

Zakład Optoelektroniki

W Zakładzie Optoelektroniki IF PS, współpraca zagraniczna obejmowała w 2008 r. następujące zespoły:

1 – Zespół prof. G. Bulona z Physical Chemistry of Luminescent Materials, Claude Bernard/Lyon 1 University, UMR CNRS 5620, Bat. A. Kastler, 10 rue Ampere, 69622 Villeurbanne, France – wytwarzanie i charakteryzacja monokryształów stosowanych w optoelektronice (lasery, scyntylatory).

2 – Zespół dr L.I. Ivleviej z A.M. Prokhorov General Physics Institute Russian Academy of Sciences, 119991 Moscow, 38 Vavilov Street, Russia – wytwarzanie i charakteryzacja monokryształów laserowych

Ad. 1.

a). Wytworzony w laboratorium Physical Chemistry of Luminescent Materials, Claude Bernard/Lyon 1 University, monokryształ BaY_2F_8 domieszkowany iterbem, został u nas zbadany pod względem właściwości strukturalnych, optycznych i magnetycznych, a wynik tych badań przedstawiono na sympozjum ICL oraz skierowano artykuł do J. of Luminescence.

Udział we wspólnym sympozjum: ICL'08 – International Conference on Luminescence w Lyonie w dniach: 7-12.07. 2008. Przedstawienie prezentacji:

Optical and EPR properties of BaY_2F_8 single crystals doped with Yb

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Abstract

Yb^{3+} doped materials have turned out to be relevant for efficient high-power continuous-wave lasers, up to the kilowatt class. In the present paper we analyze optical and EPR properties of $\text{BaY}_2\text{F}_8:\text{Yb}$ (5wt.%) single crystals obtained using Czochralski method. The Yb^{3+} energy levels for the crystal are as follows: $^2\text{F}_{7/2}$ (0, 216, 430, 530 cm^{-1}), $^2\text{F}_{5/2}$ (10277, 10395, 10735 cm^{-1}). BaY_2F_8 highly doped with Yb reveals complex spectral properties due to possible double structure being partly monoclinic and partly orthorhombic as in case of BaYb_2F_8 . Thus, applied to laser host, it shows some limitations connected with quenching processes by energy transfers to defects and other impurities. Yb^{2+} centers are not observed in the absorption spectrum of “as-grown”

crystal but small amount of the centers we observed just after irradiation of the crystal with γ -quanta (bands with maxima at: 213, 257, 345 and 520 nm). The shape of the additional absorption suggests the Yb^{2+} centers are formed as a result of recharging of one of Yb^{3+} ions from a pair. So, among different kinds of the defects one can observe also Yb^{3+} pairs formation, and, as a consequence Yb^{3+} pairs emission. When we excited $\text{BaY}_2\text{F}_8:\text{Yb}$ (5wt.%) single crystal with 980 nm, we have observed emissions in the VIS and NIR range as bands centered at 478, 560, 649 and 789 nm. The pairs were also observed in the EPR spectra, as satellite lines near central Yb line but only for specific angles (15° - 65° in ZY plane) with respect to magnetic field direction (see Fig. 1). We have analyzed also anisotropy of EPR lines to describe possible symmetries of Yb ions in BaY_2F_8 lattice and temperature dependencies of the Hamiltonian parameters to show the kind and strange of magnetic interactions. It is shown that when pairs are not observed in the EPR spectrum as satellite lines (see Fig. 2), three temperature ranges one can observe. For temperatures lower than 6 K, g-factor is growing up from 3,15 to 3,24, magnetic moment is slowing down. Between 6 K and 30 K g-factor stays constant and equal to 3,24 (a value characteristic for isolated Yb^{3+} ions), magnetic moment is constant, while integral intensity in the range 3-30 K obeys Curie-Weiss law, with $T_c=1,11$ K (Fig. 3). Above 30 K g-factor is again growing up and magnetic moment slowing down. When pairs are observed, two temperature ranges one can recognize. Below 30 K, g-factor stays constant and equals to 2,045, the integral intensity of the EPR line obeys Curie-Weiss relation with $T_c=-3,5$ K suggesting low antiferromagnetic interactions (Fig. 4.). Above 30 K, g-factor is growing up and interactions change to ferromagnetic type. In both cases the linewidth is slowing down when temperature increase from 3 K up to 30 K and next it is growing up. EPR signal vanishes above 50 K (Fig. 5, 6).

