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Domain nucleation and growth in relaxor-ferroelectric $\text{Sr}_{0.58}\text{Ba}_{0.42}\text{Nb}_2\text{O}_6$ doped with chromium and ytterbium

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Abstract

We present real-time studies of domain nucleation and growth processes in $\text{Sr}_{0.58}\text{Ba}_{0.42}\text{Nb}_2\text{O}_6$ (SBN:58) crystals doped with Cr and Yb ions, by the nematic liquid crystal (NLC) decoration technique. Our results show that the polarization reversal in doped SBN crystals is strongly affected by a random-field environment related to the relaxor properties of this material. An important result is the increase of the relative contribution of transient nucleation to polarization reversal in a constant electric field application. The sideways domain wall motion proceeds through a pinning–depinning mechanism. This is reflected in a very fine, irregular domain structure during polarization reversal. Registration of domain dynamics in conjunction with the switching current recording have shown that there are slow switching regions that do not contribute to the switching current signals, even under electric field exceeding the coercive field. The kinetics of polarization in SBN:58 doped crystals is described with a stretched exponential function.

1. Introduction

The mechanism of polarization reversal in ferroelectrics has been intensively discussed in recent years due to potential use of ferroelectric thin films in the construction of non-volatile memory devices [1]. In normal homogeneous ferroelectrics the switching process has often been analysed on the basis of Kolmogorov–Avrami–Ishibashi (KAI) statistical theory [2]. This model assumes that a great number of domain nuclei are randomly distributed over the volume of the crystal and then overlap each other (domain coalescence). It has been found that over a very wide range of electric fields, the KAI model is not a good description of switching processes in relaxor-ferroelectrics [3–5] and ferroelectric thin films [6], as it can give physically unclear values of fitting parameters. Experiments reveal that in relaxor-ferroelectrics polarization develops with quite long relaxation times under electric fields, in comparison to normal ferroelectrics. The origin of the slow kinetics in relaxor-ferroelectrics is not established at present. A model based on the assumption of charge disorder giving rise to random fluctuations of the crystalline internal field has been proposed to explain the dielectric relaxation and switching behaviour in relaxor-ferroelectrics [7]. Optical techniques are much more valuable in obtaining spatio-temporal information

about domain switching and are a useful completion to the usual electrical measurements. Direct observations of the polarization reversal permit us to extract the slowly relaxing component of polarization from the rapid response.

Although there have been several studies of switching processes in SBN crystals [8–10], there is still a remarkable lack of information on their domain dynamics. Recently, nanoscale-domain structures in SBN single crystals [11–13] and thin films [14] have been studied using an atomic force microscope (AFM). However, applying voltage pulses to a conductive AFM tip enables the controlling domain dynamics in a highly localized electric field, thus hindering the identification of the sites of domain nucleation. The observation of domain reversal in SBN single crystals was reported by electric–optic imaging microscopy [10, 15] and the NLC method [16].

In this paper direct observation of the evolution of the domain structure during polarization reversal was performed by the NLC technique [17]. This method limits the spatial resolution (up to 1 μm in size) and affects the switching behaviour, however it does enable uniform switching across the large area of the crystal sample. This paper presents the investigation of the switching process in SBN:58 crystals doped with Cr and Yb ions, by observation of the

domain structure in low electric fields and switching current registration in high fields. Because of a large luminescence efficiency, at room temperature, SBN doped with Yb ions has promise for use in laser systems [18]. In a previous study [19] we showed a significant effect of a large leakage current on the switching properties in pure SBN:33 crystal. A large leakage current means that it is not possible to obtain a well-saturated ferroelectric hysteresis loop which thus hinders its practical applications. Doped SBN crystals with rare-earth or alkali ions show more stable and reproducible ferroelectric parameters in comparison with pure crystals [20]. The doping has resulted in a reduction in the leakage current and improvement in the ferroelectric properties of SBN. The effect of the dopants on the domain dynamics will be analysed in another publication.

