/Si$  substrates [12] and of films of pure BiFeO<sub>3</sub> on LaNiO<sub>3</sub>/SrTiO<sub>3</sub> [18], no polarization dependence was observed because of the polycrystalline character of the films. The spectra of tetragonal c-domain BiFeO<sub>3</sub> films grown on SrTiO<sub>3</sub>(001) substrates [19] were polarized in accordance with the selection rules for the tetragonal point group P4mm. Namely, in the scattering geometry  $Y(XZ)\overline{Y}$ (the coordinate system coincides with that selected by us), modes of only E symmetry are observed. The Raman spectrum corresponding to the geometry  $Z(YX)\overline{Z}$  contains only weak lines which appear from diagonal geometries because of polarization disturbances, whereas no intrinsic lines, corresponding to a given type of symmetry, have been revealed, which confirms the tetragonal symmetry of the film with a polarization direction along [001]-axis in the film grown on an SrTiO<sub>3</sub> (001) substrate.

The polarization characteristics of the Raman spectra exclude the rhombohedral symmetry of the BNFO film on MgO. In addition, the presence of intense lines in the scattering geometry  $Z(YX)\overline{Z}$  in our BNFO film unambiguously indicates that the film on an MgO substrate is not a tetragonal c-domain one. As is shown in Fig. 4, the lowfrequency mode (72 cm<sup>-1</sup>) in the geometry  $Z(YX)\overline{Z}$  is almost twice as wide as the low-frequency peak in the  $Z(YY)\overline{Z}$ spectrum, whose frequency amounts to 75 cm<sup>-1</sup>. This fact testifies to the splitting of the E(TO) mode of the rhombohedral bismuth ferrite phase into two components manifesting themselves in different scattering geometries, which is possible only as a result of a reduction of the BNFO film symmetry to orthorhombic or even monoclinic. The spectra in the  $Z(YY)\overline{Z}$  and  $Z(XX)\overline{Z}$  geometries are virtually equivalent, and a rotation of the sample about the Z-axis through an angle of 45° does not affect the polarization characteristics of the spectra, which appears to be caused by a complex 90°-domain structure. The presence in the  $Z(YX)\overline{Z}$  spectrum of an intense component of the soft mode suggests the orientation of the spontaneous electric polarization in the film plane or, at least, the presence of a polarization component that lies in this plane.

A symmetry analysis of structural distortions induced by the rotation of oxygen octahedra and by polar displacements of cations in films of perovskites obtained on cubic substrates was recently performed in Ref. [20], where more than 30 possible phases have been identified. To determine the symmetry of bismuth ferrite films doped with neodymium that are grown on MgO(001) single-crystal substrates, precision structural investigations are required. Since the destruction of a spin cycloid in the films can occur due to mechanical stresses, of special interest are investigations of films with different thicknesses.

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