Electron beam domain patterning of MgO-doped lithium niobate crystals covered by resist layer

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The electron beam domain patterning of MgO-doped congruent lithium niobate (LN) single crystals covered by electron beam resist layer were studied for various layer thicknesses and electron accelerating voltages. The obtained domain patterns were divided in four types. The increasing of the domain structure quality under presence of the resist layer on irradiated surface was confirmed. This effect was attributed to high concentration of the electron traps in resist, which localized incident electrons in limited volume over LN surface and formed effective electrode. The voltage dependence of space charge localization relative to the LN surface was studied by computer simulation.

Keyword: electron beam patterning, domain structure, lithium niobate, regular domain structure, electron beam resist

Short title: Electron beam domain patterning of MgO:CLN

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1. Introduction

The periodically poled lithium niobate (PPLN) crystals with stripe domains are widely used for different nonlinear-optical [1] and photonic applications [2]. Several poling methods have been developed for creation of the regular domain structures [1, 3-25]. The creation of the stable precise domain structures with reproducibility of the domain period about tens of nanometers allows using the quasi-phase-matching effect for frequency conversion of the laser irradiation [1]. Thus, the main efforts are directed to creation of precise periodic structures with micron and submicron periods needed for manufacturing of nonlinear-optical devices with improved characteristics [26-29]. The most popular method of creation of such periodic structures is electric field poling using photolithography-defined stripe electrodes at the polar surface [1, 3-5]. At the same time, electric field poling encounters several obstacles, such as necessity of application of high voltage, using elevated temperatures, domain broadening outside of electrodes, and spontaneous backswitching after field switch off [6].

This was the reason for developing alternative methods, such as scanning methods including tip-induced domain patterning by scanning probe microscope (SPM) [7-9] and electron beam patterning (poling) (EBP) [10-25]. The SPM poling allows achieving the periods about 100 nm in thin plates with simultaneous visualization of the domain structure with high spatial resolution by piezoelectric force microscopy. In spite of advantages, SPM poling is very slow and the poling area is limited.

EBP method looks more preferable as it is essentially faster and more compatible with semiconductor device processing [22]. The electron beam size, easily scaled down to few nanometers, allows discussing the ability of creation of the nanoscale domain structures. However, the best experimentally achieved resolution for 2D domain structure in lithium niobate crystals, obtained by irradiation of Z^--polar surfaces, is about one micron [19]. Nevertheless, the crystal irradiation by focused electron beam is still considered as one of the

promising methods for nanodomain engineering. EBP has been applied for creation of 1D and 2D domain gratings in $LiNbO_3$ (LN) [10, 11, 13-23, 25]. The subsequent selective etching of engineered domain structures allowed fabricating 2D patterned surface structures for photonic crystals [17].

Usually applied EBP is realized by irradiation of Z^--polar surface controlled by means of electron-beam lithography system, when Z^+-surface is coated by grounded conductive electrode. Such method cannot produce domain patterns with acceptable uniformity and regularity due to dispersion of the beam electrons over irradiated surface and in the crystal bulk caused by interaction between the initially accumulated electrons in the surface layer and the ones incoming later [22].

Several attempts have been made recently for poling by irradiation of Z^+-surface [24, 25]. The submicron domains were achieved, but the structure period was rather irregular due to inhomogeneous charging and electrostatic interaction between domains [25].

Improvement of domain patterning by EBP has been achieved by coating of irradiated Z^--surface by thin insulating surface layer [21-23]. High trap concentration enables localization of primary electrons in the insulating layer thus reducing the electron dispersion over the surface and in the bulk. The proposed approach allowed producing the domain patterns with essentially better quality [22, 23].

Glickman et al. [21] applied the indirect EBP method for two-dimensional rectangular periodic poling of LN. They used 2 μm dielectric layer of Shipley S-1818 photoresist and accelerating voltage of 15 kV, with charge dose of 500 $\mu C/cm^2$. The formation of clusters of isolated micron dots was demonstrated on the Z^--surface in each irradiated region of square lattice with periods above 10 μm. The length of non-through domains reaches 350 μm. Authors pointed out that their method suffered from charging of the resist layer, which limited the spatial resolution.

Li et al. [22] demonstrated the fabrication of uniform regular domain structures using electron beam (e-beam) resist layer for EBP on the Z^--surface of stoichiometric $LiTaO_3$ (SLT) single crystals. The samples thickness 25 and 250 μm and e-beam resist ZEP520-22 (Zeon Corporation, Japan) have been used. It was shown that increasing of resist thickness led to increase of pattern regularity, reduction of threshold voltage and beam current by one order, which improved the fabrication rate [22]. The successful switching of MgO:SLN crystals, which was believed to be unsuitable for domain patterned by electrical poling due to large bulk conductivity, has been achieved [23].

