International Workshop on Phase Transitions and Inhomogeneous States in Oxides

June 25-30, 2017, Kazan, Russia

Book of Abstracts

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PROGRAM

26 June, 2017 **Opening session** **16.20-16.30**

Session chairs:

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Organizing Committee Deputy Chair R.F. Mamin (Kazan, Russia)

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4. MirHasan Yu. Seyidov (Gebze, Turkey)
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INVITED AND ORAL

PRESENTATIONS

Nanoscopic Investigation of the Magnetoelectric Effect in Composite Multiferroic Ceramics

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Materials showing the magnetoelectric (ME) effect, i.e. a dependence of magnetization or polarization on an electric or magnetic field, respectively, have attracted significant attention due to a number of potential applications [1]. The large ME effect at room temperature has been observed in composite multiferroic materials that are combined of piezoelectric and magnetostrictive phases [2]. The ME effect in such case is mechanically mediated at the interfaces between the phases. It means that the macroscopic ME response depends strongly on the coupling at the local scale in vicinity of the interfaces. Here we present an approach towards studying the magnetoelectric coupling at the nanoscale using scanning probe microscopy (SPM) methods.

We addressed both the direct and converse ME effect in multiferroic composites consisting on magnetostrictive hexaferrite ($BaFe_{12}O_{19}$) or spinel ferrite ($CoFe_2O_4$, $NiFe_2O_4$) phase and ferroelectric ($BaTiO_3$) phase. We studied the effect of a magnetic field on the local polarization and ferroelectric domain structure using piezoresponse force microscopy (PFM). We also looked on variation of the magnetic structure in an electric field by using magnetic force microscopy (MFM).

PFM revealed that the local piezoelectric coefficient is modulated by the magnetic field, which may be considered as the intrinsic ME effect. To map regularities of the local coupling the principal component analysis method was applied. Furthermore, we found that the magnetic field affects the polarization switching kinetics [3]. Strength of the local ME effect was maximal in the vicinity of the interfaces between piezoelectric and magnetostrictive phases, which agrees well with the strain–stress mediated mechanism of the ME coupling for these composites.

For the converse ME effect we observed that the electric poling leads to irreversible changes of the magnetic domain configurations. The effect depends on microstructure: the grain size distribution, homogeneity of the phase distributions, and relative grain orientations. In order to quantify the observed variation of the MFM contrast, an image-processing algorithm was developed, which yielded the field-induced net domain wall displacement and broadening/thinning. Analysis of the domain wall displacement revealed an hysteretic change in the magnetization, which could be rationalized by existence of localized concentration of defects.

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Electronic properties of a 2D electron gas at the interface between complex transition metal oxides

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For the paradigmatic oxide heterostructure with $LaAlO_3$ (LAO) thin films grown on $SrTiO_3$ (STO) substrates, distinct electronic phases have been extensively characterized at the LAO/STO interface: for LAO films with more than three layers and LaO termination towards the TiO_2 interface, a metallic state is formed in the STO layers next to the interface which becomes superconducting below a temperature scale of 300 mK [1, 2]. Strikingly, the superconducting state coexists with a magnetic state possibly formed in patches of an inhomogeneous interface state. The magnetism appears to be stable up to the room temperature but its origin has not been settled. It may be well related to oxygen vacancies which lead to an orbital reconstruction of nearby Ti-sites and generate a local triplet state [3].

In our work by means of *ab-initio* calculations within GGA+*U* approach we performed a systematic variation of the values of the Coulomb parameters applied to the Ti 3*d* and La 4*f* orbitals [4]. We put previous suggestions to include a large value for the La 4*f* states into perspective in order to shift levels to the higher energy and avoid spurious mixing of La 5*d* and 4*f* states. Our calculations provide deeper insight into the band gap landscape in the space spanned by these Coulomb parameters and the resulting complex interference effects. In addition, we identify important correlations between the local Coulomb interaction within the La 4*f* shell, the band gap, and the atomic displacements at the interface.

We demonstrated an impact of electron-donor defects (H-adatom, O-vacancy and also H-adatom+O-vacancy) in different concentration and located in different layers of LAO and STO slabs separately and in the heterostructure on the structural and electronic properties. We have shown that surface adsorbates shift the Fermi-level to the higher energy, which leads to a insulator-metal transition in a STO slab and in the LAO/STO heterostructure with three LAO overlayers, whereas a LAO slab undergoes a transition from semiconductor to insulator state. We addressed the defect profiles through the entire heterostructure and reconsider orbital reconstruction of the Ti 3*d* states.

As a continuation of the study of complex oxides we extended our research to pairs of manganites and ferroelectric compounds.

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Magnetic anisotropies and optical tuning of ferromagnetic resonance properties in epitaxial $Fe_2B/BiFeO_3$ heterostructure

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Nowadays contactless, fast, compact and low-power approaches for tuning of magnetic properties of ferromagnetic thin films are of great technological and scientific importance due to applications in information storage, sensor, spintronic and tunable microwave magnetic devices. In the talk the studies of the magnetic anisotropies and an optical tuning of ferromagnetic resonance (FMR) properties in epitaxial $Fe_2B/BiFeO_3$ heterostructure related to the photostriction in $BiFeO_3$will be presented.

$BiFeO_3$ layer (~ 45 nm) and Fe_2B layer (~ 20 nm) were deposited onto the $SrTiO_3$ (001) substrate by means of the pulsed laser deposition. Only (00*l*) diffraction peaks of $BiFeO_3$ and Fe_2B were observed in the X-ray diffraction pattern of the heterostructure, which indicates the epitaxial growth of the Fe_2B/BFO heterostructure on the $SrTiO_3$ (001). The magnetic anisotropies that determine the angular variation of the resonance field in the plane of the system are represented by the tetragonal four-fold and uniaxial terms for Fe_2B layer, uniaxial term for antiferromagnetic $BiFeO_3$ layer and the exchange coupling at the interface. It was found that both the exchange bias and the in-plane FMR resonance field of the heterostructure are strongly affected by the illumination with $\lambda = 405$ nm light.

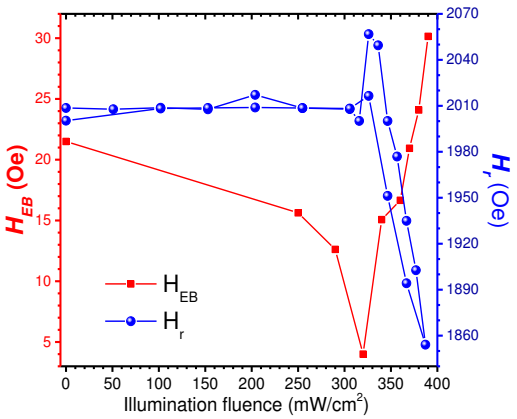

Fig. 1. Exchange bias and in-plane FMR resonance field dependences on light intensity.

The variation of exchange bias field, H_{EB} and the in-plane FMR resonance field, H_r, as a function of the illumination intensity are shown in Fig. 1. Clearly, critical illumination intensity for H_{EB} of ~ 320 mW/cm^2 is revealed. On increase of the illumination intensity causes H_{EB} decreases first, reaches its minimum at the critical intensity, and increases on further intensity increase. The H_r stays almost constant with the light intensity below the critical value and decreases on further increase of the intensity. These properties in our opinion are related to the photostriction in the $BiFeO_3$ layer. It has been reported that under 405 nm illumination the (001) BFO film undergoes a tensile deformation along the out-of-plane direction (001) accompanied by the in-plane compressive deformation [1]. Strain in the $BiFeO_3$ layer can modify the in-plane magnetization of this layer [2]. Exchange bias field depends on the magnetization of the antiferromagnetic layer. Therefore the change of the BFO antiferromagnetic magnetization due to the photoinduced strain can affect the exchange bias field and the in-plane FMR field.

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Polarazed Effects in La Doped $TlInS_2$ Ferroelectric – Semiconductor

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Lanthanum - doped high quality $TlInS_2$ ($TlInS_2$:La) ferroelectric - semiconductor was characterized by photo - induced current transient spectroscopy (PICTS). Different impurity centers are resolved and identified. Analyses of the experimental date were performed in order to determine the characteristic parameters of the extrinsic and intrinsic defects. The energies and capturing cross section of deep traps were obtained by using the heating rate method. The observed changes in the Thermally Stimulated Depolarization Currents (TSDC) near the phase transition points in $TlInS_2$:La ferroelectric - semiconductor are interpreted as a result of self - polarization of the crystal due to the internal electric field caused by charged defects. The TSDC spectra show the depolarization peaks which are attributed to defects of dipolar origin. These peaks provide important information on the defect structure and localized energy states in $TlInS_2$:La. Thermal treatments of $TlInS_2$:La under an external electric field, which was applied at different temperatures allowed us to identify a peak in TSDC which was originated from La – dopant. It was established that deep energy level trap BTE43 which are active at low temperature ($T \leq 156$ K) and have activation energy ~ 0.29 eV and the capture cross section 2.2×10^{-14} cm^2 corresponds to the La dopant. According to the PICTS results the deep level trap center B5 is activated in the temperature region of incommensurate (IC) phases of $TlInS_2$:La, having the giant static dielectric constant due to the structural disorders. From the PICTS simulation results for B5, native deep level trap having an activation energy of ~ 0.3 eV and the capture cross section of 1.8×10^{-16} cm^2 were established. A substantial amount of residual space charges is trapped by the deep level localized energy states of B5 in IC – phase. While the external electric field is applied, permanent dipoles, which are originated from the charged B5 deep level defects are aligned in the direction of the applied electric field and the equilibrium polarization can be reached in a relatively short time. When the polarization field is maintained while cooling the temperature of sample to a sufficiently low degrees, the relaxation times of the aligned dipoles drastically increases. Practically frozen internal electric field or electrets states remain inside the $TlInS_2$:La when the applied bias field is switched off. The influence of deep level defects on TSDC spectra of $TlInS_2$:La has been revealed for the first time.

