TUNABLE DIFFRACTION GRATING WITH TRANSPARENT INDIUM-TIN OXIDE ELECTRODES ON A LITHIUM NIOBATE X-CUT CRYSTAL

V.D. Paranin¹, S.V. Karpeev^{1, 2}, K.N. Tukmakov¹, B.O. Volodkin¹

¹ Image Processing Systems Institute of RAS, – Branch of the FSRC "Crystallography and Photonics" RAS, Samara, Russia, ² Samara National Research University, Samara, Russia

Abstract

A tunable diffraction grating based on an electrooptic X-cut lithium niobate crystal has been manufactured and experimentally analyzed. The period of electrodes is 290 μ m, the electrode width is 117.5 μ m, and the thickness of an electrode is 150–160 nm. The electrodes are made of a transparent conducting indium-tin oxide that serves as an antireflection coating with the aim of increasing the optical transmission. In order to prevent crystal polarization switching and electrical breakdown an optimized electrode topology with end ellipticity 1:1 and increased interelectrode gap is used.

The optical diagram of the tunable grating with alternating electrode potentials for various gap voltages is analyzed. The intensity of the zero order of diffraction is shown to decrease by 40% at a voltage of 800 V. At the same time, the origination of new diffraction orders at angles $\pm \lambda/(2d)$ is noted. The measurement of the forward-bias and reverse-bias regions of the modulation characteristic reveals the absence of hysteresis, which confirms the correctness of the electrode topology design.

Keywords: tunable diffraction grating, linear electro-optical effect, lithium niobate.

<u>Citation</u>: Paranin VD, Karpeev SV, Tukmakov KN, Volodkin BO. Tunable diffraction grating with transparent indium-tin oxide electrodes on a lithium niobate X-cut crystal. Computer Optics 2016; 40(5): 685-688. DOI: 10.18287/2412-6179-2016-40-5-685-688.

<u>Acknowledgements</u>: The work was funded by the Russian Foundation for Basic Research (RFBR grants 16-29-11698 and 14-02-97033 r_povolzhiye_a).

Introduction

Tunable diffraction gratings based on the electrooptical effect remain the subject of various studies. Part of publications is devoted predominantly to synthesis and application of highly sensitive electrooptical materials for the production of substrates [1-7]. In other studies more attention is given to the design of control electrodes. Thus, in papers [7, 8] a three-element electrode is discussed that forms a quasitriangular phase profile of the grating causing deflection of transmitted light. This approach, however, can be applied only to thin electrooptical layers comparable to the interelectrode gap of, for instance, liquid crystals. In the case of thick crystals (about 1 mm) the electric field of the electrodes is concentrated near the surface they are located on and the interaction of the lower and upper electrodes is insignificant.

In [9] improvement of transmission of the tunable grating due to the use of transparent 130 nm thick indium-tin oxide In_2O_3 -SnO₂ (ITO) electrodes is discussed. According to the results presented in the specified paper the resistivity of ITO-electrodes is quite high, approximately 48 Ω /sq. This fact caused the necessity of evaporating narrow aluminum stripes on top of the ITOelectrode. This approach complicates the fabrication process, it causes origination of higher diffraction orders and undesirable scatter of optical light. It is necessary to reduce the resistance of indium tin-oxide by maintaining stoichiometry of the film in the process of evaporation or at the postprocessing stage.

It should be noted that the design of electrodes determines the processes of polarization switching of the lithium niobate surface layer (with the electrical field higher than coercive). These effects can be observed mostly in the vicinity of rectangular electrode ends and to a smaller degree in interelectrode gaps [10]. Origination and growth of needle-shaped domains determines the hysteresis of tunable gratings and reduces the diffraction efficiency due to the depolarization of transmitted light [11].

The aim of the study was to develop improved electrode topology characterized by improved optical transmission due to the use of transparent indium-tin oxide. Other tasks included reduction of the film resistance and optimization of electrode form and dimensions to prevent polarization switching of the crystal surface layer.

1. ITO tunable grating technology

Polished chips of congruent lithium niobate with the dimensions $15 \times 15 \times 1$ mm were used as substrates. An oxide of indium-tin alloy of electronic grade deposited in vacuum by magnetron sputtering served as the material of electrodes.