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b). Wytworzony w tym laboratorium kolejny monokryształ, CaF_2 domieszkowany iterbem, został u nas zbadany pod względem właściwości optycznych i magnetycznych, a wynik tych badań opublikowano we wspólnej pracy:

S.M. Kaczmarek, G. Leniec, G. Boulon, "EPR results and Raman spectroscopy as a complementary characterization of isolated Yb ions and Yb pairs in $\text{CaF}_2:\text{Yb}$ single crystals", *J. All. Comp.*, 451 (1/2) (2008) 116-121

Ad. 2

a). Wytworzone w tym laboratorium metodą Stiepanova monokryształy SBN czyste i domieszkowane chromem, niklem, cerem, kobaltem zbadano u nas pod względem właściwości optycznych i dielektrycznych, a wyniki prac przedstawiono na konferencjach: IWASOM'08 (Second International Workshop on Advanced Spectroscopy and Optical Materials) w Gdańsku, w dniach 13-17.07. 2008 oraz FNMA'08 (Functional Materials) we Lwowie, w dniach 31.08-6.09.2008. Odpowiednie artykuły wysłano do Opt. Mat. oraz J. Non-Cryst. Solids.

Optical and dielectric properties of SBN single crystals doped with Co, Cr, Ni and Ce

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Abstract

SBN61 pure, SBN61:Cr (0,01wt.%), SBN61:Co (0,01wt.%), SBN61:Ni (0,01wt.%, 0,5wt.%) and SBN61:Ce (0,01wt.%) single crystals were grown using modified Stepanov technique at A.M. Prokhorov General Physics Institute, Russian Academy of Sciences. Absorption and photoluminescence measurements of the crystals were performed using Perkin-Elmer spectrophotometer and spectrofluorimeter and compared to SBN33 and SBN58:Cr (0,02wt.%) single crystals obtained by Czochralski method, and, analyzed elsewhere [1].

In the frame of dielectrical measurements the following parameters were analyzed: dielectric permittivity ϵ' , conductivity and loss tangent of the compound, both as a function of a temperature (290-400 K) at 10 kHz and frequency (1 Hz-100 kHz). Conductivity activation energies and ferroelectric temperatures of relaxor type phase transitions of pure and SBN61 crystals doped with Co, Cr, Ni and Ce were established and compared to SBN33 and SBN58 crystals [2, 3]. The influence of the stoichiometry, type and concentration of a dopant on the dielectric properties were analyzed. The measurement set has consisted of Hewlett Packard bridge HP4192A, wide temperature range measurement chamber, temperature control unit (SHIMADEN TSR25 /Pt100), and computer.

References

- [1] S.M. Kaczmarek, M. Berkowski, K. Repow, M. Orłowski, A. Worsztynowicz, M. Włodarski, *Rev. Adv. Mat. Sci.* 14 (2007) 49
- [2] K. Matyjasek, K. Repow, S.M. Kaczmarek, M. Berkowski, *J. Phys.:Cond. Mat.*, 19 (2007) 466207 (10pp)
- [3] K. Matyjasek, K. Wolska, S.M. Kaczmarek, , *J. Phys.: Cond. Mat.*, sent 2008

Ferroelectric properties of relaxor type SBN single crystals doped with Co, Cr, Ni and Ce

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Abstract

SBN61 pure, SBN61:Cr (0,01wt.%), SBN61:Co (0,01wt.%), SBN61:Ni (0,01wt.%, 0,5wt.%) and SBN61:Ce (0,01wt.%) single crystals were grown using modified Stepanov technique in the A.M. Prokhorov General Physics Institute, Russian Academy of Sciences. Absorption measurements of the crystals were done using Perkin-Elmer spectrophotometer and compared to SBN58 single crystals obtained by Czochralski method, and, analyzed elsewhere [1].