2. Experimental details

SBN crystals exhibit domains only 180° because the paraelectric phase above T_c has a tetragonal symmetry ($4/mmm$) and the transition to the ferroelectric phase ($4mm$) occurs along the [001] direction [21]. Details of the growing procedure, by the Czochralski method, are presented elsewhere [22]. Doping was performed by adding Cr_2O_3 (0.02 wt%) and Yb_2O_3 (0.5 wt%) in the melt, yielding $T_c \approx 340$ K at frequency 1 kHz. The striation free crystal samples were cut perpendicularly to the polar c -axis and polished to optical quality. To observe 180° domains the NLC mixture of p-methoxybenzylidene-p-n-butylaniline (MBBA) and p-ethoxybenzylidene-p-n-butylaniline (EBBA) was used. The static domain structure on the polished polar surface of the SBN crystal gave no contrast. For dynamic observation in an electric field, a crystal plate with a thin NLC layer, on its upper and lower surfaces, was sandwiched by two glass plates with a tin oxide coating as transparent electrodes. The observation of domains was carried out on the polar surface (normal to the c -direction) using a polarizing microscope. The method for studying the domain dynamics is based on the visualization with the NLC of the moving domain walls. In the regions where the repolarization occurs, switching current flows followed by a decrease in the impedance of the ferroelectric; hence the major part of the applied voltage falls on the liquid crystals. Thus the reversed regions, where reorientation of domain structure still occurs, look somewhat darker than the ‘white’ surrounding domains, as in these regions a certain electrohydrodynamic instability, particularly dynamic scattering, takes place [17]. A video camera with a 40 ms resolution clock was used to monitor the evolution of the domain pattern in the imaging area.

Hysteresis loops were obtained by means of a modified Sawyer–Tower bridge, using an ac electric field of 50 Hz. The switching currents were measured by applying square wave electric pulses amplified with a Kepco bipolar amplifier. The voltage across the 50 Ω resistor, connected in series with the crystal sample, was measured using a digital oscilloscope. The electrical measurements were carried out on the sample having an electrode area of 0.12 cm^2 and thickness of 0.6 mm with air-drying silver paste as the electrodes. The results presented refer to one chosen crystal sample. However, these results are typical of a fairly large number of doped SBN crystal samples.

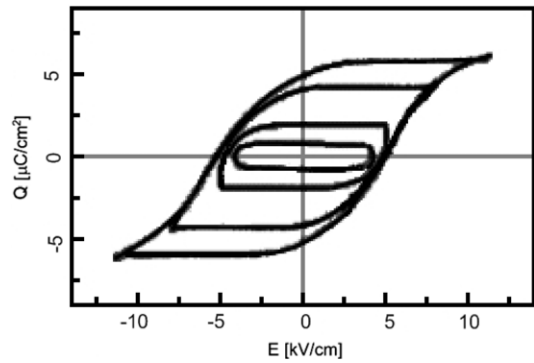


Figure 1. Family of hysteresis loops for increasing amplitude of the ac field at a frequency of 50 Hz.

