Localized Ferroelectric Domains via Laser Poling in Monodomain Calcium Barium Niobate Crystal

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Tightly focused femtosecond infrared optical pulses are employed to invert spontaneous polarization in the bulk of single-domain ferroelectric calcium barium niobate crystal. Such created localized ferroelectric domains could be arranged in 1D, 2D, and 3D patterns. The fabricated domain structures are subsequently used to demonstrate nonlinear diffraction via transverse second harmonic generation. This work constitutes the first realization of optically induced spatially localized ferroelectric domains in the bulk of a single domain ferroelectric medium and has a potential for applications in nonlinear light processing, frequency conversion, and others.

1. Introduction

Ferroelectrics, that is, media exhibiting spontaneous polarization below the Curie temperature[1] have found numerous applications in modern science and technology.[2] Majority of these applications stem from the ability of ferroelectrics to form ferroelectric domains, that is, regions of uniform orientation of spontaneous polarization. These domains have different orientations and coexist in the medium being separated by domain walls. Domain formation has been a subject of continuous research interest and investigations because of a number of actual and potential applications of domain structures in a variety of fields, including nonlinear optics[3] (e.g. frequency conversion, nonlinear volume holography), future domain walls electronics,[4,5] non-volatile memories,[6–8] photovoltaics,[9–11] etc. The ferroelectric domains may appear in the medium either spontaneously or be induced by external means. In the former case they arise via phase transition during the cooling stage of the crystal growing/or thermal processing and typically exhibit spatially random distribution in size and location.[12] In the latter situation, which is actually practically more relevant, domains form or/and switch in special conditions under external stimuli such as mechanical stress,[13] thermal processing,[14] temperature gradient,[15] electron,[16] or ion beam[17] and/or external or internal electric fields.[18–20] In fact, the use of electric field for domain formation and patterning is nowadays a method of choice.[18,21] In it, the ferroelectric crystal is placed between electrodes connected to high voltage power supply. When the electric field under the electrodes exceeds the coercive field, the initial spontaneous polarization can be reversed and the ferroelectric domain or domains of desired spatial form are formed. This so called poling technique has been used widely in the formation of domain patterns in commonly used ferroelectrics. However, because of the necessity of external electric field to be applied along the direction of spontaneous polarization, the electrical poling can be used predominantly for Z-cut samples. For atypical single-crystal orientations, such as X-cut lithium niobiate, special measures must be employed to facilitate electrical poling involving for instance, the use of ridge waveguide.[22] However, in this case the poling regions are typically restricted to shallow near surface layers.[21,23] Furthermore, some ferroelectrics like lithium niobate exhibit extremely high value of coercive field (tens of kV mm$^{-1}$)[24] restricting the technique to thin (hundreds of micrometers) samples. Finally, because of application of external electrodes, the domain pattern must always start at the surface and no isolated ferroelectric domain in the bulk of the crystal could be created. We have recently demonstrated new poling technique involving tight focusing of powerful laser pulses inside ferroelectric crystal. The nonlinear absorption of light in the focal volume leads to heating of the material. The subsequent spatial temperature gradient leads to the appearance of bipolar thermal electric field[25–27] which may invert locally spontaneous polarization if it is strong enough. The method is based on earlier demonstrated poling with UV light. However, unlike the latter which is
applicable to thin subsurface regions because of strong UV absorption, our approach allows for the operation deep inside the sample as the material is transparent for infrared light and the only absorption is nonlinear process taking place in the limited focal volume of the beam.\(^{[28,29]}\) As a result, the light induced poling can be applied to any crystallographic orientation and any location inside the medium. We have performed domain engineering in variety of crystals and demonstrated their application in transverse second harmonic generation,\(^{[30]}\) nonlinear volume holography,\(^{[31]}\) formation of 3D photonic crystals,\(^{[32]}\) as well. When illuminated by tightly focused laser beam, these charged wall regions contribute toward polarization reversal in a manner similar to that observed in laser filaments in LiNbO\(_3\).\(^{[20]}\)

However, it is not apparent what kind of anchoring centers could exist in monodomain ferroelectrics. Therefore, it is not obvious if nondestructive, controllable spontaneous polarization reversal inside the bulk of single domain crystals can be optically realized.

In this paper we show, for the first time, the light-induced reversal of spontaneous polarization and formation of fully localized 3D ferroelectric domains in a single domain calcium barium niobate (CBN) ferroelectric crystal. We show that for carefully chosen input power of infrared laser pulses, close to damage threshold, the tight focusing of optical beam leads to formation of isolated ferroelectric domains and their 2D and 3D arrangements deep inside the crystal. We demonstrate the existence of inverted ferroelectric domain pattern by employing it in the process of transverse second harmonic generation. Furthermore, we confirm that these are indeed domains and not local amorphization of the material, as reported elsewhere, by erasing the light induced domain pattern by heating the crystal above the Curie temperature. Finally, we confirm experimentally that the effective nonlinearity is indeed almost two times larger in the single-domain than in the multidomain crystal.