In the frame of dielectrical measurements the following parameters were analyzed: electric permittivity ϵ' , conductivity and loss tangent of the compound, both as a function of a temperature (290-400 K) at 10 kHz and frequency (1 Hz-100 kHz). Conductivity activation energies and ferroelectric temperatures of relaxor type phase transitions of pure and SBN61 crystals doped with Co, Cr, Ni and Ce were established and compared to SBN58 ones [2, 3]. The measurement set has consisted of Hewlett Packard bridge HP4192A, wide temperature range measurement chamber, temperature control unit (SHIMADEN TSR25 /Pt100), and computer. Direct observation of the evolution of the domain structure during polarization reversal was performed for pure SBN61 by the nematic liquid crystal decoration technique and compared to previous results for SBN58:Cr crystal.

References

- [1] S.M. Kaczmarek, M. Berkowski, K. Repow, M. Orłowski, A. Worsztynowicz, M. Włodarski, *Rev. Adv. Mat. Sci.* 14 (2007) 49
- [2] K. Matyjasek, K. Repow, S.M. Kaczmarek, M. Berkowski, *J. Phys.:Cond. Mat.*, 19 (2007) 466207 (10pp)
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Optical and EPR properties of BaY₂F₈ single crystals doped with Yb.

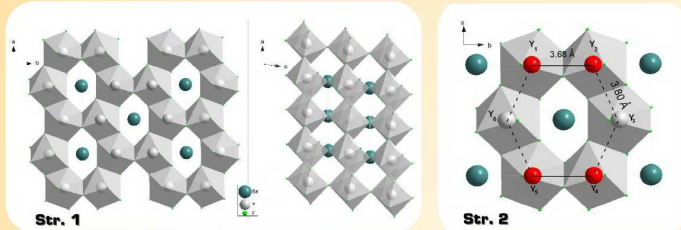
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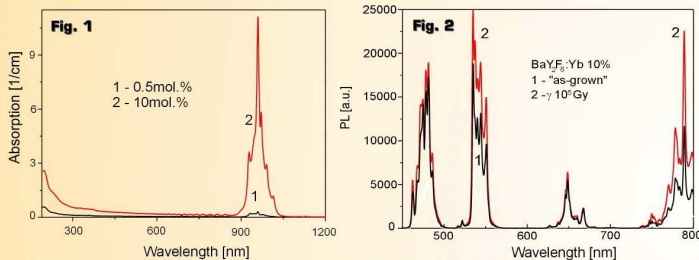
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INTRODUCTION

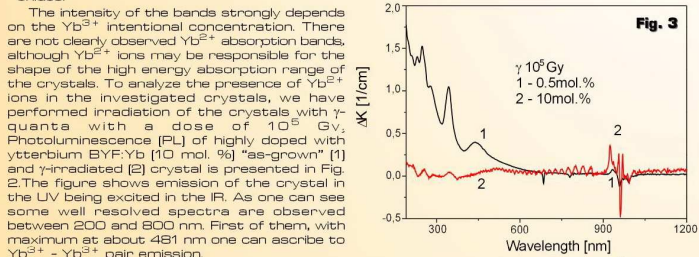


The symmetry of the BYF crystal is monoclinic [space group C_{2h}^2-C2/m with lattice parameters: $a=0.6972$ Å, $b=1.0505$ Å, $c=0.4260$ Å, $\beta=99.45^\circ$]. The primitive cell contains two BaY_2F_8 molecules. The Y^{3+} ion lattice site [in International Tables for Crystallography the site has Wyckoff letter g, multiplicity 4, site symmetry 2] is surrounded by eight F⁻ anions forming slightly deformed Thompson cube. Itrium polyhedra, sharing a common edge, form parallel (001) planes consisting of six-segmented rings. Substituting ion may occupy mainly Y^{3+} sites that are all crystallographically equivalent, but some discrepancies from C_{2h} local point symmetry of the ytterbium ion in BYF matrix due to e.g. high concentration and lattice defects could be expected.