3. Results

3.1. Hysteresis loops measurements

Electrical treatment is used as a standard technique, allowing a fast characterization of the ferroelectric crystal. Figure 1 shows the shape of the hysteresis loop of the examined crystal sample at various amplitudes of an ac field. With the increase of the electric field the part of the crystal which has undergone switching increases. One can observe that the coercive field, E_c , remains almost unchanged. This means that there is a threshold electric field (nucleation field) below which the realignment of domains cannot occur. The symmetry of the hysteresis loop indicates a lack of a built-in directional internal field, which could stabilize the domain structure in a preferential direction. The remnant polarization P_r and coercive field E_c of the SBN:58 doped crystal were $6.2 \pm 0.2 \mu\text{C cm}^{-2}$ and $5 \pm 0.6 \text{ kV cm}^{-1}$ respectively, at an applied field of 11.5 kV cm^{-1} (about twice E_c). By comparison, for crystal SBN:50 doped with only a small Cr concentration (0.02 wt%) the values of P_r and E_c at room temperature were about $17 \mu\text{C cm}^{-2}$ and 3.5 kV cm^{-1} , respectively [23]. Thus, there is an optimum dopant concentration for the maximum improvement of electrical properties in doped SBN crystals. These results suggest that in the examined SBN:58 crystal, even in $E > E_c$, there are frozen regions that do not participate in the switching polarization and/or that a large contribution to the polarization is due to sidewise domain wall motion, which is a very slow mechanism [24]. We have also observed a significant decay of polarization (by $\sim 50\%$) with the increase of the frequency from 50 Hz to 1 kHz (at an ac field amplitude $E \sim 2E_c$). Interestingly, the drastic loss of polarization with the frequency does not involve a distinct increase in the E_c (about 10%), only the hysteresis loop became more slanted. The value of E_c became more frequency dependent only at frequencies below 50 Hz, in the freshly annealed crystal sample. This means that domains do not switch instantaneously, but need a certain time to reverse their orientation, thus E_c seems to shift to higher values at higher frequencies. Similar results were obtained for cerium doped SBN:61 during quasistatic registration of the hysteresis loop by the surface-charge-detection method [25]. While macroscopic

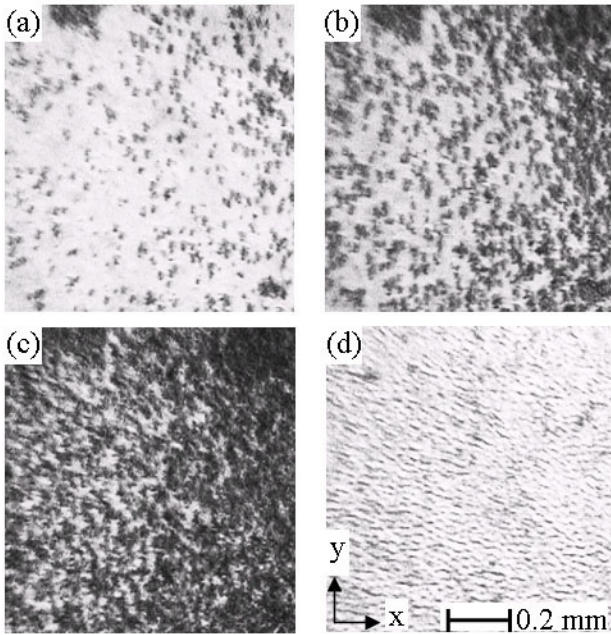


Figure 2. Selected video frames of domain pattern evolution in an electric field of 3.2 kV cm^{-1} (applied at time $t = 0 \text{ s}$); Image (a) corresponds to $t = 0.2 \text{ s}$, and successive parts are 0.2 s apart from each other. Before applying E the crystal was monodomenized.

hysteresis loop measurements can be used to investigate the collective switching process of all domains under the electrode, local microscopic observations can reveal more about nucleation and growth of domains during polarization reversal.

3.2. Domain structure dynamics

The domain dynamics was investigated by recording the domain pattern during switching from a single domain state, induced by external electric field. Direct observation of the domain structure confirms that there is a minimum threshold field which can be used to realign the domains. The threshold nucleation field depends on the state of ageing of the sample. When an electric field is switched off for several hours, then a larger E must be applied to initiate the domain nucleation. Applying larger E to overcome internal fields that stabilize the domain structure may fully restore the aged sample to its initial state of polarization (i.e. initial value of threshold nucleation field).