Dielectric hysteresis loops of pure and lanthanum doped $TlInS_2$ ferroelectric – semiconductors were studied at the frequency 50 Hz for different temperatures below the Curie temperature (T_c). It has been revealed that, without any poling procedure, pure $TlInS_2$ exhibits normal single hysteresis loops at $T < T_c$. After electric field - cooled treatment of $TlInS_2$ the shape of hysteresis loops was strongly affected by corresponding charged deep level defects which were previously activated during poling process. As a result, an additional defect polarization state from space charges accumulated on the intrinsic deep level defects has been revealed in pure $TlInS_2$ at the temperatures below T_c. Besides, unusual multiple hysteresis loops were observed in La doped $TlInS_2$ at $T < T_c$ after application of different external perturbations (electric field, exposition and memory effect) to the sample. Measurements of the hysteresis loops in $TlInS_2$:La revealed the slim single, double and even triple - like the polarization – electric field ($P - E$) hysteresis loops. This intriguing phenomenon is attributed to the domain pinning by photo – and electrically active La – impurity centers. The temperature dependences of double - hysteresis - like loops on were also investigated. Due to the heat elimination of the random local defect polar moments, the double – hysteresis - like loops was transformed into a normal single hysteresis loops on increasing the temperature.

Phase Transitions in Nanocomposite Multiferroics Derived Using Ion Implantation Technique

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Multiferroic materials, which exhibit ferromagnetism and ferroelectricity in the same structural phase with strong magnetoelectric coupling have been a great interest in recent decades due to their various physical properties and wide application potential in the next generation of multifunctional electronic devices [1, 2]. In these materials the coupling interaction between ferroelectric and ferromagnetic substances could produce a magnetoelectric effect in which change in magnetization is induced by an electric field or the change in electric polarization is induced by an applied magnetic field. Historically, very few single phase multiferroic materials had been known and the magnetoelectric coupling in them was too small to be useful. On the contrary, it has been obtained that strong magnetoelectric effect via strain mediated interactions could be realized in the composites consisting of magnetostrictive and piezoelectric substances.

In recent years we suggested that an ion implantation is a very useful and effective technique for preparation of nanoparticulate multiferroic composite structures on the base of ferroelectric $BaTiO_3$, $TlInS_2$ and $TlGaSe_2$ crystals. In this work we report the results of investigations of the succession of the ferroelectric phase transitions in these structures and the study of the influence of external magnetic field on these structural transformations.

The samples were prepared by implantation with 40 keV Co^+ and Fe^+ ions into single crystalline plates of $BaTiO_3$, $TlInS_2$ and $TlGaSe_2$ at the fluencies between 0.5×10^{17} and 1.5×10^{17} ion/cm^2 and with ion current density of 8 μA/cm^2. The implantation was carried out at room temperature and residual vacuum of 10^{-5} Torr by using the ion-beam accelerator *ILU*-3 (Kazan Physical-Technical Institute of RAS).

Then the temperature dependences of the dielectric susceptibility and magnetization of the samples were measured. It has been revealed that the formation of metal nanoparticulate composite layer in the near-surface irradiated region in a result of high-fluency ion implantation brings to considerable shifts of well-known successive structural phase transition points and increase of the temperature hysteresis of the dielectric susceptibility. The application of the magnetic field in the direction perpendicular to implanted surface resulted to reverse shifts of the phase transition points. Additionally, the influence of the structural phase transitions of the ferroelectric substrate on the magnetic moment of the ferromagnetic layer has been revealed.

The observed peculiarities are considered as magnetoelectric effects, which appeared in a result of magnetoelectric lock-in interaction between domains of ferroelectric and ferromagnetic substances of the composite structure.

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EPR of the Local Magnetic Moments in the undoped Bi_2Te_2Se

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Local magnetic moments in topological insulators (TI) are useful for studying electromagnetic state of TI: they can play a role of a local spin probe. One can expect that correlation between magnetic moments doped in TI can break time reversal symmetry of TI and affect electromagnetic properties of a conducting surface layer. Therefore, studying behavior of local magnetic moments in TI is a subject of great importance.

Commonly term "magnetic TI" implies a TI compound doped with magnetic ions. However, it was discovered that local magnetic moments in topological insulators can originate not solely from doped magnetic ions but also from structural imperfections of undoped compound[1-2]. In case of Bi_2Te_3 and Bi_2Se_3 these local moments appear due to the substitutional defects in Se(Te) layer of typical quintuple structure: some of Se(Te) atoms are replaced by Bi atoms. Consequent local magnetic moment equals $0.6\mu_B$ and it arises from p-orbital of guest Bi atom. Unfortunately, there is a lack of experimental data on such magnetic moments. Our work is concentrated on studying their properties in Bi_2Te_2Se by EPR methods.

We observed the magnetic resonance signal of the local magnetic moments in Bi_2Te_2Se, that were apparently caused by substitutional Bi atoms in the central Se layer of the quintuple structure. The characteristic spectrum is shown in Figure 1. The temperature dependences of signal parameters were studied. The sample magnetization was measured by the homemade SQUID magnetometer.

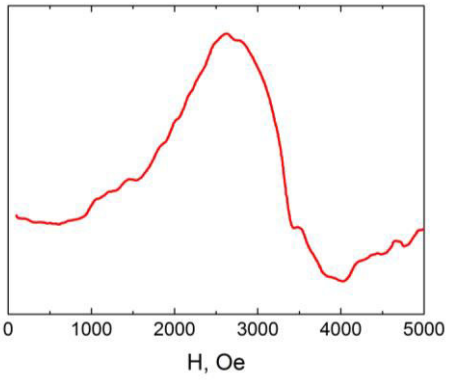

Fig. 1. EPR spectrum of Bi_2Te_2Se, T=15K, magnetic field parallel to the sample surface, applied microwave power 80 mW.

The EPR signal was observed at the temperature range from 10 K to 125 K. The amplitude of the signal was increasing with lowering temperature. The signal position is constant in whole temperature range. We estimated the symmetry of the line shape. The signal is almost symmetrical at high temperatures and drastically becomes asymmetrical at 20 K. The integral intensity of the EPR signal slightly grows while decreasing temperature from 125 K to 40 K, it decreases at 40 K>T>20 K and has a sharp rise at 20 K. Temperature dependence of the sample magnetization obtained by SQUID coincides with the temperature dependence of integral intensity: it has local maximum at 40 K. Such behavior can be a manifestation of spin-glass state. Furthermore, EPR signal has apparent angle dependence: while its position remains fixed, width and integral intensity have local extrema at Θ=0° (external magnetic field parallel to sample surface) and Θ=90° (magnetic field normal to sample surface).

The experimental results obtained demonstrate possibility of future implementation of magnetic resonance technique to control electronic state of TI surface layer.

The work was supported by RFBR through the grant № 15-42-02477.

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Study of domain formation in congruent lithium tantalate single crystals induced by ion beam irradiation

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The domain formation induced by focused ion beam irradiation has been studied in congruent lithium tantalate single crystals. The obtained results have been used for periodical poling with short periods.

We have investigated Z-cut optical grade 0.5-mm-thick plates of congruent lithium tantalate (CLT). Irradiated Z^+ polar surface of CLT wafers was covered by 500-nm-thick resist layer. Opposite Z^- surface was covered by solid Cu electrode, which was grounded during irradiation. Irradiation was performed by Auriga Crossbeam Workstation (Carl Zeiss). Exposure parameters and beam positioning were controlled by ion-beam-lithography system Elphy Multibeam (Raith) [1, 2]. The obtained domain structures were visualized by optical microscopy and atomic force microscopy after selective chemical etching, and by piezoresponse force microscopy and confocal Raman microscopy (CRM) without etching.

The formation of circular isolated domains at the Z^+ polar surface was revealed. The domains grew through the wafer and appeared at an opposite surface with rounded triangle shape. The exceeding of the threshold dose led to lose of domain wall shape stability which appeared as a rough domain wall with oriented domain rays at the domain wall predominantly at the upper side of circle domains. The domain rays were oriented along Y-crystallographic directions. It was shown that the sizes of isolated domains linearly increase with dose. Such dependence is typical for polarization reversal by conductive tip of scanning probe microscope [3]. The change of the domain shape with a depth from circular to triangular revealed by CRM measurements was explained in terms of the kinetic approach [4]. The interior structure of isolated domains was revealed by selective chemical etching.

The interaction between neighboring domains in the gratings has been studied. It was shown that the difference between the largest and the smallest domain decrease with increase of dot array period. This effect was attributed to contribution of total electric filed produced by injected charges of whole array which decrease with increase of the distances between irradiated dots.

The obtained knowledge allowed optimization of the periodical poling process and creating of the 2-μm-period stripe domain structure with high quality and homogeneity.

The equipment of the Ural Center for Shared Use “Modern nanotechnology” UrFU was used. The research was made possible in part by Government of the Russian Federation (Act 211, Agreement 02.A03.21.0006) and by President of Russian Federation grant for young scientists (Contract 14.Y30.16.8441-MK).