OPTICAL INVESTIGATIONS



The interpretation of the Yb^{3+} energy levels in the different fluoride hosts turned out to be difficult mainly because of the strong phonon-electron coupling of Yb^{3+} ion which results in the appearance of additional peaks making the interpretation of the different absorption and emission lines complex, especially at room temperature. Room temperature absorption spectra of Yb^{3+} in BYF single crystals due to the $^2F_{7/2} \rightarrow ^2F_{5/2}$ transition are presented in Fig. 1. The intense, well resolved bands are observed at 930, 948, 980 and about 980 nm. Some weak bands one can also recognize due to vibronic transitions as observed in various Yb^{2+} -doped oxides.



The intensity of the bands strongly depends on the Yb^{3+} intentional concentration. There are not clearly observed Yb^{2+} absorption bands, although Yb^{2+} ions may be responsible for the shape of the high energy absorption range of the crystals. To analyze the presence of Yb^{2+} ions in the investigated crystals, we have performed irradiation of the crystals with γ -quanta with a dose of 10^5 Gy. Photoluminescence (PL) of highly doped with ytterbium BYF:Yb (10 mol. %) "as-grown" (1) and γ -irradiated (2) crystal is presented in Fig. 2. The figure shows emission of the crystal in the UV being excited in the IR. As one can see some well resolved spectra are observed between 200 and 800 nm. First of them, with maximum at about 481 nm one can ascribe to $Yb^{2+} \rightarrow Yb^{3+}$ pair emission.

The second one, with a maximum at about 550 nm seems to be assigned to Yb^{2+} emission. Two other spectra may come from ytterbium clusters: $Yb^{3+} \rightarrow 650$ nm and $Yb^{2+} \rightarrow 800$ nm. It is observed that γ -irradiation (curve 2) leads to an increase in the amount of Yb^{2+} luminescence centers (the curves are normalized each other). For low doped BYF:Yb (1 - 0.5mol.%) crystals we have not observed PL for as-grown crystal while a weak one for γ -irradiated. The observed bands confirmed the presence of Yb^{2+} centers in γ -irradiated crystals.

The results of the investigations of the γ -induced absorption of low and high Yb doped BYF:Yb (1 - 0.5mol.%, 2 - 10 mol.%, respectively) crystals are presented in Fig. 3. The registered additional absorption bands are characteristic for Yb^{2+} ions [e.g. 340 nm] and color centers [e.g. 440 nm] absorption. One can conclude that in Yb^{2+} -doped BYF, Yb^{2+} are created by γ -irradiation, giving rise to absorption bands in 200-300 nm range.

ELECTRON PARAMAGNETIC RESONANCE

Free Yb^{3+} ion has a $4f^{13}$ electronic configuration. The free-ion Hamiltonian produces the $^{2F_{7/2}}$ multiplets separated by about 10^3-10^4 cm⁻¹. According to Hund's rules the ground state is $^2F_{7/2}$ and the excited state is $^2F_{5/2}$. The crystal field of the host material removes the $2J+1$ degeneracy of the $^{2F_{7/2}}$ states and gives twofold degenerate states called Kramers doublets, separated by about 10^3-10^4 cm⁻¹. This degeneracy could be lifted by an external magnetic field. At low liquid temperature, only the lowest doublet is populated and thus the EPR spectrum can be described by a Hamiltonian with an effective spin $S=1/2$. In addition to even isotopes ($I=0$, natural abundance 68%), ytterbium has two odd isotopes which give rise to hyperfine structure, ^{171}Yb ($I=1/2$, natural abundance 14.4%) and ^{173}Yb ($I=5/2$, natural abundance 15.6%). The resulting effective spin Hamiltonian of the Yb^{3+} monomers is