We have studied EBP in the MgO doped LN crystals of congruent composition (MgO:CLN). LN exhibits unique electro-optical, pyroelectric, and piezoelectric properties combined with good mechanical and chemical stability, and wide transparency range and high optical damage threshold [30, 31]. These properties and the existence of only 180° ferroelectric domains make LN well-suited for numerous applications for different acoustic, piezoelectric, and nonlinear-optical devices, such as electro-optic lenses [32] and frequency converters [27, 33]. Doping by MgO increases essentially the photorefractive damage threshold [34, 35]. It is necessary to point out that the electric fields appeared during polarization reversal in the surface layer of ferroelectric crystals leads to electron emission under the action of depolarization field [36].

In this work, we represent the experimental study of the resist assisted electron beam domain patterning of MgO:CLN for the various thicknesses of resist layer and electron accelerating voltages.

2. Experimental

The studied samples representing Z-cut optical grade 1 mm thick CLN single crystalline wafers, grown by Czochralski method and doped by 5% weight of MgO

(MgO:CLN) were produced by Yamaju Ceramics, Japan. The sputtered solid 100 nm thick Ta electrode deposited on Z^+-polar surface was grounded during electron beam irradiation.

The Sawatec SM 180 spin coater was used for deposition of negative AZ nlof 2020 (Microchemicals, Germany) photo- and electron beam resist with nominal thickness from 0.5 to 1.5 μm on Z^--polar surface. The resist layers were soft baked at temperature 80°C for 3 minutes with cooling/heating rate below 3°C/min.

The polarization reversal was performed by irradiation of Z^--polar surface using workstation Auriga CrossBeam (Carl Zeiss NTS, Germany) with electron beam generated by the Schottky field emission gun. The exposure parameters and electron beam positioning are driven by electron-beam lithography system Elphy Multibeam (Raith GmbH, Germany). The regular grating of 10 stripes with 10 μm period, 100 μm length, and 0.5 μm width was used as a writing design. The design has been specified by Raith Nanosuite software. The dose was defined as $D_S = I \times t/S$, where t is exposure time and S is area. The sample was irradiated by different doses at fixed U and I.

After the sample exposure to electron beam, the post exposure bake was performed with temperature 80°C for 5 minutes with cooling/heating rate below 3°C/min. The resist was developed in AZ726 MIF developer (Microchemicals, Germany) for 30 to 60 s depending on the thickness.

The static domain structures formed after polarization reversal were visualized by optical microscope (Olympus BX-51, Japan) after chemical removal of the electrodes. The domain structures were revealed by selective chemical etching during 90 s in pure hydrofluoric acid (HF) at room temperature [37]. The surface relief corresponding to the domain structure was visualized by optical microscope in dark field mode with estimated lateral resolution about 300 nm.

3. Results and discussion

3.1. Polarization reversal for various thicknesses of resist layer

The influence of the resist layer thickness and accelerating voltage on the domain structure was studied in two stages. At the first stage, the accelerating voltage was fixed and the resist layer thickness was varied. Electron beam irradiation was carried out for various thicknesses of e-beam resist (0.5, 1, and 1.5 μm), fixed charge dose (1500 $\mu C/cm^2$) and accelerating voltage (10 kV). The chosen value of accelerating voltage corresponds to the minimal value needed for domain formation during irradiation of uncoated Z^- polar surface [15].

The best structures of mostly continuous stripe domains on both polar surfaces were obtained for 1 μm thick resist layer (Fig. 1c, d). For 0.5 μm thick layer, the dashed domain lines with rough walls appeared at Z^- surface and essentially irregular broadening of domain stripes was observed at the Z^+ surface. Formation of microdomain ensembles along irradiated lines was revealed at both polar surfaces for 1.5 μm thick layer. It should be noted that obtained hexagonal shape is typical for isolated domains appeared in LN and stoichiometric lithium tantalate under equilibrium switching conditions [38, 39, 40].

The obtained results confirm improvement of the domain structure quality for the resist layer on irradiated surface [22, 23]. This effect was attributed to high concentration of electron traps in the e-beam resist, due to localization of the incident electrons in limited volumes over the crystal surface (Fig. 2b) compared with uncovered surface of LN (Fig. 2a). Moreover, the resist layer allowed reducing the electron beam reflection due to suppression of charging effect.

During exposure, the electrons accumulated in the resist over the surface produced the electric field, which exceeded the threshold value for sufficiently high dose and stimulated

polarization reversal. The position of the accumulated space charge depended on the resist thickness.

The computer simulation of primary electron scattering in the target volume was carried out using free software Casino v.2.4.8 based on the numerical Monte Carlo algorithm [41]. The simulated target represented a resist layer with thickness ranged from 0.5 to 1.5 μm deposited on LN plate. The simulated paths of primary and backscattered electrons for accelerated voltage 10 kV and various resist thicknesses are presented in Fig. 3.

For 0.5 μm thick resist, the electrons penetrate through resist to the substrate and form a space-charge region located mostly below the resist/LN interface (Fig. 3a). Thus, the polarization reversal and the formation of the domain structure in this case should be similar to results of irradiation without resist [18].