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Giant dielectric permittivity and magneto-capacitance effects in low doped manganites

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The importance of the inhomogeneous phase-segregated states in the explanation of the anomalous transport and magnetic properties in manganites and in high-temperature superconductors (HTS) has been widely discussed. In doped manganites, the complex interactions between different degrees of freedom lead to unusual magnetic and transport properties. Moreover, it has been suggested [1] that the spiral magnetic order observed in undoped manganites ReMnO3 may lead to multiferroic behavior. It has also been suggested [2] that the charge ordering in magnetic systems may cause the magnetoelectric effect.

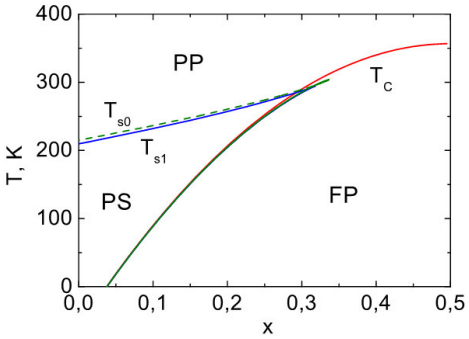

Fig. 1. Phase diagram of the system. PP is the region of the stable high-temperature phase; FP is the region of the stable ferromagnetic low-temperature phase; and PS is the region of the stable inhomogeneous phase and the metastable homogeneous high-temperature phase.

The effect of giant dielectric permittivity due to phase separation accompanied by charged inhomogeneities in low-doped manganites is discussed. The effect appears in the vicinity of the second-order magnetic phase transition and is caused by long-range Coulomb forces. The long-range Coulomb interaction is responsible for the formation of inhomogeneous charged states and determines their characteristic length scales. We derive the phase diagram of the inhomogeneous charged states in the framework of the phenomenological theory of phase transitions (Fig.1) [3]. The large value of the static dielectric function reduces the characteristic value of the Coulomb energy of the inhomogeneous state and makes the appearance of the magnetoelectric effect possible. We discuss the formation of a state with giant dielectric permittivity and magneto-capacitance effects in that case.

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Role of the Jahn-Teller Structural Ions in Formation Excellent Physical Properties of Transition Metal Oxides

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Complex transition metal oxides (ABO_3) span a wide range of crystalline structures. Lithium niobate and tantalate are known to have exceptional ferroelectric, pyroelectric, piezoelectric, elastic and nonlinear optic properties [1, 2]. Another well-known class of such oxides – ABO_3 – $La_{1-x}Sr_xMnO_3$ is the classical example of the strong magneto-elastic system, on which certain original ideas were tested [3].

The origins of these unusual physical properties are the noncentrosymmetric crystal structures at low temperatures. The phase transitions to noncentrosymmetric structures is considered to be induced by Jahn-Teller effect of the NbO_6, TaO_6 or MnO_6 octahedral units in $LiNbO_3$, $LiTaO_3$ and in $La_{1-x}Sr_xMnO_3$ respectively [4, 5].

Elastic and ferroelectric characteristics of $LiNbO_3$ and $LiTaO_3$ crystals were investigated in a wide range of temperatures by complex acousto-optic means. The contribution from Jahn-Teller NbO_6 and TaO_6 systems to the characteristics of elastic moduli, ultrasonic attenuation and nonlinear optical coefficients is analyzed using a new phenomenological model. It is hypothesized that the displacement of Nb^{5+}, Ta^{5+} and Mn^{3+} ions along trigonal axis $\bar{C}$ and the subsequent ordering of octahedral, result in unusual elastic and ferroelectric or magnetic (manganites) properties [6].

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Structural and Magnetic Phase Transitions in Multiferroic $RFe_3(BO_3)_4$ Compounds

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This presentation is a brief review of the results on the studies of phase trnsitions in rare-earth (RE) iron borates with huntite structure, performed in our laboratory in collaboration with other Russian and foreign institutions.

Multiferroic RE iron borates $RFe_3(BO_3)_4$ (R = Pr - Er or yttrium) crystallize in a noncentrosymmetric trigonal structure of the natural mineral huntite (space symmetry group $R32$, №155). These compounds demonstrate a rich variety of magnetic, magnetoelectric, magnetoelastic, magnetodielectric, and optical properties and phenomena, depending on a particular R element. $RFe_3(BO_3)_4$ with smaller than Sm^{3+} ions undergo a weak first-order structural phase transition at the temperature T_S, into also trigonal but less symmetric phase corresponding to the enantiomorphic space-group pair $P3_121$ (№152) and $P3_221$ (№154) [1,2]. New phonon modes that appear below T_S exhibit a hysteretic behavior of intensities indicative of a first-order phase transition and, simultaneously, a soft-mode-like behavior typical for a second-order phase transition [1, 2]. Giant changes of the dielectric constant accompany this structural phase transition [1]. The temperature dependences of frequencies, oscillator strengths, and damping constants of some low-frequency modes in Gd and Tb iron borates reveal an appreciable lattice anharmonicity [2].

We detect a strong effect of impurities (that enter the crystal from a flux in the course of the crystal growth) on the structural phase transition temperature and demonstrate a coexistence of both $R32$ and $P3_121$ ($P3_221$) phases down to the lowest temperatures in a $EuFe_3(BO_3)_4$ crystal grown with the $Bi_2Mo_3O_{12}$ based flux, due to inhomogeneous distribution of impurity Bi^{3+} ions. We have demonstrated suppression of the structural phase transition in the $Eu_{0.85}La_{0.15}Fe_3(BO_3)_4$ crystal and a possibility to control the temperature of the structural phase transition in RE iron borates, which implies an application potential [3].

All RE iron borates undergo an antiferromagnetic phase transition at temperatures that linearly grow with diminishing the ionic radius of R^{3+}. Iron borates of Gd and Ho exhibit also a spin-reorientational phase transition at low temperature. We have recorded spectral signatures of interactions between the lattice, magnetic, and electronic degrees of freedom in RE iron borates, which is characteristic of multiferroics. In particular, peculiarities in the phonon mode behavior at the temperature of an antiferromagnetic ordering were observed and explained by the spin-phonon interaction and an enhancement of anharmonicity due to magnetic ordering [2].

I would like to thank my coauthors of Refs. [1-3] who contributed decisively into the presented study. This research was supported by the Russian Science Foundation under Grant № 14-12-01033.

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Elastic Properties of PMN Single Crystal at Cryogenic Temperatures

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A prototype relaxor ferroelectric $PbMg_{1/3}Nb_{2/3}O_3$ (PMN) with disordering in B-position of the perovskite lattice ABO_3 is considered as a model for the study of the physical properties of relaxors. According to current models of relaxors based on the results of many research groups the extraordinary physical properties that underlie many applications, are determined by the existence of the natural nanostructure - polar nanoregions (PNR) or polar clusters in the paraelectric matrix - in such substances. Physical properties of PMN at cryogenic temperatures were investigated very scarce [1], in contrast to numerous studies of these properties in the dielectric relaxation temperature range around T_m – temperatures of the maxima of the dielectric constants. The unique feature of PMN is to preserve the cubic average structure Pm3m from paraelectric phase down to low-temperature relaxor phase which includes about 20% of polar nanoregions with a rhombohedral structure at cryogenic temperatures [2]. This makes PMN a very interesting and appropriate subject to study.

The study is focused on the temperature evolution of the longitudinal V_L (LA) and shear V_S (SA) ultrasonic wave velocities propagating along [100] and [110] directions at temperatures down to 4.2 K. The ultrasonic velocity measurements were performed with a RITEC Advanced Ultrasonic Measurement System RAM-5000 with an accuracy of 10^{-4}. The experiment was carried out in Oxford Instruments continuous flow cryostat in the temperature range from 300 K to 4.2 K.

Temperature dependences of all elastic constants for the cubic structure (C_{11}, C_{44} and C_{12}) were obtained for the first time from the velocity data set. Temperature dependences of longitudinal and shear elastic constants (C_{11} and C_{44}, respectively) are in qualitative agreement with relations originating in an Einstein-oscillator model which applies to essentially all solid substances and are characterized by increasing and saturating at cryogenic temperatures. Elastic constant C_{12} shows abnormal monotonic decrease from inflection temperature $T_i \approx 220$ K upon cooling followed by saturation at $T < 25$ K. Temperature dependences of Bulk modulus B, degree of lattice anisotropy δ and anisotropy factor A are closely related with C_{12} temperature behavior and show monotonic decrease from T_i upon cooling with saturation at cryogenic temperatures. Specific features of PMN anharmonic properties such as additional electrostriction contribution to thermal expansion and nonlinear elastic constants are considered as possible reasons of the behavior.

Moreover, important PMN material parameters such as Debye temperature Θ, Grüneisen parameter H and Poisson ratio v are determined at cryogenic temperatures, as well.

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Magnetic bubble blowing with electric field: how to eliminate the nucleation barrier

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Recently discovered effect of electric field induced magnetic bubble domain nucleation and blowing in iron garnet films [1] brings new electrical dimension to the physics of micromagnetism and requires the revision of classical magnetic bubble theory [2].

In this work the result of experiments on iron garnet films are presented and the theory accounting for the role of electric field in the balance of domain wall surface energy, magnetostatic demagnetization energy and Zeeman external magnetic field term. We argue that inhomogeneous electric field of the tip electrode has two effects on the micromagnetic structure:

1) it decreases the effective surface tension of the domain wall and develops the radial instability (fig.1) leading to the nucleation of the magnetic bubble.
2) it acts with antiparallel forces on the opposite sides of the magnetic bubble thus inflating it to the radius higher than for zero-electric field case.