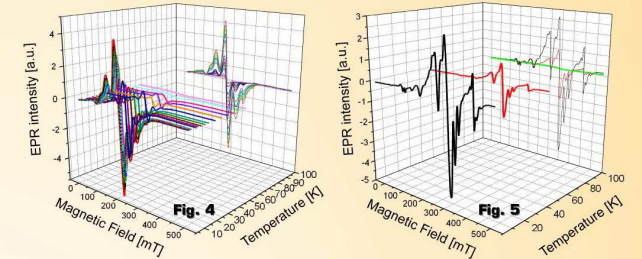
$$H_{\text{mono}} = \mu_B \mathbf{B} \cdot \mathbf{g} \cdot \mathbf{S} + \mathbf{S} \cdot \mathbf{A} \cdot \mathbf{I}$$

where μ_B is the Bohr magneton, \mathbf{S} is the electron spin in the ground state, \mathbf{I} is the nuclear spin and \mathbf{B} is the external Zeeman field. The $\mathbf{S} \cdot \mathbf{A} \cdot \mathbf{I}$ term represents the hyperfine interaction. As the doping level of Yb in BYF is high the existence of Yb pairs and clusters should be considered. For pairs of similar ions (two coupled Yb^{3+} ions in neighbouring sites A and B) the following Hamiltonian is relevant:

$$H_{\text{pairs}} = \mu_B \mathbf{B} \cdot \mathbf{g}^A \cdot \mathbf{S}^A + \mu_B \mathbf{B} \cdot \mathbf{g}^B \cdot \mathbf{S}^B - 2J \cdot \mathbf{S}^A \cdot \mathbf{S}^B + \mathbf{S}^A \cdot \mathbf{D}^A \cdot \mathbf{S}^B$$

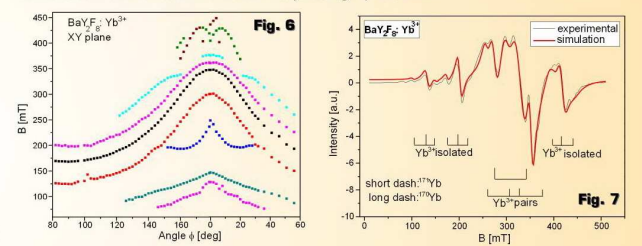
where the two first terms are the Zeeman terms, the third is an isotropic part (Heisenberg type) of the exchange interaction and the fourth term is the magnetic dipole-dipole interaction. J is the isotropic exchange interaction constant that is usually not directly calculated from the EPR spectrum.

When J is of the order of kT it could be determined from the temperature dependence of the EPR signal intensity. Otherwise it could be measured by high-resolution spectroscopy or from EPR hyperfine spectrum in case of paramagnetic species with nuclear magnetic moments. The dipolar tensor has the following well-known expression[1].



Figs. 4 ($\phi=54^\circ$) and 5 (crystallographic b axis) present a few of the EPR spectra registered at different temperatures, for two specific orientations of the external magnetic field relative to the crystallographic axes.

The observed EPR spectrum consists of many overlapping lines, mainly in the 250-450 mT range. It resembles in many ways the spectrum of rare earth pairs registered. With increasing temperature the intensity of the lines quickly decreases and above 50 K the EPR spectrum is hardly visible. This is to be expected for the rare-earth ions due to a strong increase with temperature of the relaxation rates of the spin system. There is also a large variation of the resonance lines positions due to changes of the external field orientation relative to crystallographic axes.



In Fig. 6 the angle dependence of selected, relatively well separated EPR lines observed in the XY plane (ab crystallographic plane) is presented. This angular dependence shows that the resonance lines have axial symmetry about the crystallographic b axis. The angle dependence also enables to group the resonance lines into two sets: one set contains the lines that are attributed to separated Yb^{3+} ions and the other to the Yb^{3+} pairs. The lines attributed to pairs should have $(3\cos^2\phi - 1)$ -type dependence, where ϕ is the angle between magnetic field and the pair axis. The ytterbium monomers might show a different type of pattern in the angle dependence.

Fig. 7 shows the results of the fitting of the observed spectrum registered at 23 K for magnetic field directed along the y axis. Although there are differences between experimental [black line] and simulated spectra [red line], the main features of the EPR spectrum are reproduced correctly.