An essential difference is observed between domain dynamics observed in normal ferroelectric materials and relaxor SBN crystals. In homogeneous crystals most nuclei involved in the switching process might originate in the defects at the crystal/electrode interfaces or inside the crystal, and arise as soon as the electric field is applied [24]. Because energetically the growth of existing domains is more favourable than the creation of new ones, they expand with little or no resistance under an electric field. So that small domains start to coalesce into larger ones, accompanied by a decrease of the domain density.

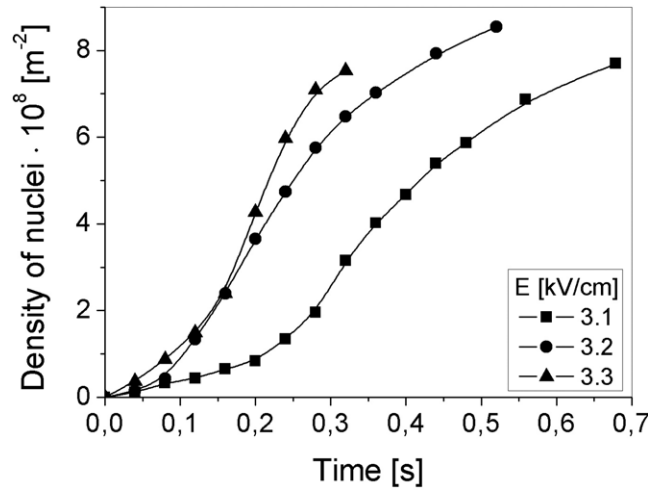


Figure 3. Temporal dependence of nucleated domains per unit area at various electric fields.

Figure 2 illustrates the domain pattern evolution observed in a small (about 1 mm^2) region of the crystal sample after applying an electric field of 3.2 kV cm^{-1} . Unlike homogeneous crystals in relaxor SBN, the creation of new domains is more favourable than their growth. Thus, the nucleation process continues to occur nearly during the whole polarization reversal process, at constant electric field, as is clearly seen in figures 2(a)–(c), where the sites in which intensive nucleation takes place are seen as ‘black’ regions, and ‘white’ regions represent the original polarization state before switching. In the last stage of the polarization reversal process, when the rapid nucleation in the bulk of the crystal is finished, further switching proceeds more slowly through lateral expansion of domain walls, which expand with a great resistance under an electric field. The pronounced slowing down of the domain walls is clearly reflected in a poor orientation contrast of NLC above the moving domain walls, as is seen in figure 2(d). It should be emphasized that the thickness of the ‘visible’ walls is not the real physical thickness of the domain walls, which are usually of the order of a few lattice constants. In consequence a fine domain structure with a large density of domain walls is formed with an irregular fuzzy shape.

The specific mechanism driving the nucleation in SBN crystals results from the local structural irregularities, and could be interpreted in terms of a wide distribution of activation energies for nucleation. The term ‘nucleation’ is used to describe the emergence of new, sharp, visible, antiparallel domains within the original single domain state, as they appear in the video images. The diameter of such a domain is about $5 \mu\text{m}$. From the image analysis of the domain patterns, the temporal dependence of the total number of nucleated domains per unit area (density of nuclei) has been obtained, as presented in figure 3. One can see that the nucleation rate does not stay constant throughout the switching process. This indicates that the description of switching current that postulates a constant nucleation rate is not valid for relaxor SBN crystals.

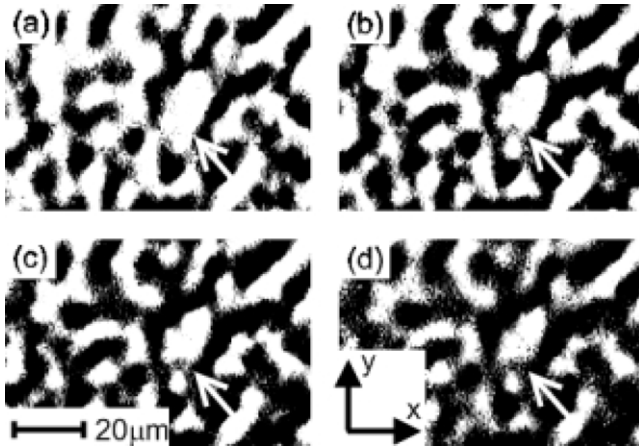


Figure 4. Selected video frames of domain pattern evolution during the switching process in $E = 2.8 \text{ kV cm}^{-1}$. Times from the moment of applying E are (a) 2 s, (b) 2.6 s, (c) 3.2 s, (d) 3.8 s. In order to show the details of the domain structure the contrast has been enhanced.