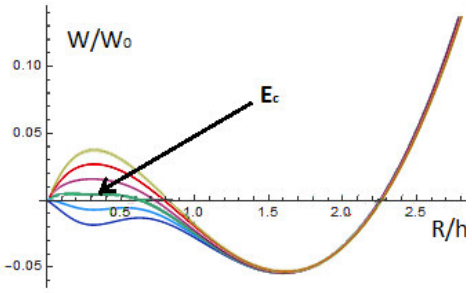

Fig. 1. The total energy dependence on the bubble domain radius in units of film thickness. Ec is critical electric field of barrier lifting. From top to bottom electric field is: 0, 1/3 Ec, 2/3 Ec, Ec; 4/3Ec; 5/3 Ec. $W_o=2\pi M_s^2 h^3$.

The new way of electric field control of magnetic structure provides the viable alternative to the recently reported bubble and skyrmion blowing methods [3-5] that suffers either from ultra-high density of spin polarized current [3] or low temperatures [4] or sporadic character of bubble nucleation process [5].

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Inhomogeneity of the magnetic state in the $Pr_{1-x}Sr_xMnO_3$/YSZ films studied by electron magnetic resonance

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Polycrystalline films $Pr_{0.8}Sr_{0.2}MnO_3$ (d = 20-150 nm) and $Pr_{0.6}Sr_{0.4}MnO_3$ (d = 20-130 nm) were prepared by dc magnetron sputtering with the "facing-target" scheme [1], that allows transferring elements from a target to a substrate without changes in the composition. Single-crystal zirconium oxide stabilized by yttrium (YSZ) was used as the substrates. The study of the magnetic properties of the films showed a low value of Curie temperature (T_C) of the samples and a complex pattern of the magnetic inhomogeneity above T_C (Fig. 1).

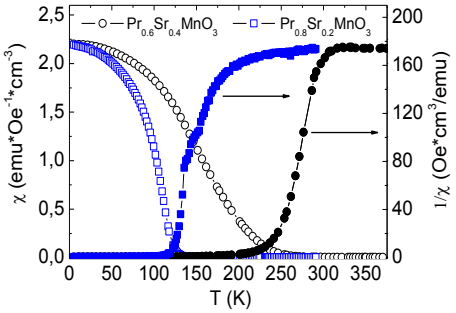

Fig. 1. Temperature variation of magnetic susceptibility and inverse magnetic susceptibility for the studied samples at H = 100 Oe.

The temperature dependences of the electron magnetic resonance (EMR) spectra for the $Pr_{1-x}Sr_xMnO_3$/YSZ films were studied in the X band at two mutually perpendicular magnetic field orientations relative to the films plane and temperatures from T_C to 350 K. For the $Pr_{0.6}Sr_{0.4}MnO_3$ films, two temperature ranges was revealed indicating on two different magnetic states in the samples: $T_C < T < T_X$ and $T > T_X$, where T_X is a temperature close to the Curie temperature of bulk samples of the same composition, 300 K [2]. For the $Pr_{0.8}Sr_{0.2}MnO_3$ films, three temperature regions were revealed: $T_C < T < T_X$, $T_X < T < 200$ K, and $T > 200$ K, where $T_X \sim 150$ K [3].

The temperature dependences of the effective g-value, the line width, the intensity line, and the angular variation of the resonance fields were investigated. The experimental observation in the ranges of $T_C < T < T_X$ is discussed within the framework of the Griffiths theory that predicts the existence of ferromagnetic clusters above T_C [4]. The presence of a magnetic phase in the samples at high temperature (Fig. 1) suggests the formation of an interface layer at the boundary film-substrate. The possible formation of an interface in the samples is also considered.

The work was supported by the Russian Foundation for Basic Researches, Grant No. 16-32-00209 mol_a and the Grant of the President of the Russian Federation no. NSh-7559.2016.2.

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Transient response of ferroelectric capacitor with negative capacitance to piecewise constant voltage pulses

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Nowadays all over the world research activity in the sphere of integrated ferroelectrics is very intensive [1]. The report presented follows to mainstream of this interdisciplinary scientific direction namely applications of thermodynamically stable negative capacitance effect in a nanoscale bilayer of ferroelectric and nonlinear dielectric at room temperature are under our investigation. Experimental observation of this effect was first described in article [2]. We call such bilayer ferroelectric-dielectric systems with negative capacitance by 'ferroelectric negative capacitors' (FNC).

We consider series combination of FNC and a resistor R under the action of arbitrary piecewise constant input voltage U(t). This electric circuit is shown in Fig. 1.

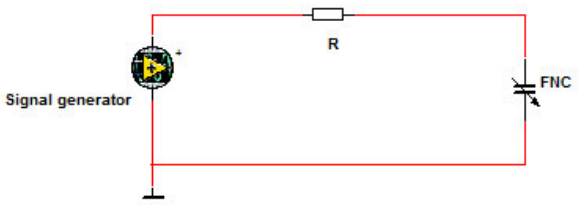

Fig. 1. Series combination of FNC and resistor R under the action of input voltage

In order to construct transient response of the device on Fig.1 we have found the exact solution of the next ordinary differential equation:

$$R \cdot \frac{dQ}{dt} - \alpha \cdot Q + \beta \cdot Q^3 = U_0, \qquad (1)$$

where Q is charge of FNC, U_0 is real constant and positive values α and β ought to be determined from the Landau-Ginzburg-Devonshire expression for free energy density of ferroelectric and from the geometry of FNC. It is obvious that equation (1) is just the Kirchhoff's voltage law for the circuit presented on Fig. 1.

Our solution is suitable for real circuit design applications. In particular we have calculated transient response of our system to a number of piecewise constant ultrawideband signals proposed in paper [3]. The simplest of them is equal to:

$$U(t) = U_0 \cdot (-\theta(t) - \theta(t-T) + 2 \cdot \theta(t - T/2)),$$

where θ(t) is the well-known Heaviside function and T is duration of the signal.

The results obtained can be extended to the taking into account the 6th power of polarization in the expansion of free energy density of ferroelectric in FNC.

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POSTER SESSION

Explosive laser-induced crystallization of ferroelectric microstructures

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Explosive crystallization is an interesting crystallization regime from the point of view of kinetics [1, 2]. In this case, during the movement of the crystallization front, latent heat that supports the front movement is released. If crystallization occurs in a film, the stresses arising in it can have a significant effect on the process. This is shown in our experiment on local laser crystallization of ferroelectric precursor films by femtosecond pulses [3]. Stresses have arisen at the film/metallized substrate boundary due to the difference in the coefficients of thermal expansion. This is seen from symmetrical film exfoliating on both sides of the heated region, Fig. Authors thank O.M. Zhigalina, Crystallography inst. for this investigation. Due to these stresses, the crystallization began from a free surface, and not from the film-metal interface, on which the laser radiation was absorbed. For a theoretical description, a model for explosive crystallization was used [4]. The experimental parameters of crystallization (radius of crystallized regions) fall into agreement with the model. The crystallization front velocity, which depends on the distance from the center, was calculated. The laser annealing method can be used to obtain both local ferroelectric regions in a quasi-amorphous environment and more complex structures, for example, waveguides. Such structures can be used in MEMS technologies, photonics.

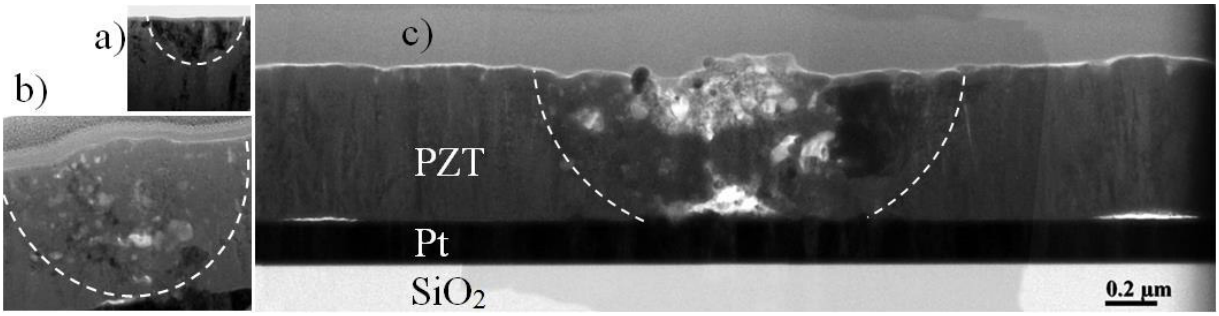

Fig. Transmission electron microscopy. Cross sections of crystallized regions with different exposition durations. a - 0.1 s, b - 4 s, c - 11 s.

Due to the calculations made, it is possible to obtain ferroelectric structures with a predetermined size up to hundreds of nanometers.

Nonlinear optical diagnostics of functional regions showed the distribution of the ferroelectric phase. Optimal crystallization modes (duration and power density of laser radiation) determined from nonlinear optical images to obtain structures with better nonlinear optical properties. Piezo-forced microscopy was also performed, which showed the polarization switching in the obtained structures.

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Influence of the thermal treatment on dielectric properties of $BaTiO_3$ nanoparticles

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Ferroelectricity does not appear in ultrafine particles due to the so-called "size effect". Accordingly to [1], the critical size of $BaTiO_3$ nanoparticles varies from 9 nm to more than 100 nm, depending on the method of their synthesis. The difference in the values of the critical size are caused by the presence of different defects and different their concentration.