The substrates were cleaned by rinsing in an alkaline solution, soaking in a chrome solution H_2SO_4 :K₂CrO₇ for 5 minutes and rinsing in deionized water. The lift-off process was used to produce a diffraction mask. A layer of FP-051K photoresist 0.4 mkm thick was spindeposited in two stages: 20 seconds at the rate of 500 rev/min, and then 45 seconds at the rate of 4500 rev/min. The photoresist was air-dried for 2 minutes and then dried on a heater for 12 minutes at the temperature 95 °C. Immediately after the inverse photomask exposure the photoresist was developed in UBP-1F developer solution for 1 minute, rinsed in deionized water and dried by pure dried air flow.

In order to accomplish vacuum deposition of the indium-tin layer the substrates with the photoresist inverse mask were fixed on a heating substrate holder of the ETNA-100-MT vacuum unit. Reactive sputtering in oxygen environment with additional heating is necessary for successful formation of indium-tin oxide. The temperature of heating is limited by photomask degradation in such aggressive conditions. It was experimentally established that heating up to the temperature of 200 °C in the presence of argon-oxygen mixture does not cause any significant degradation of the photoresist used and the resulting mask is completely washed off with acetone at a later step.

After evacuation to marginally hard vacuum of the order of 10^{-4} Pa heating of the substrate holder was switched on with the help of a resistive helix. The temperature was tunable by an automatic PID-regulator. The rate of temperature growth did not exceed $10 \,^{\circ}\text{C}/\text{min}$ and the nominal temperature of the substrate holder was not higher than 200 °C. This temperature was maintained during the whole process of ITO deposition.

The process of sputtering itself started with spraying of the upper layer of the target material, indium-tin alloy of electronic grade (target diameter 3") onto the closed shutter under the following conditions: atmosphere - argon with the chamber pressure 0.5 mTorr, the rate of supply through the magnetron $-9 \text{ cm}^3/\text{sec}$; the magnetron power increased up to 200 W in the course of 30 seconds and then was maintained at this level for 5 minutes in direct current conditions. Let us note that the closed magnetron shutter and the supply of gas from the bottom of the magnetron determined higher argon working pressure in the magnetron as compared to that in the main chamber, which ensured stable operation of the magnetron.

After the target was cleaned the shutter was opened, the flow of argon through the magnetron increased up to 22 cm^3 /sec and oxygen was additionally supplied to the substrate at a rate of consumption $9 \text{ cm}^3/\text{sec}$ (the pressure in the chamber therewith grew up to 1.1 mTorr). The magnetron power decreased down to 110 W and its operation condition changed to the intermittent current mode with a periodic inverted pulse to minimize the effect of anode loss. The film thickness was measured with the help of a quartz thickness gage using a check test piece placed near the samples. The rate of ITO oxide formation on the test piece is somewhat different, consequently, the film density may also differ from that on the sample film. Therefore, to control the process the rate of film growth was used (it amounted to 0.26 nm/sec), the constancy of which meant the process was stable.

To improve the conductivity of the ITO-film deposited a higher temperature $(300-500 \,^{\circ}\text{C})$ is required [13–14]. Therefore, additional annealing was used after the completion of the lift-off process. The crystals were air heated with the help of a heater at a rate of 30 $^{\circ}\text{C}$ /min from 20 $^{\circ}\text{C}$ to 250 $^{\circ}\text{C}$, which was maintained for 2 min. Both the crystals and the heater were cooled down to room temperature by air convection. Annealing resulted in considerable decrease of the film surface resistivity from 50 Ω /sq to 26 Ω /sq.

The refraction index of the ITO-film was measured by the method of ellipsometry using a J.A. Woollam V-VASE automatic spectral ellipsometer. The wavelength was assigned to be equal to 632.8 nm, the width of the light spectrum did not exceed 2.3 nm. To reduce the impact of reflection from the back surface of the substrate it was mat-finished. The refraction indices determined with the help of the ellipsometer software were as follows: $n=1.8841\pm0.0027$, $k=0.0176\pm0.0030$ according to the data of *p*-polarization.