3.2.1. Domain wall motion. A close inspection revealed that the sideways velocity of a particular domain wall is not constant during its propagation. Furthermore, we never observed any motion of the existing domains as a whole. Local structural irregularities provide strong pinning locations at which domain walls remain attached. As a result, the large domains are broken up into smaller ones, as is clearly seen (comparing four images in figure 4) in the positions indicated by arrows. Thus, the domain structure consists of irregular patches. Figure 5 presents the real-time dynamics of the domain wall front by measuring the distance travelled (along the diagonal $[110]$ direction) versus time, under an electric field of 2.8 kV cm^{-1} . The velocity calculated as a function of time has maxima, thus showing the jerky process of the domain wall motion, which proceeds through a stop-and-go fashion. The fluctuations of the domain wall velocity by nearly two orders of magnitude are indicative of a broad distribution of the domain wall mobility. This means that SBN crystals contain very powerful pinning centres, which stabilize certain parts of the sample while switching the rest of the sample with an external field. Such pinning centres are probably due to the charge disorder inherent in doped SBN crystals.

It must be noted that jerky motion of the domain wall was observed in the SBN crystal in low electric fields, lower than the coercive field. A kinetic model describing the jerky nature of domain wall motion in PZT films has been presented in [26]. It is interesting to note that using electro-optic imaging microscopy it has been found that in SBN:61 the domain wall velocity was constant during its propagation in $E > E_c$. Because of a slow response time (of the order of several ms) NLC molecules may not reflect the changes of the domain structure in $E > E_c$ that exhibit high speed switching.

3.3. Correlation between domain dynamics and transient currents

The fast polarization switching was investigated by measuring current transients in response to square wave electric pulses.

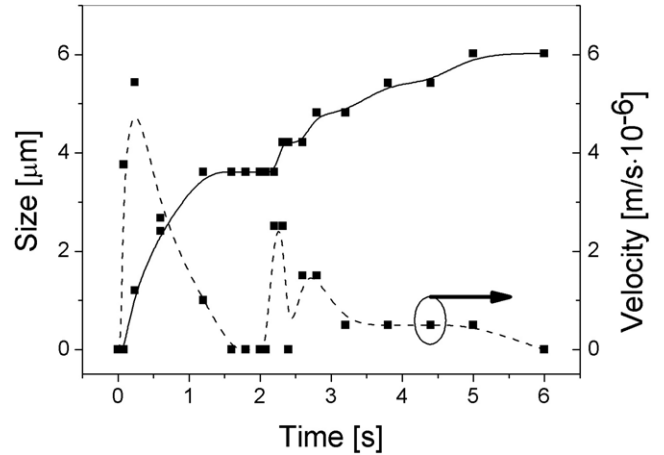


Figure 5. Sideways movement of the domain wall of an independently growing domain in the $[110]$ direction as a function of time upon application of $E = 2.8 \text{ kV cm}^{-1}$.