Thermal treatment in air atmosphere may lead to the lattice defects disappearance that results in an emergence of ferroelectric properties in nanoparticles. Samples in the form of tables were prepared by compacting of barium titanate powder (Sigma-Aldrich, USA). Initial particles of cubic perovskite modification of $BaTiO_3$ had a spherical shape with average diameter 100 nm. Obtained samples were annealed in the electrical resistive furnace at air atmosphere under the following scheme: 700 °C (1 h.) → 1000 °C (5 h.) → 1200 °C (1 h.).

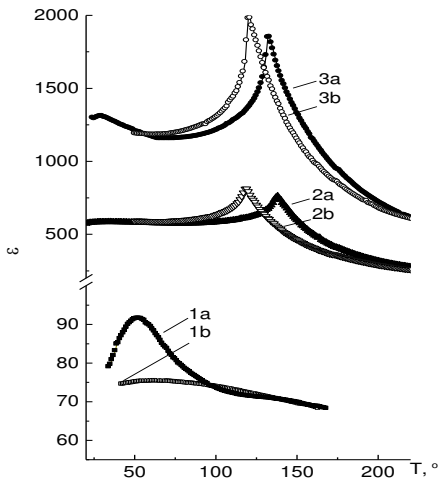

Fig. 1. Temperature dependences of ε observed at 10 kHz during heating (1a – 3a) and cooling (1b -3b) samples of $BaTiO_3$ nanoparticles, annealed previously at 700 (1a and 1b), 1000 (2a and 2b) and 1200 ° C (3a and 3b).

Analysis of the experimental results allows the following conclusions:

1. Thermal annealing of $BaTiO_3$ nanoparticles with initially cubic perovskite crystalline lattice at 1000 °C during 5 h leads to the formation of ferroelectric tetragonal phase. It was found that the increase of tetragonal distortions increase simultaneously with the annealing temperature (c/a = 1,0085 and 1,00976 for the samples annealed at 1000 and 1200 °C respectively).

2. Heat treatment of $BaTiO_3$ nanoparticles under experimental conditions does not lead to visible increasing of their sizes. Thus, appearance of ferroelectricity in $BaTiO_3$ nanoparticles is not produced by the increase of their sizes, but caused by the lattice defects concentration decrease owing to thermal annealing.

3. Temperature hysteresis of dielectric permittivity observed at cyclic temperature variation in the vicinity of Curie temperature indicates the first order phase transition in the annealed $BaTiO_3$ nanoparticles (Fig. 1). The width of thermal hysteresis decreases after high-temperature annealing of the sample. It shows that the anomalously broad hysteresis of the dielectric permittivity emerges due to an interaction of interphase boundaries and "random field" type lattice defects.

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Magnetic properties of heterogeneous materials based on manganites

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Here we present the investigation of microstructural and magnetic properties of (80-x)%($La_{0.7}Sr_{0.3}MnO_3$)+20%GeO_2+x%NaF (LSMO/GeO/NaF) composite materials for x = 0-0.20. Synthesis technology and magnetoresistive properties of composites based on lanthanum-strontium manganite without sodium fluoride are described in [1]. For the preparation of fluorinated compounds 5-20 weight percent of NaF was added in the stoichiometric mixture of La_2O_3, $SrCO_3$, Mn_2O_3, GeO_2 at the percolation threshold [1]. Composites containing NaF mainly consist of hexahedral hollow tubes and demonstrate more regular microstructure than composites with x=0 and (Fig. 1). The addition of NaF to composite also leads to the increasing of the absolute value of the magnetization (Fig. 2), which demonstrates the superparamagnetic behavior. We suggest that the replacement of the manganese ions of fluorine leads to the change in the parameters of the double exchange and improves the ferromagnetic properties.

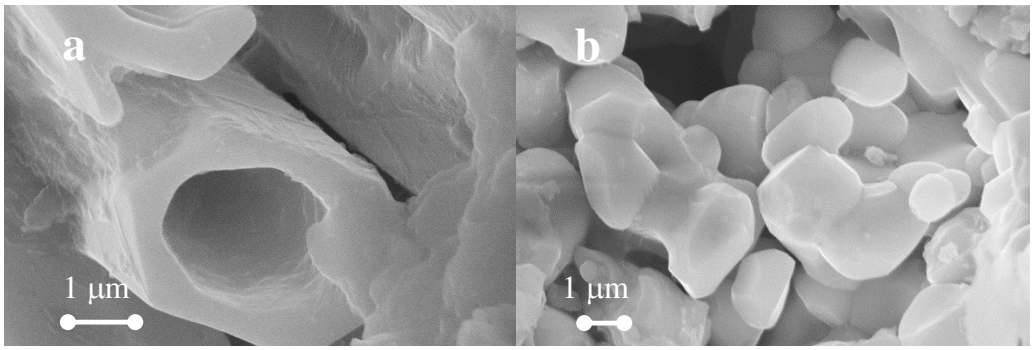

Fig. 1. SEM images of LSMO/GeO/NaF composites: (a) x=0.1, (b) x=0.

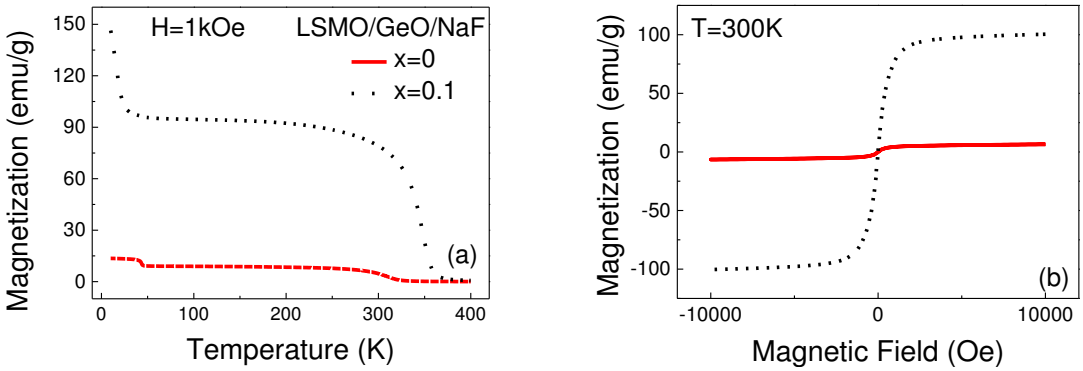

Fig. 2. (a) Temperature dependence and (b) field dependence of magnetization in LSMO/GeO/NaF

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Microwave absorption study of superconducting fluctuations in $Bi_2Sr_2Ca_{1-x}Y_xCu_2O_{8+\delta}$ crystals

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Pseudogap state of high-temperature superconducting material is studied now in many laboratories around the world [1]. Without understanding the nature of the pseudogap, it is impossible to establish a mechanism for high-temperature superconductors and to continue improving the critical parameters of these materials. Particular attention is paid to the region of the phase diagram bordering with the superconducting region, where the effect of fluctuations of the superconducting order parameter has a great influence on all electronic and magnetic properties. In addition, the problem of the location of the fluctuation region and its relationship to the pseudogap state are still open and require further investigation using a new technique.

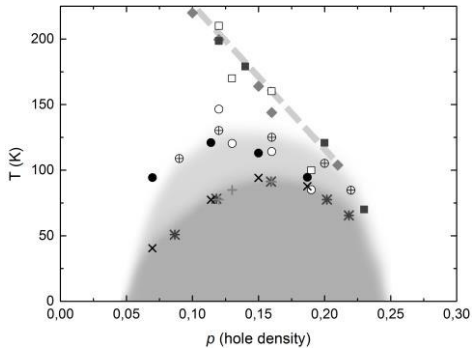

Fig. 1. Phase diagram of the $Bi_2Sr_2Ca_1Cu_2O_{8+\delta}$ compound.

In our work, we use several experimental methods (measurement of susceptibility, resistance and non-resonant microwave absorption (MWA)). The advantage of MWA method is the high sensitivity to the magnetic particles and excitations. The objects of investigation were single crystals of $Bi_2Sr_2CaCu_2O_{8+\delta}$ doping with yttrium. This doping allows us to change the concentration of current carriers (holes).

The results obtained are presented in the "temperature – hole density" phase diagram (Fig. 1). This figure shows that for underdoped samples the superconducting fluctuations have a wide temperature range. This range decreases upon the hole density increase and becomes zero for the overdoped sample. Our estimates are in good agreement with the data of ARPES [3] and STS [4], but deviate from measurements of the Nernst effect [2] in the region of high hole density. The region of a pseudogap state has the same property. This indicates a correlation between these two states. The MWA measurements of the $Ba(Fe_{0.9}Co_{0.1})_2As_2$, which were also carried out in this work, sample confirm this assumption. These results allow us to assume the stimulating effect of the pseudogap on the superconducting fluctuations development. The detection of SCF using the MWA method indicates a fluctuation lifetime on order of 10^{-10} s or more.