2. Results and Discussions

2.1. Ferroelectric Domain Patterning

The experiment contains two steps. The first is to convert a naturally grown CBN crystal with random domain structures into a single domain crystal. To this end, we used the electrical poling technique. The second step is to create periodic domain patterns in the single domain CBN crystal with a laser poling system, as schematically depicted in Figure 1. All relevant technical specifications of the system and experimental details are described in

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**Figure 1.** Schematic of the laser poling setup.
The hexagonal domain structures described above constitute the so-called nonlinear photonic crystals with spatially modulated (here periodic) quadratic nonlinearity in 2D and 3D, respectively.\,[32,41,45,46] Such nonlinear crystals offer an opportunity to phase match parametric processes along many directions allowing efficient power transfer among interacting waves. In this case, the periodic modulation of the second-order nonlinearity gives rise to the reciprocal lattice vectors which participate in the nonlinear interaction process enabling phase synchronization of the polarization and generated waves. This is so-called quasi phase matching (QPM) interaction widely used nowadays in parametric interaction in quadratic media\,[47,48] to ensure efficient and spatially controlled power exchange between interacting waves.

Below we illustrate the operation of our fabricated hexagonal 2D and 3D domain patterns as nonlinear photonic crystals, in the transverse second harmonic generation experiment. To this end each structure was illuminated by the loosely focused linearly polarized near infrared beam. The emitted second harmonic beams were projected on a screen and imaged by a CCD camera. The specifications of the fundamental beam and experimental details are described in the Experimental Section.

Figure 4 shows the second harmonic light intensity distribution for both, single- and multi-layered domain structures, as a
Figure 3. Čerenkov nonlinear microscopy images of a,c) 1-layer and b,d) multi-layer hexagonal domain structures. a,b) Top views and c,d) 3D visualization of parts of both domain patterns indicated by square selections in (a,b).

Figure 4. SHG patterns obtained for single layer (left) and five-layers (right) hexagonal structures. a–d) show the SHG patterns when the structures were pumped with fundamental beam with wavelength set to be 1150, 1300, 1450, and 1600 nm.
function of the wavelength of the incident fundamental beam. The transversely emitted SH acquires regular pattern which is known as nonlinear Raman–Nath diffraction \([49–51]\). We checked that the emitted SH is radially polarized what, for the ordinary polarized input beam, indicates the OO→E type of nonlinear interaction. The location of SH intensity peaks is determined by the transverse phase matching condition

\[
k_\text{transverse} \sin \theta = G_L
\]

(1)

where \(\theta\) and \(G_L\) denote emission angle of the second harmonic beam and the transverse component of the reciprocal lattice vectors, respectively. Obviously, because of hexagonal symmetry of the nonlinearity pattern, the second harmonic emission, exhibits hexagonal symmetry too. The intensity of the emitted SH beam depends on the longitudinal phase mismatch, which for fundamental beam propagating along \(z\)-axis is defined as

\[
\Delta_z = k_{2\omega} \cos \theta - 2k_\omega - G_z
\]

(2)

with \(G_z\) denoting \(z\)-component of the reciprocal lattice vector. As long as the longitudinal phase matching is not fulfilled, the second harmonic power oscillates in propagation, limiting conversion efficiency. In special situation when \(\Delta_z = 0\), the nonlinear Raman–Nath diffraction turns into nonlinear Bragg diffraction. In this particular case the power of the second harmonic grows monotonically with propagation \([51]\).

The emission of the second harmonics from the multilayer domain structure is clearly much stronger than that from the single layer. In fact it is roughly five times stronger. This is a result of longer propagation (and interaction) distance in the former structure. In our case the thickness of the single layer structure is roughly 26 \(\mu\)m while it is roughly 220 \(\mu\)m for multilayer pattern. Notice also that the nonlinear Raman–Nath diffraction pattern is rather nonuniform with lower orders barely visible for short wavelength of the fundamental wave (as in Figure 4a,b). This is a distinct indication of the nonlinear character of wave interaction. As the emission order increases, the longitudinal phase mismatch \((\Delta_z)\) gets smaller. When it approaches zero the nonlinear Raman–Nath diffraction becomes nonlinear Bragg diffraction at which the vectorial phase matching is fully fulfilled. When this happens the power of the generated harmonic grows monotonically with propagation distance enhancing these higher diffraction orders. When the wavelength of the fundamental beam increases the nonlinear Bragg diffraction takes place at smaller emission angles and, hence, the lower order Raman–Nath peaks experience enhancement (Figure 4c,d).