A tunable diffraction grating made of indium-tin oxide on X-cut lithium niobate was formed in the way described. Fig.1 shows a fragment of topology in the form of interdigital electrodes that ensure alternating distribution of potentials of the 0V0V kind. The coordinate axis z corresponds to the crystal optical axis. The electrode tips are rounded so as to reduce maximum intensity of the electric field E_z acting along the optical axis. The impact of the field results in the origination of microdomains that change the crystal's optical properties and contribute to electrical breakdown of the interelectrode gap [10, 11]. As modeling showed [12], tip ellipticity 1:1 yields twofold reduction of the maximum field E_z .



Fig. 1. Topology of a tunable diffraction grating

The electrode dimensions were measured using a Biomed-5P microscope and a WLI-DMR white light interferometer. The period was 290 µm, the electrode width was 117.5 µm, the thickness was 150..160 nm. The film thickness and refraction index determined approximately similar transmission of the electrodes and the substrate at the wavelength 632.8 nm, equal to 83-84 % with account for the influence of acrylic layer on the output surface. The roughness of the electrodes corresponded to the roughness of a polished substrate $R_a < 1..2$ nm.

2. Experimental study of ITO tunable grating

The optical diagram of the ITO-grating was analyzed with the help of an optical setup that comprised a heliumneon laser, a spatial filter- beam expander, a Glan-Taylor polarizer, an experimental sample, an image-forming cylindrical lens and a CCD-matrix. The illumination of the tunable diffraction grating was close to uniform. The negative photos of diffraction orders for various voltages are shown in fig. 2.

It follows from the data obtained that a diffraction grating on the basis of indium-tin oxide is an amplitude– phase one. The fact is confirmed by the presence of the first diffraction orders in the absence of voltage. As the electrical voltage increases the intensities of the zero and first diffraction orders decrease. At the same time the intensity of new ± 01 orders located between the zero and the first ones at the angles $\pm \lambda/(2d)$ increases. The dependence of the zero order intensity on voltage was measured with the help of a PM100D secondary converter equipped with a temperature-compensated S120C sensor. To improve spatial resolution quartz fiber with the core diameter $62.5 \,\mu\text{m}$ was used. The center of the fiber was made coincident with the zero diffraction order image with the help of a positioning stage by the maximum value of intensity. In fig. 3 the measured normalized modulation characteristic is shown.



Fig. 2. Diffraction orders of a tunable grating at different voltages: a) 0 V, b) 400 V, c) 800 V



on electrical voltage

The modulation characteristic is of the typical kind $I = \cos^2(k_U U)$ where k_U is a constant determined by the electo-optical material, the electrode period and width. The analysis of the forward-bias and the reverse-bias regions of the characteristic revealed absence of hysteresis observed earlier [11]. This is due to the fact that the electrodes are rounded, which reduces maximum of the electric field near the electrode ends, as well as to a greater interelectrode gap (172.5 µm instead of 140 µm in [11]). Since crystal polarization switching is a threshold process domains of the kind of optical nonuniformities do not originate. The analysis of the tunable grating by the polarization optical method confirmed absence of microdomains after carrying out experiments in the vicinity of electrode ends and in the interelectrode gap.

Conclusion

A tunable diffraction grating with improved topology of control electrodes has been developed and experimentally analyzed. Intensity of diffraction zero order is shown to decrease by 40 % at interelectrode voltage of 800 V. A two-fold increase of optical transmission is achieved at the operating wavelength 632.8 nm due to the use of transparent conducting indium-tin oxide of half-wave thickness instead of chrome-copper electrodes [10, 11]. Electrode's surface resistivity is shown to decrease from $50 \Omega/sq$ to $26 \Omega/sq$ in the case of air annealing at $250 \,^{\circ}C$ for 2 min. The intensity of the electric field is reduced due to the increase of the interelectrode gap from 140 μ m go 172.5 μ m and rounding of electrode ends in the 1:1 proportion. Formation of needle-shaped domains in the crystal surface layer and hysteresis of dependence of diffraction order intensity on voltage is prevented.