	Parameter	Value
Yb^{3+} monomers	$g_{ }$	5.97 3.69 2.17
	g_{\perp}	6.36 2.86 1.36
	$g_{ }$	2.216
$Yb^{2+}-Yb^{3+}$ pairs	g_{\perp}	2.241
	D_1 [10^4 cm ⁻¹]	235.14
	D_2 [10^4 cm ⁻¹]	229.92
	R [Å]	3.59

The dipolar parameters D_1 and D_2 are given by the following equations:

$$D_1 = \frac{\beta^2(-g_{||}^2)}{2R^3} \quad D_2 = \frac{\beta^2(g_{\perp}^2)}{2R^3}$$

The location of the Yb^{3+} pair is proposed. The two Yb^{3+} ions forming a pair are assumed to enter substitutionally at Y sites. In that case there is no problem with charge compensation and the ionic radii of both ions in eightfold coordination are similar ($r(Y^{2+})=1.015$ Å, $r(Yb^{3+})=0.98$ Å). The distances between six neighboring Y^{3+} ions forming the ring in ab plane (designated as Y_1-Y_6 in Str. 2) are not the same; the distances Y_1-Y_2 and Y_4-Y_6 are equal ($L_1=3.68$ Å) and different from Y_2-Y_3 , Y_3-Y_4 , Y_5-Y_6 and Y_6-Y_1 distances ($L_2=3.80$ Å). It is proposed that the Yb^{3+} pair generating the observed EPR spectrum is placed at Y_1-Y_2 and Y_4-Y_5 sites. There are two main arguments for selecting such location: the axis of a pair should be directed along b crystallographic axis (it follows from the notational diagram) and the calculated separation between Yb^{3+} ions in a pair (3.59 Å) is closer to that particular ytterbium distance. The smaller separation in the Yb^{3+} pair could be mostly accounted for by a smaller ionic radius of the substituting ion so the relaxation effect should play only a minor role. It is more difficult to propose the locations of paramagnetic centers attributed to the monomeric Yb^{3+} species because there are many possibilities in the crystallographic structure of BYF to accommodate these centers. They could be associated with a single Yb^{3+} ions substituting for Y^{3+} ion at any of the Y_1-Y_6 sites or forming the $Yb^{3+}-Yb^{2+}$ pair located at two neighboring Y sites. Due to the mentioned above differences in Y-Y distances the substitution of that pair in e.g. Y_1-Y_2 or Y_4-Y_5 positions would produce two magnetically inequivalent paramagnetic centers.

[1] O. Guillot-Noel, Ph. Golden, P. Higel, D. Gournier, J. Phys.: Cond. Matter, 16 (2004) R1-R84



Optical and dielectric properties of SBN single crystals doped with Co, Cr, Ni and Ce



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ABSTRACT

Sr_{0.61}Ba_{0.39}Nb₂O₆ (SBN61): SBN61 pure, SBN61:Cr (0,01mol%), SBN61:Co (0,01mol%), SBN61:Ni (0,01mol%, 0,5mol%) and SBN61:Ce (0,01mol%) single crystals were grown using modified Stepanov technique at A.M. Prokhorov General Physics Institute, Russian Academy of Sciences. In this work the SBN61 crystals pure and doped with Ce, Co, Cr and Ni were analyzed using transmission, photoluminescence and dielectric measurements to look for their optical and dielectric properties and compare them to the crystals obtained by usual Czochralski method. Moreover, direct observation of the evolution of the domain structure during polarization reversal was performed by the nematic liquid crystal (NLC) decoration technique and compared to previous results for SBN58:Cr crystal.

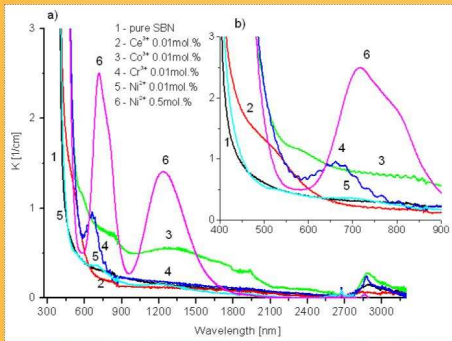


Fig. 1. Absorption spectra of SBN crystals: pure (1) and doped with Ce (2), Co (3), Cr (4) and Ni (5, 6).