In the case of irradiation of the target with 1 μm thick resist, most of the electrons located near the resist/LN interface (Fig. 3b). The electrons localized in the resist formed an effective electrode, which generated the switching field. The domains started to grow, when the switching field reached the threshold value. The primary electrons and currents along the conductive charged domain walls screened the changes of depolarization field appearing during polarization reversal [42].

For 1.5 μm thick resist, most of the primary electrons do not reach the interface and remain in the resist layer (Fig. 3c). In this case, the polarization reversal is similar to the switching with artificial dielectric layer and is characterized by ineffective compensation of depolarization field by slow bulk screening [43, 44]. As a result, the so called discrete switching occurs leading to formation of the microdomains ensemble (Fig. 1e, f) [43-45]. The formation of similar domain has been demonstrated after irradiation of LN with thick resist layer [21].

3.2. Polarization reversal for various accelerating voltages

Dependence of the domain structure on the accelerating voltage was investigated for fixed 1 μm thick resist and the voltage ranged from 5 to 15 kV. The typical domain images are shown in Fig. 4.

Increasing of the accelerating voltage leads to higher energy of the primary electrons, resulting in longer path of the primary electrons and localization of the space charge deeper in the resist. Thus, the irradiation with various accelerating voltages leads to the similar effects as for various thicknesses of the resist layer. At an accelerating voltage below 10 kV, the space charge is localized in the resist layer, while for voltage above 10 kV – in the vicinity of the interface and in the crystal bulk.

3.3. Polarization reversal for various doses

The formation of the stripe regular domain structures was studied in details for fixed 1 μm thick resist at accelerating voltages ranged from 7 to 13 kV and the irradiation doses ranged from 500 to 2500 $\mu C/cm^2$.

All obtained domain structures can be divided into four types: (I) chains of isolated microdomains (Fig. 5a), (II) "dashed" domain stripes consisting of isolated hexagonal and elongated domains (Fig. 5b), (III) solid stripe domains (Fig. 5c), and (IV) isolated hexagons and merged domain stripes (Fig. 5d).

The obtained results allowed us to reveal the optimal thickness of the resist layer (about 1 μm) and electron acceleration voltage (about 10 kV) for creation of the regular domain pattern by electron beam irradiation. It should be noted that the resist layer essentially decreases the threshold acceleration voltage down to 5 kV, as compared with the conventional 10 kV [15]. This fact was revealed also in SLT [22] and can be attributed to

better charge localization.

4. Conclusion

The domain patterning by electron beam irradiation of the polar surface covered by resist layer has been studied in single crystalline MgO:CLN. It has been shown that the quality of the regular domain patterns depends on the thickness of resist layer and electron accelerating voltage. Increasing of the voltage has led to the same effects as decreasing of the thickness of resist layer. All obtained domain structures can be divided into four types: (I) chains of isolated microdomains, (II) “dashed” domain stripes consisting of isolated hexagonal and elongated domains, (III) solid stripe domains, and (IV) isolated hexagons and merged domain stripes. The voltage dependence of spatial distribution of space charge localization relative to the LN surface has been studied by computer simulation. The obtained results confirm improvement of the domain structure quality for the case when resist layer is deposited on irradiated surface due to suppression of the charge spreading occurring during irradiation of free surface. This effect was attributed to localization of the incident electrons in confined volumes over the crystal surface in the electron beam resist with high concentration of electron traps.

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Figure captions

Fig. 1. Domain structure obtained by irradiation of Z^--surface of MgO:LN crystals covered by resist with thickness: (a), (b) 0.5 μm, (c), (d) 1 μm, and (e), (f) 1.5 μm. Dark field optical images of domains revealed by chemical etching on (a), (c), (e) Z^- surface, and (b), (d), (f) Z^+ surface.

Fig. 2. Scheme of electron beam irradiation: (a) without resist; (b) with resist.

Fig. 3. Computer simulation of the primary electron scattering in the target. Blue – path of the primary electrons, red – path of the backscattered electrons. The horizontal dashed line – the boundary between the resist and LN. Thickness of resist: (a) 0.5 μm, (b) 1 μm, and (c) 1.5 μm.

Fig. 4. Domain structure induced by electron beam irradiation of Z^--polar surface of MgO:CLN crystal covered by 1 μm-thick resist layer. Irradiation dose of 1500 μC/cm^2. Accelerating voltage: (a), (b) 8 kV; (c), (d) 10 kV; (e), (f) 12 kV. Dark field optical images of domains revealed by chemical etching on (a), (c), (e) Z^- surface; (b), (d), (f) Z^+ surface.

Fig. 5. Typical domain structures induced by electron beam irradiation of Z^+ polar surface of MgO:CLN crystal covered by 1 μm-thick resist layer. (a) U = 7 kV, dose 1000 μC/cm^2; (b) U = 8 kV, dose 1500 μC/cm^2; (c) U = 10 kV, dose 2000 μC/cm^2; (d) U = 12 kV, dose 1000 μC/cm^2. Dark field optical images of domains revealed on Z^- surface by chemical etching.