References

- Ye Q, Dong Z, Qu R, Fang Z. Experimental investigation of optical beam deflection based on PLZT electro-optic ceramic. Optics Express 2007; 15(25): 16933-16944. DOI: 10.1364/OE.15.016933.
- [2] Ye Q, Qiao L, Cai H, Qu R. High-efficiency electrically tunable phase diffraction grating based on a transparent lead magnesium niobate-lead titanite electro-optic ceramic. Optics Letters 2011; 36(13): 2453-2455. DOI: 10.1364/OL.36.002453.
- [3] Li Y, Li J, Zhou Z, Bhalla A, Guo R. Optical and Electrooptic Properties of Potassium Lithium Tantalate Niobate Single Crystals. Proc SPIE 2011; 8120: 81201S. DOI: 10.1117/12.904988.
- [4] Wang Z, Sun W, Chen A, Kosilkin I, Bale D, Dalton LR. Organic electro-optic thin films by simultaneous vacuum deposition and laser-assisted poling. Optics Letters 2011; 36(15): 2853-2855. DOI: 10.1364/OL.36.002853.
- [5] Spreiter R, Bosshard Ch, Pan F, Gunter P. High-frequency response and acoustic phonon contribution of the linear electro-optic effect in DAST. Optics Letters 1997; 22(8): 564-566. DOI: 10.1364/OL.22.000564.
- [6] Volk TR, Ivanov NR, Isakov DV, Ivleva LI, Lykov PA. Electro-optical properties of strontium-barium niobate crystals and their relation to the domain structure of the crystals. Phys Solid State 2005; 47(2): 305-311. DOI: 10.1134/1.1866411.
- [7] Xu D, Tan G, Wu S-T. Large-angle and high-efficiency tunable phase grating using fringe field switching liquid crystal. Optics Express 2015; 23(9): 12274-12285. DOI: 10.1364/OE.23.012274.
- [8] Paranin VD, Matyunin SA. Modeling tunable diffractive elements based on the linear electro-optic effect. Proc SPIE 2010; 7523: 75230E. DOI: 10.1117/12.852958.
- [9] Bussager R, Osman J. Design of PLZT electro-optic beam steering device. Proc SPIE 1996; 2749: 90-100. DOI: 10.1117/12.243092.
- [10] Paranin VD. Formation of needle-shaped domains in a surface layer of x-cut lithium niobate. Technical Physics 2014; 59(12): 1869-1873. DOI: 10.1134/S1063784214120226.
- [11] Paranin VD. Methods to control parameters of a diffraction grating on the surface of lithium niobate electro-optical crystal. Technical Physics 2014; 59(11): 1723-1727. DOI: 10.1134/S1063784214110206.
- [12] Paranin VD. Edge effect of electric field in the tunable diffraction optical elements [In Russian]. In book: Materials of VIII International conference "Basic Problems of Optics (BPO-2014)", Saint-Petersburg, 20-24 October 2014: 193-194.
- [13] May C, Strumpfel J. ITO coating by reactive magnetron sputtering-comparison of properties from DC and MF processing. Thin Solid Films 1999; 351(1-2): 48-52. DOI: 10.1016/S0040-6090(99)00206-0.
- [14] Bertran E, Corbella C, Vives M, et al. RF sputtering deposition of Ag/ITO coatings at room temperature. Solid State Ionics 2003; 165(1-4): 139-148. DOI: 10.1016/j.ssi.2003.08.055.

Authors' Information

Vyacheslav Dmitrievich Paranin (b. 1986) graduated from Samara State Aerospace University in 2010, majoring in Design and Technology of Radio-Electronic Equipment. Currently he works as an assistant professor at the Nanoen-gineering department of Samara National Research University. Research interests are methods and devices for control-ling of laser radiation. E-mail: <u>vparanin@mail.ru</u>.

Sergei Vladimirovich Karpeev, Doctor of Physical and Mathematical Sciences; Professor of Samara National Research University. He is a leading researcher of the IPSI RAS – Branch of the FSRC "Crystallography and Photonics" RAS. Research interests: diffractive optics, singular optics, mode and polarization transformations, optical manipulations, optical and digital image processing. E-mail: <u>karp@smr.ru</u>.

The information about authors **Konstantin Nickolaevich Tukmakov** and **Boris Olegovich Volodkin** you can find on page 672 of this issue.

Code of State Categories Scientific and Technical Information (in Russian – GRNTI)): 29.31.15. Received September 30, 2016. The final version – October 24, 2016.