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Magnetic properties of the two-phase composite system (1-*x*)$BiFeO_3$ – *x*$MgFe_2O_4$

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Currently $BiFeO_3$ is one of the most promising multiferroics. However, it has a significant drawback – it happens a spin-modulated ordering of the magnetic subsystem. This makes it difficult to practical use because of the very small value of the bulk magnetic moment. A number of different approaches was used for the destruction of the spin cycloid: a doping of the $BiFeO_3$ with various chemical elements, a creation of solid solutions, an application of a strong magnetic field, etc. However, these attempts have not resulted in a significant effect. In this work, another approach has been used: a family of two-phase ceramic composites (1-x) $BiFeO_3$ - x $MgFe_2O_4$ (x = 0.01; 0.05; 0.1; 0.15 and 0.2) was prepared using a standard ceramic technology. $MgFe_2O_4$ was taken as the second phase of the composite because we assumed that the magnetic ordering of the $MgFe_2O_4$ phase could affect the spin-modulated structure in $BiFeO_3$. Therefore, the aim of this work was to study the effect of $MgFe_2O_4$ phase on the magnetic properties of $BiFeO_3$. The analysis of the experimentally obtained magnetic hysteresis loops at various content x of the second component allowed us to plot dependencies of the magnetic momentum on x (fig. 1). As one can see, the magnitude of a magnetic response increases with x increasing. However, further researches are required to define of $BiFeO_3$ contribution to this response. The study of temperature dependences of the magnetic permeability (fig. 2) allowed us to determine a Neel temperature shift in $BiFeO_3$ phase toward to higher temperatures at x increasing. This feature can be explained by "chemical pressure" effect, which arises when the substitution of Bi^{3+} ions by of Mg^{2+} ions (with different ionic radii) occurs in the process of mutual diffusion during a high temperature sintering of ceramic samples.

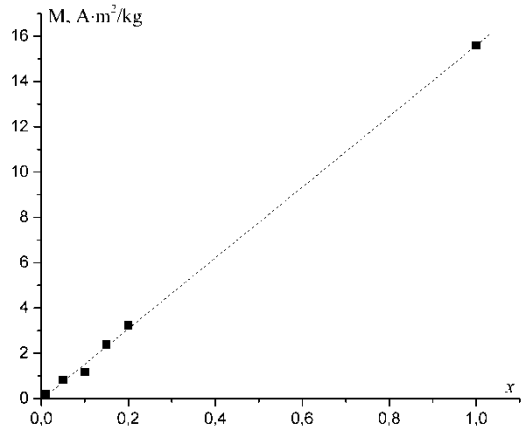

Fig. 1. Dependences of the magnetic momentum vs. $MgFe_2O_4$ concentration x at dc magnetic field 5 kOe.

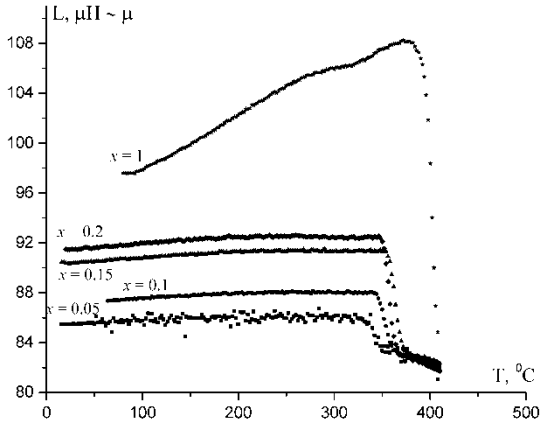

Fig. 2. Temperature dependences of the magnetic permeability of two-phase composite system (1-x) $BiFeO_3$ – x $MgFe_2O_4$.

The work was supported by the Russian Foundation for Basic Research (project No. 16-32-00697).

Temperature dependences of order parameter in nanocomposites porous glasses – sodium nitrite

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Temperature dependences of order parameter in nanocomposites (NCM) on the base of porous borosilicate glasses with average pore diameters 20 (NCM-WPG) and 46 (NCM-46) nm filled by sodium nitrite have been studied by synchrotron radiation diffraction. The average sizes of $NaNO_2$ nanoparticles in these matrices (Fig. 1) and the temperatures of ferroelectric phase transitions have been determined. From analysis of diffraction patterns the temperature dependences of order parameters η(T) on heating and cooling have been reconstructed (Fig. 2). It is shown that the ferroelectric phase transitions remain the first order transitions for sodium nitrite embedded into these matrices.

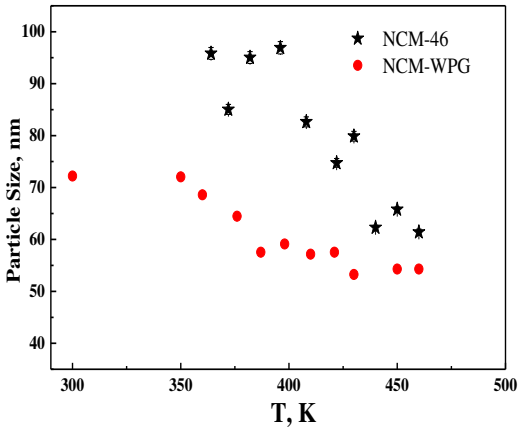

Fig. 1. Temperature dependences of average particle sizes of $NaNO_2$ embedded into NCM-46 and NCM-WPG

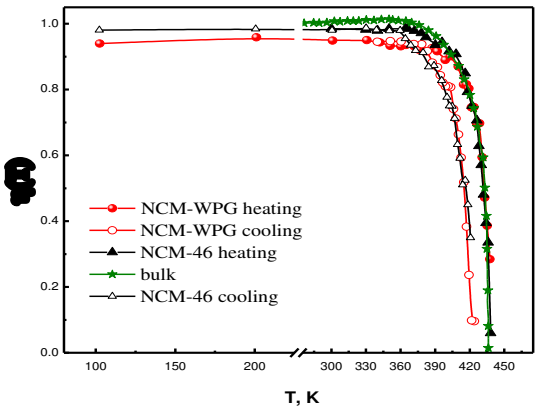

Fig. 2. Temperature dependences of order parameter for the bulk $NaNO_2$, and with $NaNO_2$ embedded into NCM -46 and NCM-WPG

At room temperature the values of order parameter for NCM-46+$NaNO_2$ and NCM-WPG+$NaNO_2$ are equal to 0.98(1) and 0.94(1) respectively. For NCM-WPG+$NaNO_2$ we have observed the hysteresis loop between "heating – cooling" with ΔT~10 K.

This work is supported by RFBR grant 15-02-01413.

Ferroelectric films and heterostructures, obtained by extraction-pyrolytic technique

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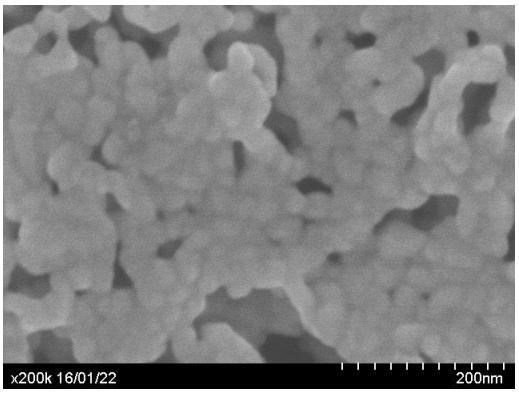
The properties of ferroelectric thin films are highly dependent on their structure (grain size, porosity, interfaces), which determined by the synthesis conditions. We have proposed a method for the preparation of ferroelectric films from the extracts solutions [1], which reduces the temperature parameters of films synthesis.

The XRD of pyrolysis products at different temperatures showed that monophasic $BaTiO_3$ film formed at 550 °C in the amorphous-crystalline state.The increasing of the annealing temperature leads to better crystallinity of the product. To form a complex oxide phase the high temperature annealing was conducted at temperatures of 500-700 °C. At the same time it should be noted that the solid-phase synthesis of barium titanate from the simple oxides takes place at temperatures of 1300 °C. The developed method enables not only decrease the temperature of synthesis but also the purity of the complex oxides.



However, even the high temperature annealing does not lead to a significant increase in particle size, as evidenced by broadened peaks, i.e. the product remains nanocrystalline. $BaTiO_3$ films were obtained in the nanocrystalline state at the annealing at 600 - 800 °C with grains size 20 nm.

Measurements of the physical parameters of the films showed the presence of a square hysteresis loop and high dielectric constants.

The films of barium titanate and barium strontium titanate used to create a heterostructure systems "porous silicon-ferroelectric" with high dielectric constant. The low-frequency dielectric constant of these structures generally exhibits relatively weak temperature dependency in the temperature range from 20 to 120 °C with a minor peak at a phase transition. The absolute value ε thus retain high values, which may be useful in practice.

The films of barium titanate were used to enhance the effect of light-collection and reduce recombination losses in Grettsel solar cells which made by films deposition on glass by applying extraction-pyrolytic method.

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Multifunctional properties of ferroics and multiferroics in conditions of photoexcitation

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The results of a theoretical investigation of the dynamics of states in ferroics and multiferroics arising from irradiation with light in the vicinity of phase transitions are presented. In the compounds of some ferroics and multiferroics, nearby the photostimulated phase transition, regions of phase coexistence arise, and also the emergence of unstable states. We investigated the appearance of a dynamic regime in ferroics and multiferroics nearby the phase transition under illumination conditions. To this end, a system of differential equations describing the change in the order parameter and the change in the number of electrons nearby the sticking zone is analyzed. It is shown that in the region of a phase transition under the conditions of a illumination, the existence of three stationary points on the phase plane corresponding to the equilibrium states of the system is possible. The corresponding values of the pairs of the order parameter and the concentration of charge carriers on the defects are obtained. The equations of motion of the system are written. The regions of the appearance of the dynamic regime in the material near the phase transition are obtained for a nontrivial case of the order parameter, called the hysteresis region. The intensity and temperature in the hysteresis region are estimated. The dynamics of the system in the space of the order parameter and the carrier density at the defects in the region of the phase transition are considered. A phase diagram of the various states of the system in the region of the phase transition in the parameter space is obtained in the coordinates of light intensity and temperature.