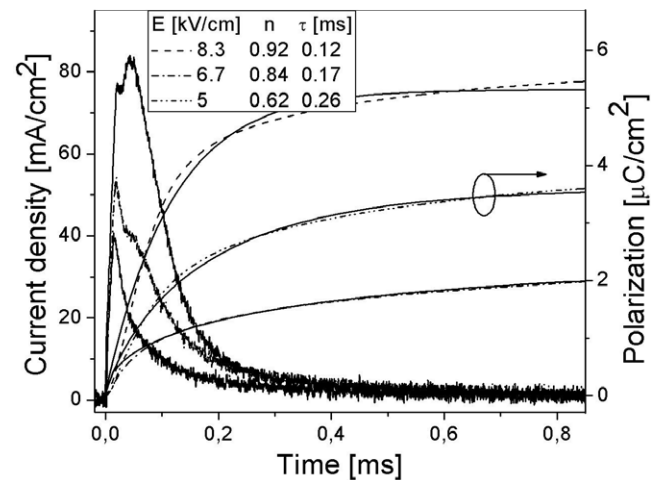


Figure 6. Switching currents and switched polarization versus time.

The switching current pulse $i(t)$ provides integral information about all randomly polarized regions. One can deduce the details of the polarization reversal process by analysing the shape of the current pulse. In normal homogeneous ferroelectrics the switching process proceeds slowly under low external fields and polarization can be fully switched even at low field, but after a sufficient waiting time. This means that the saturated polarization is independent of applied field. The switching process is strongly enhanced when the external field approaches the coercive field. For $E > E_c$, one current peak was observed, of several microseconds' duration [24].

In SBN crystals, unlike in homogeneous ones, the kinetics of polarization switching even in high fields $E > E_c$ is contributed to by fast and slow components. Thus a slow current tail with a time of many milliseconds is observed even at large fields, as presented in figure 6. One can presume that the main current peak (duration of about 0.2 ms), obtained at $E = 8.3 \text{ kV cm}^{-1}$, corresponds to domain nucleation and growth in the polar direction, and the subsequent decay to the slow sideways domain wall motion. In this time interval the

progressive slowing down of the domain walls is registered. Thus, the switching times in SBN crystals by several orders of magnitude exceeds the switching times in normal ferroelectric materials (microseconds), and may be accounted for by the relaxor origin of SBN crystals.

The rate of polarization switching at constant E can be found from the switching current $i(t)$ by integration of $i(t)$ from $t = 0$ to the instant t . The results are presented in figure 6. One can see that the saturation value of the switched polarization increases with the increase of the electric field. The observed partial ‘freezing’ of polarization may confirm the fact that there are slow switching regions that do not contribute to the switching current signals, even in $E > E_c$. This should be related to the hindering effect of the domain walls in SBN crystals. We have found that the switched polarization can be well fitted by a stretched exponential function

$$P(t) = P_0[1 - \exp(-t/\tau)^n], \quad (1)$$

with exponential coefficient n ranging from 0 to 1, commonly referred to as Kohlrausch, Williams and Watts (KWW) relaxation [27]. The fits are represented by the solid lines in figure 6. This type of relaxation was found to apply not only to ferroelectrics, but also to other forms of relaxation, including mechanical, magnetic relaxation, light scattering, and luminescence decay [28]. A stretched exponential behaviour with $0 < n < 1$ has been addressed as a dispersive transport or random walk process [28]. The values of the fitting parameters n and τ are presented in figure 6. The decrease in the exponent n , at lower electric fields, corresponds to a more stretched relaxation and is a reflection of a very broad distribution of relaxation times. It is interesting to note that the same relaxation law described the relaxing domains on a nanoscale, with PFM imaging of the domains’ configuration in SBN:61 doped with cerium [12]. Similarly, the same kinetics of relaxation was reported for a prototype relaxor $\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3$ (PMN) in field-induced linear birefringence measurements [4], as well as in KTaO_3 crystals doped with Li ions, using an optical second harmonic generation microscope [5].