OPTICAL MEASUREMENTS

As one can see from Fig. 1a, b, the absorption of the SBN61 single crystals doped with Co, Ni, Cr and Ce reveal the presence of some well resolved bands in the VIS and IR part of the absorption spectrum due to: Ce³⁺ absorption (curve 2, 522 nm band 4f→5d), Co²⁺ and Co³⁺ absorption (curve 3, 570 nm band ⁴T₁(F)→⁴T₁(P), 1400-2000 nm band ⁴T₁→⁴T₂ and 1280 nm band ³T₂→⁴E, respectively), Cr³⁺ absorption (curve 4, 667 nm band ⁴A₂→⁴T₂), Ni²⁺ absorption (curves 5 and 6, 717 and 801 double band ³B₁→³A₂, ³E and 1239 nm band ³B₁→³E, ³B₂). As compare to Czochralski grown SBN61:Cr crystal, the ⁴A₂→⁴T₂ absorption band of the crystal obtained by Stepanov modified Czochralski growth is shifted towards longer wavelengths (650 nm for the crystal grown by the Czochralski method). Fundamental absorption edge (FAE) changes from 374 nm for pure and Ce, Co (0,01mol%) doped, 378 nm – Cr, Ni (0,01mol%) doped, to 419 nm for Ni (0,5mol%) doped SBN single crystals. For crystals obtained by the Czochralski method FAE=370 nm.

DOMAIN NUCLEATION AND GROWTH IN RELAXOR-FERROELECTRIC SBN61

The microscopic observations revealed that the formation of domains is possible if the amplitude of electric field exceeds some threshold value at a given site of the SBN crystal. Fig. 2. illustrates the domain pattern evolution observed on a small surface area of about 1 mm², after applying an electric field of 2,5 kV/cm to the monodomain crystal sample. The polarization axis is normal to the image plane. We can observe a fast partial switching, by intensive nucleation of domains, instantaneously after the field application. Further switching continues to occur more slowly through sidewise domain wall motion.

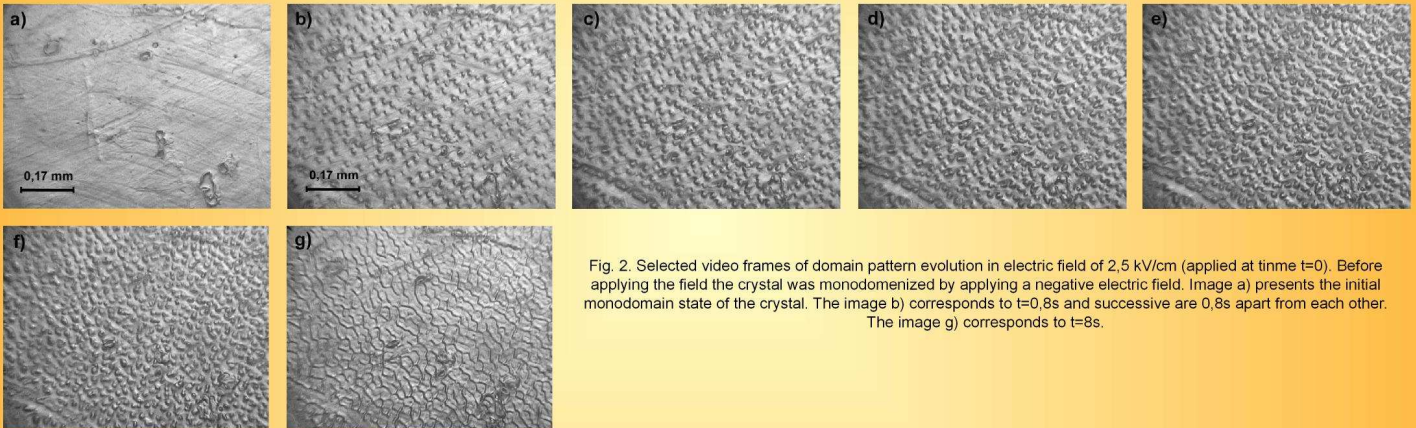


Fig. 2. Selected video frames of domain pattern evolution in electric field of 2,5 kV/cm (applied at time t=0). Before applying the field the crystal was monodomainized by applying a negative electric field. Image a) presents the initial monodomain state of the crystal. The image b) corresponds to t=0,8s and successive are 0,8s apart from each other. The image g) corresponds to t=8s.