Ferroelectric phase transitions in nonstoichiometric sodium-bismuth titanate ceramics

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Strong demands on solving ecological problems stimulated investigations of lead-free oxides on the base of sodium-bismuth titanate $(Na_{0.5}Bi_{0.5})TiO_3$.

In this work, phase formation, structure, microstructure, dielectric and ferroelectric properties of the $(Na_{0.5+x}Bi_{0.5})TiO_3$ (I) and $(Na_{0.5}Bi_{0.5-x})TiO_3$ (II) (x=0 – 0.1) nonstoichiometric ceramics has been studied.

Ceramic samples were prepared by the two-step solid-state reaction method at temperatures T_1=1070 K (6 h) and T_2=1370 - 1470 K (2 h) using Na_2CO_3, Bi_2O_3 and TiO_2 ("pure" grades) as starting materials.

The samples were characterized using the X-ray Diffraction, Scanning Electron Microscopy, Second Harmonic Generation (SHG), and Dielectric Spectroscopy (in the frequency range of 100 Hz – 1 MHz and in the temperature range of 300 – 900 K) methods.

According to the X-Ray data dense single phase samples were received after sintering samples with x<0.075 at T_2=1450 - 1470 K (2 h). Compositions with higher x contained $Na_2Ti_6O_{13}$ as admixture phase.

Phase transitions marked by steps near ~400 K and by peaks at ~ 600 K were observed in the dielectric permittivity versus temperature curves of the NBT compositions prepared. Phase transitions at ~ 400 K demonstrated relaxor behavior attributed to the presence of polar nanoregions in a nonpolar matrix [1].

It was confirmed that Na-deficiency influences structure parameters and functional properties of nonstoichiometric NBT ceramics [2]. With increasing Na/Bi content increase in the spontaneous polarization value was proved using the SHG method. Increase in the $q=I_{2w}/I_{2w}(SiO_2)$ value from q~10 at Na/Bi<1.05 till q~130 at Na/Bi>1.2 points to improvement of dielectric and ferroelectric properties of the samples studied.

Acknowledgment
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Crystallophisic models of ferroelectrics type ABO_3 (A - Ba, Pb, Bi; B - Ti, Fe)

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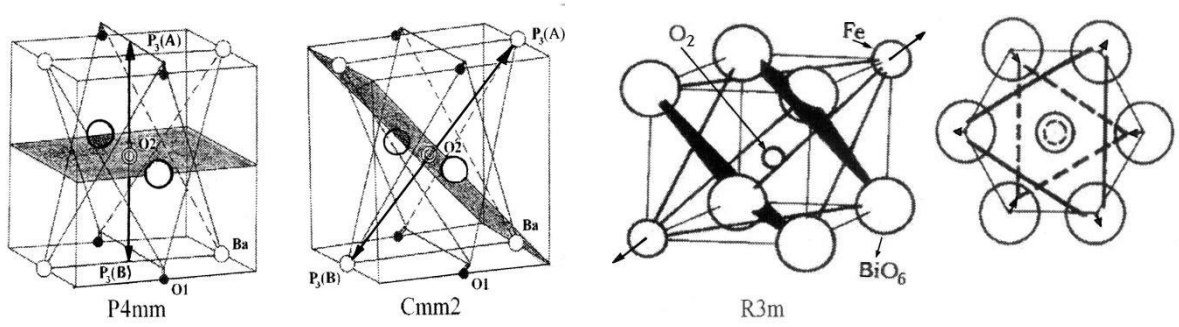
Physical properties of crystalline materials can be described using a theoretical approach, which is based on the conception of ideal crystal structure that obeys some symmetry rules. After all the simplifications, that are caused by the symmetry, a complete theory of an ideal crystal, which is a main subject of the solid-state physics, can never be built since it would be the theory of a great number of strongly interacting particles. Using current theory of the ideal crystal, some models have been creating that are based on crystal-chemical structures. Basic idea of such a construction is that several subsystems with finite number of joint degrees of freedom may be selected in the ideal crystal. It can be achieved to a first approximation under a crystal-physical approach [1] based on an expansion of 3 rd- rank polar tensor into the tensor subspaces weighing L=1,2,3 [2].

Tetrahedrons and their combinations can be considered as main structural elements of non-centrosymmetrical crystals. Such a look at the structure agrees with the crystal-physical approach because the tetrahedron gives most obvious idea of a loss of the inversion symmetry in crystals. Structural features of non-centrosymmetrical crystals, which are materialized in various combinations of the tetrahedrons, can be described taking some mathematical values into consideration type of **vectors, pseudodeviators and septors.** There are macroscope values of dipole, pseudo-quadrupole and octupole moments of non-centrosymmetrical crystals, which correspond to aforesaid elements. Results of the tensor expansion for ten pyroelectric classes give a chance to provide mathematical basis for the classification of the ferroelectrics on Abrahams-Keve [3]. Considering that the oxygen sites in the initial structure are split, following models are suggested of **$BaTiO_3$, $PbTiO_3$** and for **$BiFeO_3$.**



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Visualization of Initial Nanodomain Structures in Relaxor SBN Single Crystals and PLZT Ceramics

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Relaxor ferroelectrics are widely used in optical devices, actuators, sensors and modulators due to their outstanding electromechanical, piezoelectric and electrooptical properties [1]. These materials are disordered ferroelectrics with the maze initial nanodomain structures appeared after cooling below freezing temperature [2-4]. We have studied experimentally the structures appeared at different cooling rates and their dependence on compositions of the relaxor single crystals and ceramics.

We have studied as-grown domain structures and structures appeared after thermal depolarization (with various cooling rates) in $Sr_xBa_{1-x}Nb_2O_6$ (SBNx) single crystals and $Pb_{1-x}La_x(Zn_{0.65}Ti_{0.35})_{1-x/4}O_3$ (PLZT x/65/35) ceramics. SBN61 undoped and Ni doped (0.01 – 1 wt.% Ni_2O_3) single crystals were grown in Institute for General Physics of the Russian Academy of Sciences. PLZT x/65/35 ceramic samples (with x = 6-9 %) were produced in Institute of Solid State Physics of University of Latvia.

The depth dependence of the initial structures was studied by domain visualization using piezoresponse force microscopy (PFM) and scanning electron microscopy (SEM). Oblique cut, ion etching and ion beam polishing were used for investigation of the domain structures on the depth from the surface.

The nanodomain structures were characterized by fractal dimension, average period and correlation length. The dependences of the main parameters on the depth and cooling rate were obtained for different compositions in PLZT and for degree of doping in SBN.

The grain size and correlation length in different grains have been measured in PLZT ceramics. The dependence of the correlation length on the grain size was revealed. It was shown also that the averaged value of the piezoelectric response decreases upon approaching to the grain boundary.

The equipment of the Ural Center for Shared Use “Modern nanotechnology” UrFU was used. The research was made possible by Russian Foundation for Basic Research (Grant 16-02-00821-a).

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Low-frequency electrochemical strain microscopy in $LiMn_2O_4$

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Electrochemical strain microscopy (ESM) method gives unique possibility to inspect mobility of ions in the large amount of materials used in energy redistribution and storage. ESM basis is the registration of the local surface displacement appeared due the change of ion concentration per the Vegard law under the action of external electric field. Usually time spectroscopy is used for quantification of local diffusion coefficients and Li ion concentration [1]. In frame of this approach, the surface displacement relaxation is measured after the step-like electrical field pulse application. However, additional contributions appearing as results of electrostatic tip-surface interaction, electrostriction surface displacement and irreversible chemical reactions under the DC bias make difficult quantitatively interpret the ESM data [2].

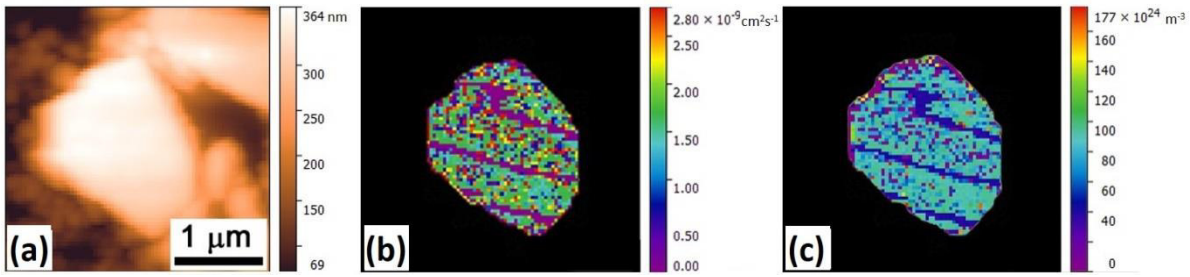

Fig. 1. ESM of LiMn2O4 cathodes: (a) topography, (b) local diffusion coefficient, (c) concentration of Li ions.

In this contribution, we assigned new method for the measurements of local diffusion coefficients and Li ion concentration from the frequency dependence of displacement signal. This method can be done in any electrochemical system with electrode non-blocking for electrons. The visualization of local diffusion coefficient and Li concentration distribution was realized by the analysis of ESM images measured at different frequencies of excitation voltage and approximation of the ESM signal frequency. We found that the Li ions distributed non-uniformly in the grain and prolong areas poor by lithium could be identified. This distribution can be due to macroscopic defects presenting in material. The values of average Li ion concentration 7.1×10^{25} m^{-3} and local diffusion coefficient 1.7×10^{-9} cm^2s^{-1} in the grain of $LiMn_2O_4$ were close to the values obtained by the macroscopic methods [3].