Even though the structures discussed in this work are relatively simple, our approach can be now used to create more complicated 2D and 3D ferroelectric domain patterns, which could generate second harmonic wave with nontrivial phase structures, for example, vortex or Bessel beams \([42]\).

As we mentioned earlier, the presence of sub-micron, random domains in the crystal leads to unwanted conical emission of second harmonic which may have detrimental effect on the quality of desired SH signal beam. We demonstrate this effect in Figure 5a, where we show second harmonic emission from a 2D hexagonal domain pattern fabricated in a multidomain sample. It is clear that randomness-induced Čerenkov emission frustrates the regular SH hexagonal pattern decreasing the effective contrast of the latter in the overlapping region. Furthermore, we found that the regular, hexagonal SH signal constitutes only 10% of the total SH emission and the majority of the generated SH power goes into spurious Čerenkov background. However, note that the Čerenkov background builds up over the whole sample, while the desired signal was generated from 25 \(\mu\)m-thick hexagonal domain pattern. Therefore, the power of SH signal can be significantly increased by using thick domain structures and operating at the resonance. For instance, the SH signal reached 30% of total emitted SH power in our five-layer hexagonal structure. It should be stressed, though, that while the power of SH signal can be increased using multilayer 3D domain structures and operating at the resonance, the emission will be always accompanied by the inherent noisy SH background.

For comparison, Figure 5b depicts the SH emission from the 2D hexagonal domain pattern fabricated within a single domain crystal. In both cases, the same domain reversal conditions were employed. Now the Čerenkov background emission is absent ensuring high contrast of the SH signal.

By comparing the conversion efficiency in both cases, we can determine the effective quadratic nonlinearity in the nonlinear diffraction in multidomain crystal. This quantity is expected to be effectively two times smaller than that in a single domain sample, as the random sub-micron domains will effectively not contribute to the emission process induced by macroscopic periodic structure of poled regions. To this end we restricted ourselves to the resonant Čerenkov emission, which is fully phase matched, and when the SH intensity grows monotonically with propagation. In this regime the emitted SH power is directly proportional to the second power of the effective nonlinearity. However, it is impossible to separate in the experiment the SH emission from random and periodic domain structures in the multidomain medium. Hence, we numerically subtracted the Čerenkov background from the data of Figure 5a and compared the resulting SH power with that emitted in the monodomain sample (Figure 5b).

We found that the SH signal in the single domain sample was over five times larger than that generated in multi-domain sample. This corresponds reasonably well to the expected theoretical value of 4. The discrepancy is most likely caused by the use of numerical processing of the emission data in Figure 5a.
as well as unavoidable differences in both fabricated structures, such as duty cycle, domain length, etc.

3. Conclusion

We used tightly focused short pulse infrared beam to locally invert spontaneous polarization in the bulk of single domain ferroelectric calcium barium niobate crystal. We created 2D and 3D hexagonal patterns of inverted domains deep inside the crystal. These domain patterns, which constitute 2D and 3D nonlinear photonic crystals were subsequently used in second harmonic generation experiment via the nonlinear Raman–Nath diffraction. The laser inverted localized domain appear to be stable surviving for a period of 3 months even under the illumination by strong, loosely focused, infrared beam. We confirmed that the fabricated quadratic nonlinearity patterns are indeed ferroelectric domains and not structural modification of the medium, by completely erasing them via thermal annealing above Curie temperature. Our results open up possibilities to fabricate complex multi-dimensional QPM domain structures inside single domain ferroelectrics allowing their integration with other ferroelectrics based devices such as phase modulators and switches.

4. Experimental Section

Electric Field Poling: The process started with a slab of Z-cut calcium barium niobate crystal (Ca$_{12}$Ba$_2$Nb$_2$O$_{23}$) with a size of 5 x 5 x 0.5 mm$^3$. This was as grown sample so it contained randomly distributed microscopic size oppositely oriented ferroelectric domains. To convert it into a single domain crystal electrical poling technique was used. To this end, two liquid carbon electrodes roughly 2 x 2 mm$^2$ in size were painted on both Z-sides of the crystal. Then the electric voltage of 200 V was applied to the electrodes while the sample was kept at 200°C for 30 min. As shown in (52), the coercive field of CBN crystal decreases with temperature and at 500 K was around 150 V mm$^{-1}$, so the 400 V mm$^{-1}$ used in the experiment should be enough to achieve poling. After the crystal cooled down to room temperature the electrodes were removed and the sample was cleaned. To evaluate the quality of the poling process simple experiment of second harmonic generation was resorted to. To this end, the crystal was illuminated by loosely focused femtosecond beam at 1200 nm and the emitted second harmonic signal at 600 nm was observed behind the sample. When the input beam illuminated the virgin multi-domain region of the sample, conical emission of second harmonic light could be observed. This, so called Čerenkov second harmonic emission (44-51) is very well known to originate from the sharp modulation of quadratic nonlinearity, provided by the presence of random domains. However, as soon as the beam illuminated the region subjected to external poling field, the Čerenkov emission disappeared completely confirming uniform orientation of the spontaneous polarization.