DIELECTRIC PROPERTIES

The variations of dielectric constant with temperature for SBN61, SBN61:Ce and SBN61:Cr samples at two different frequencies (1 kHz and 10 kHz) are shown in Fig. 3(a-b). The graphs show a broad dielectric peak indicating that the measurement samples undergo a phase-transition. The dielectric constant maximum of SBN61, SBN61:Ce and SBN61:Cr were observed at 96, 86 and 74 C during heating; and at 104, 86 and 62 C during cooling at 8 kHz frequency. This inconsistency is due to existing thermal stagnation. The dielectric constant vs temperature plots of SBN61:Ni at different frequencies (1kHz and 10 kHz), are shown in Fig. 4. For all measurement samples, with increasing frequency, dielectric constant decreases.

CONCLUSIONS

- 1) For Stepanov modified Czochralski growth, band gap energy changes from 3,28 eV (pure and Ce, Co doped), 3,24 eV (Ni – 0,01mol% doped), 3,17 eV (Cr doped) to 2,83 eV for Ni (0,5mol%) doped crystals. Unexpectedly, photoluminescence measurements have not shown any emission from the above crystals.
- 2) For the measurement samples of SBN61, SBN61:Ce, SBN61:Cr and SBN61:Ni we obtained Curie temperature (T_c) was equal to 100, 86, 68 and 89 C, respectively.
- 3) When compare both methods of crystal growth one can found that SBN61 crystals obtained by Stepanov method reveal higher mechanical and optical quality and thus lower linkage current due to lower content of oxygen vacancies, lower value of electric permittivity and greater value of coercive field – domains are stronger pinned. We have found that SBN58 crystal (obtained using CZ growth) exhibits a high electrical conductivity (halls) which affects the switching properties at room temperature and may be responsible for the instability of its parameters.

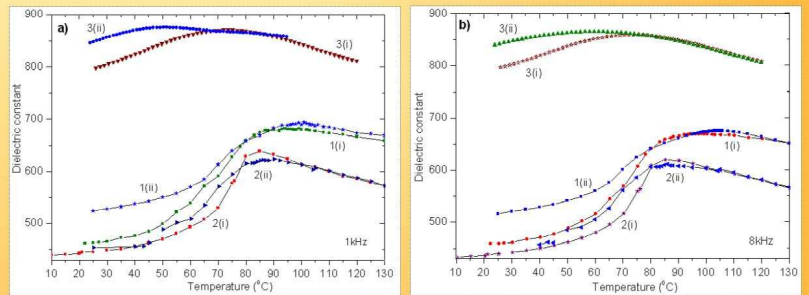


Fig.3 Temperature variation of relative dielectric constant (ε_r) registered for: (1) SBN61, (2) SBN61:Ce and SBN61:Cr at two frequencies 1kHz (a) and 8kHz (b); (i) during heating and (ii) during cooling.

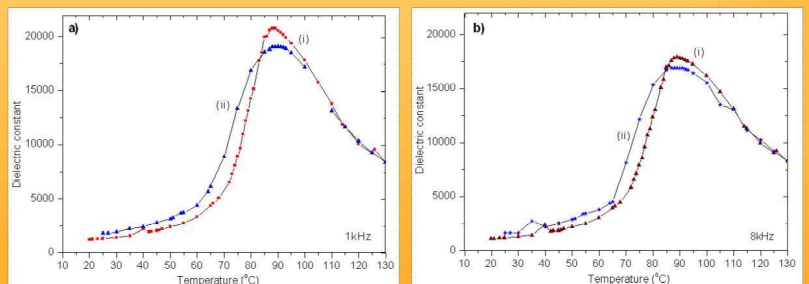


Fig.4 Temperature variation of relative dielectric constant (ε_r) registered for SBN61:Ni at frequencies 1kHz (a) and 8kHz (b); (i) during heating and (ii) during cooling.