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Temperature dependence of Raman spectra and structure of δ-BiB_3O_6 Crystal

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Phase diagram of Bi_2O_3-B_2O_3system had been studied intensively in sixties [1], and crystals of bismuth triborate BiB_3O_6 were synthesized among them for the first time. This crystal attracted special interest of several scientific groups in the last years due to its high optical nonlinearity, that provide wide practical applications for it as an active medium in systems of optical frequencies transformations. For a long time only one α-phase was known for this composition, but six new phases of BiB_3O_6 have been found in the recent years [2]. However only α-phase has been investigated in details up to now. Its structure is built of chains of [BO_3] triangles and [BO_4] tetrahedrons in 1:2 ratio. First principles simulations of its electronic structure showed that [BO_4] tetrahedrons provide the main part of optical nonlinearity. Among other phases of BiB_3O_6 only γ and δ include [BO_4] tetrahedrons exclusively; and centrosymmetrical γ phase is of scanty interest for nonlinear optics, while structure of δ phase ($Pca2_1$, $Z = 4$) looks more attractive for such applications. Still physical properties of this phase have been studied rather poorly, and its vibrational spectra at different temperatures has never been investigated up to now. In this work we have performed such measurements.

To obtain Raman spectra Ar^+ polarized radiation at 514.5 nm were used as an excitation (Spectra-Physics Stabilite 2017, 100 mW output, 15 mW at the sample). Spectra in backscattering geometry were obtained with Horiba – Jobin Yvon T64000 spectrometer in 10 to 1600 cm^{-1} range. Their temperature dependence within 8–300 K range was investigated with ASR closed cycle cryostat CS204-X1.SS.

Raman spectra of δ-BiB_3O_6 phase has been obtained and their symmetry interpretation has been performed. Experimental data are compared with results of empirical lattice dynamics simulations for δ and α phases of BiB_3O_6, performed within LADY software [3]; special optimization software has been designed to determine empirical parameters of the model, taking into account lattice stability conditions. Using such models experimental Raman lines have been assigned to structural units vibrations and stability of these BiB_3O_6 phases are compared. Considerable difference of frequencies and intensities distribution for A_1 polar modes obtained in scattering geometries corresponding to LO and TO phonons, that reveals strong effects of internal electric field induced by polar phonons on lattice dynamics and scattering process. Eigenvectors of the phonons where such effects are most manifestative were determined within lattice dynamics model.

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Development of the scanner for probe microscopes on the basis of mono-crystalline bimorph of lithium niobate

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A wide variety of micro- and nanoscale systems based on the reverse piezoelectric effect have been developed for scientific and technical applications, using different types of piezoelectric elements as active elements [1]. The most widespread in such systems is piezoelectric ceramics because of its large piezoelectric coefficient values at relatively low voltages. Besides there are single-crystal piezoelectric materials [2]. At certain ratio of geometric dimensions and the presence of a formed domain structure of single-crystal elements, it is possible to achieve displacements comparable with piezoceramics.

It is necessary to investigate the possibility of using electromechanical monocrystalline transducers from lithium niobate elements in the scanner of a probe microscope for the purpose of implementing precision movements. The scanning device consists of orthogonally arranged single-crystal working elements X, Y and Z, connected together at the point of installation of the sample holder. As a result of applying an electric field, the single-crystal elements undergo bending deformations by moving the sample holder in the corresponding direction.

The deformation and resonance characteristics of the system determine, in many respects, the basic performance characteristics of the device. For the prototype of a scanning system based on single-crystal lithium niobate bimorphs, the resonant frequency was 1.84 kHz, 1.95 kHz and 1.57 kHz for the X, Y and Z axes, respectively; The developed value of deformations at an applied voltage of 600 V is 6 μm in the XY plane and 5 μm in the Z plane.

The described above system provides high accuracy, repeatability, and no-hysteresis in the object positioning, as a result of using single-crystal elements as electromechanical converters, which are practically devoid of the disadvantages typical for piezoceramic materials

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The dielectric properties of $Bi_5Ti_3Fe_{0,5}Ni_{0,5}O_{15}$ layered perovskite

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The $Bi_5Ti_3Fe_{0,5}Ni_{0,5}O_{15}$ sample was obtained by a standard two-stage ceramic technology [1].

The X-ray diffraction and by scanning electron microscopy samples research showed that the samples belong to the orthorhombic symmetry group are single-phase layered structure.

Analysis of the dependence of the dielectric constant and dielectric loss tangent (Fig. 1) revealed a number of features.

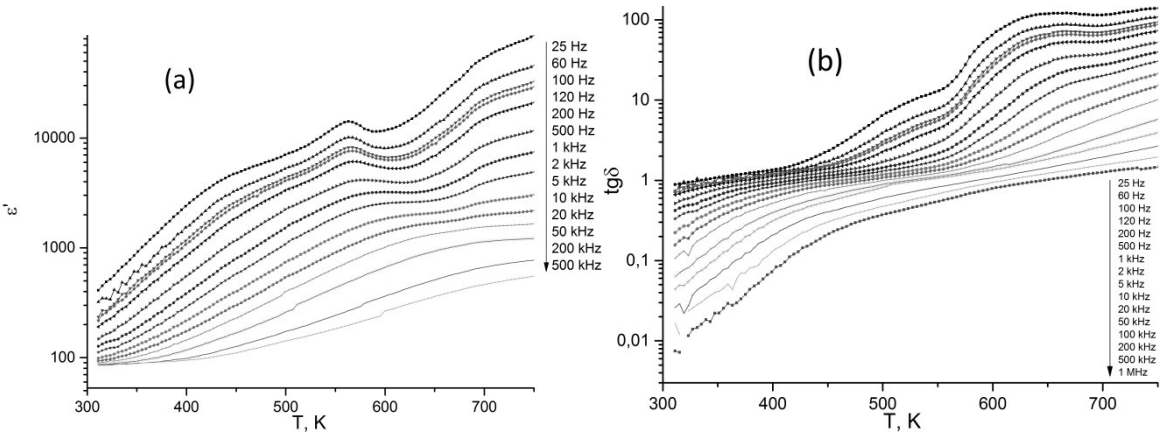

Fig. 1. Temperature dependence of the real part of the dielectric constant ε'(T) (a) and the dielectric loss tangent tgδ(T) (b) at different frequencies

On the dependence of ε'(T) at low frequencies in the 570 K observed the local maxima, the temperature position of which is almost independent of frequency, and dependencies tgδ (T) clearly expressed maxima are absent. Presumably this is due to a significant contribution to the character of the conductivity measured dependencies.

AC conductivity σ_{ac} was designed to clarify this contribution. The complex form of the dependence lnσac (1 / T) indicates the presence of different conductivity mechanisms in the field of high and low temperatures.

As for the total electrical conductivity is influenced by the microstructural characteristics of the materials was considered hodograph sample impedance. It turned out that the space charges are dependent on temperature and frequency.

From the condition of the maximum of the imaginary part of the impedance Z " is defined relaxation time τ of the dielectric polarization in the volume of the crystallite, and from the temperature dependence τ estimated activation energy of the relaxation process.

Analysis of the activation energy values of full electric conductivity and electrical conductivity in the volume of grain showed that the total electrical conductivity of the test σac co-unity is mainly determined by the electrical conductivity of the grain boundaries.

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NMR studies of nanoconfined KDP and DKDP

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NMR investigations were performed for nanocomposites (mesoporous sieves MCM-41 and porous glass with pore sizes 3.7 nm and 7 nm, respectively), filled with potassium dihydrogen phosphate KDP, and nanocomposites (mesoporous sieves MCM-41 with pore size 3.7 nm and artificial opal with globe size 220 nm), filled with deuterated potassium dihydrogen phosphate DKDP. The results were compared to those obtained for KDP [1] and DKDP crystal powders. The measurements were performed using broad band probehead and magic angle spin technique in a large temperature range from 90 K to room temperature.

The changes of ^{31}P NMR line shape and line position due to the ferroelectric phase transition were observed for bulk materials. The parameters of the chemical shift tensor including isotropic shift, anisotropy, asymmetry factor were calculated on the basis of the obtained results. At the temperature of the ferroelectric phase transition the line shifted to low frequency region. The anisotropy of chemical shift tensor rapidly changed and was growing during the further decrease of temperature. The MAS investigations of ^{31}P NMR line allowed us to reveal a smooth decrease of the isotropic shift upon decreasing temperature for bulk DKDP.

The ferroelectric phase transition in KDP embedded into porous glass and opal was not observed in the available temperature range as only the line shape specific for the paraelectric phase was observed. The examples of MAS-spectra for DKDP particles embedded into opal are shown in Fig. 1.

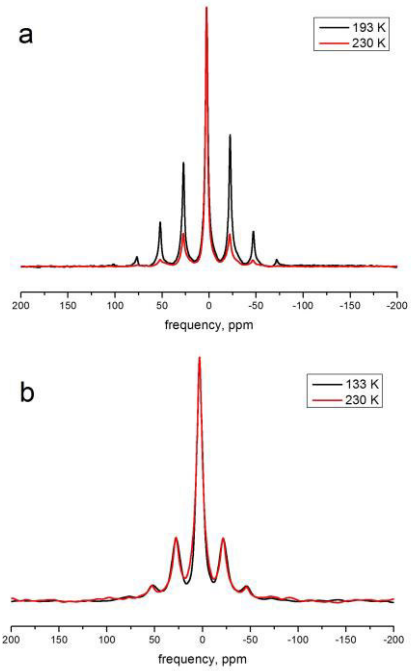

Fig. 1. MAS NMR spectra at two different temperatures: (a) for the DKDP powder, (b) for DKDP embedded into an opal.

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