Laser Poling: The uniform, single domain region of the CBN crystal described above was then used in the main experiment on laser induced localized domain formation. The schematic of the laser domain reversal setup is depicted in Figure 1. The sample was mounted on a 3D motorized stage with positioning accuracy ~100 nm. As a light source, Chameleon Ultra II (Coherent) was used operating at 730 nm, which delivered train of 130 fs long pulses with a repetition rate of 80 MHz. An optical shutter was used to block and unblock the laser beam during the writing process. The laser beam (initial diameter of 1 mm) was incident onto the Z facet of the sample and subsequently tightly focused inside the crystal, below its surface, by a ×50 microscope objective (NA = 0.65). The actual steps in the laser domain reversal process were as follows. First the sample was adjusted such that the focus of the writing beam was located at the desired depth for given layer of the pattern. Then for every spatial location of the focus, the shutter was held open for a specific time. During that time the sample was translated along the z axis, with constant speed, for a distance of 10 µm. Then the laser light was blocked, the sample was translated by the stage in X–Y plane to a next location where the domain formation process was repeated. The change of the location of the focus in transverse dimension followed the hexagonal pattern whose transverse size was 70 x 70 µm$^2$. By illuminating the z-cut sample with tightly focused femtosecond beam, and scanning it in 3D, either single layer (2D) or multilayer (3D) domain patterns could be written. It is known that the focusing in the high refractive index material such as CBN crystal (n=2.294 @730 nm), is accompanied by deterioration of the beam focus which, in the most drastic case, leads to focus splitting. (53) In order to account for changes in the light intensity in the focus, the input beam power was adjusted with the help of a motorized half wave plate and a polarizer, so the same laser processing conditions could be maintained at each writing depth. The entire laser-induced domain reversal process was automatically controlled by computer running a MATLAB script. In addition, an imaging system consisting of CCD camera, a beam splitter and an LED illuminator was used to monitor the processed area of the sample. The single layer ferroelectric domain structure was fabricated at the initial depth of 46 µm below the surface of the sample. The input laser power was 385 mW and the writing speed (along the z-direction) was 10 µm per second. Clearly a very regular domain pattern was formed. As the sample was translated 10 µm in the direction of the beam, the inverted domains attained a length of 26 µm which was close to the designed translation of the focal spots in the sample (about 23 µm). For the multilayer domain structures, the very bottom layer was formed at a depth of 252 µm in the crystal. The writing speed was again 10 µm s$^{-1}$ while the input power was 505 mW. However, each subsequent layer was written with lower power, of 465, 420, 400, and 385 mW, respectively to account for spherical aberration modifications to focal intensity. After one layer has been completed, the sample was translated vertically by 10 µm with laser beam off, and then the next layer was written. Hence the structure was periodic also along the z-direction.

Čerenkov Nonlinear Microscopy: The Čerenkov microscopy is a nondestructive technique of 3D visualization of spatial modulation of quadratic nonlinearity. It is based on the conical emission of the second harmonic induced by tightly focused fundamental beam. The generated second harmonic signal becomes very strong in the region of sharp modulation of quadratic nonlinearity and, in particular, at the domain walls. Scanning the sample with focused fundamental beam along x,y,z directions, and recording the Čerenkov signal, allows one to obtain 3D map of domain pattern in the crystal. (44)

Second Harmonic Generation: As a light source, Chameleon Ultra II (Coherent) which is pumping synchronous compact optical parametric oscillator (Coherent) was used. The system produced beam consisting of train of 130 fs long pulses with a repetition rate of 80 MHz which were used to generate non-collinear SHG signal from the hexagonal lattice patterns. The fundamental wavelength could be tuned from 1050 to 1600 nm. The linearly polarized beam was loosely focused (using a ×4 microscopic objective, NA = 0.1) on the structure. The input power was set to 250 mW for every wavelength used. The nonlinear interaction in the ferroelectric domain structures led to the emission of the second harmonic wave which was projected on a screen and imaged by a CCD camera.

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Conflict of Interest

The authors declare no conflict of interest.
Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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