4. Discussion

It is interesting to note that the polarization relaxation function $P(t)$, as described by equation (1), may also represent the switching kinetics in homogeneous ferroelectric crystals, according to the derivation by Ishibashi and Takagi (KAI model) [2]. In this case the index n varies from 1 to 3, and depends on the spatial dimension of a domain growth and on details of the nucleation and growth processes. The τ is the characteristic domain growth time. In the KAI model, it is postulated that there is a uniform nucleation probability and a constant domain growth velocity. The results obtained for SBN crystals show a strong qualitative disagreement ($n < 1$) with the switching kinetics predicted by the KAI model. This discrepancy results from the fact that the probability of nucleation and the rate of domain growth are affected by fluctuations of the random field in relaxor SBN crystals, which gives rise to local enhancement of the coercivity. The time

dependence of polarization, in the crystal sample examined, is well described by a KWW relaxation function. This has been discussed in terms of models where individual elements relax independently with an appropriately wide distribution of relaxation times [27]. Therefore, the observed $P(t)$ behaviour in the SBN crystal sample results from a superposition of many different relaxation times of relaxing domains. The spectra of the relaxation time distribution can be drawn from experimental data [8, 29].

The observed shape of the hysteresis loop in the doped SBN crystal is a further indication of a random-field model. Switching kinetics, in the crystal sample examined, show no relation to the crystal composition as is observed in crystals with defects. It is known from the literature that defects (e.g. dopes, impurities, and nonstoichiometric defects) can occupy energetically preferred sites in the lattice and form anisotropic centres which locally favour a certain direction of spontaneous polarization [30]. In such crystals asymmetric hysteresis loops about the D - E axes have been observed, with a built-in internal bias field, E_b , defined as a shift of the hysteresis loop on the E axis. The lack of E_b in the crystal sample examined can serve as direct verification of the concept of local random fields inherent to relaxor SBN crystals [7]. The fluctuations of the random field in SBN crystal bulk are too weak to influence entire domains or produce a macroscopic bias field, but are sufficient to stabilize the domain structure by pinning of the domain walls.

To summarize, the observed switching phenomena in SBN crystal could be understood in terms of the slow inhomogeneous domain nucleation in the presence of random pinning fields. According to Miller and Weinreich theory [31], the sidewise domain growth is a continuous nucleation alongside the existing 180° domain wall. A broad distribution of the heights of local pinning barriers should yield a distribution of activation energies for the nucleation of reversed steps on the existing domain walls. It will result in the distribution of the waiting times for local switching over different places of the crystal. The precise microscopic origin of the local disorder in SBN crystals is still to be determined. In pure SBN crystal the Sr^{2+} and Ba^{2+} cations occupy only five out of six possible sites in the unit cell, which results in a so-called unfilled structure of SBN [21], which may be responsible for charge disorder and relaxor behaviour. Doping SBN with trivalent Cr and Yb ions can enhance this charge disorder. Doping can induce space charge which in turn generates an internal field. We have recently shown [19] that electric field-induced charge carriers may compensate the sources of random fields, giving rise to depinning of the domain walls in pure SBN:33 crystal. Granzow *et al* [32] have reported that raising the sample conductivity by illumination eliminates the pinning centres and fully restores the polarization dynamics in cerium doped SBN:61 crystal.

It should be noted that domain patterns revealed by the NLC method may not thoroughly reflect the domain structure evolution in electrical switching at the same pulse amplitude. The results of the reported works show an influence of the interface (electrode and ferroelectric surface) conductivity on the conditions for compensation of the depolarization field

produced by bond charges and thus on the kinetics of the polarization reversal [24].

5. Conclusions

Microscale studies of the switching process enabled us to establish a relationship between the local properties of domain dynamics and macroscopic switching current responses. We present visual evidence that the domain nucleation and growth in doped SBN crystals is strongly affected by a random-field environment, related to the relaxor properties of this material. Polarization reversal, induced by external electric field, occurs in two distinct time regimes even in high fields exceeding the coercive field. The fast one (of the order of tenths of milliseconds) is dominated by inhomogeneous domain nucleation throughout the switching process and the slow one is related to sideways motion of domain walls, which proceeds through a pinning–depinning mechanism.

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