The combination methodic of diffusion and implantation technologies for creating optic wave-guided layers in lithium niobate

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Abstract. The implantation of copper into Lithium Niobate in the prohibited crystal zone forms a definite energetic level for optic transits. This paper examines conditions of optic wave-guided layers formation on Niobate Lithium due to the method of implantation copper ions with the next diffusion. Reflect Spectrum in consequences implantation is extended. The transfer of the optical power from the primary beam into the another beam was discovered and in reverse. Photo galvanic characteristics of implantation specimen identity of crystal by traditional technology and doping CuO manufacture.

1. Present state of the problem
Copper-doped Niobate Lithium is challenging to form energetic levels in the crystal prohibited zone for the optical transitions. Local intensities of the electric field appearing due to doping are accompanied by the alteration of the refraction index, occurrence of photorefractive holograms and other optic effects. It lets create optically controlled miniature crystal optoelectronic devices. As it shows [1], the largest changes of the refraction index are observed while the copper concentration is $10^{24}-10^{26}$ m$^{-3}$ using the diffusion from the scum which is made under the copper thermal evaporation. For instance, the possibility to change the refraction and absorbance index with the concentration growth of two-times ionized centers of copper Cu$^{2+}$ playing a role of traps in the prohibited zone is shown [2]. However, the realization of some electro-optic effects, while forming films on typical vacuum unit with lube means to create vacuum, is restrained by the probabilistic representation of wave-guided layers. There is an interest to simplify the technology of forming active wave-guided layers due to the primary copper ions implantation with the next diffusion on the implanted layer. The most perspective for the similar purposes are the sources of metal ions with the generation of particles from a vacuum arc cathode stain [3].

2. Problem solution technique
A solution technique of the problem of optical waveguide synthesis is based on variation of the refraction coefficient of a crystal as a result of the implantation of ions in the near-surface field and their reallocation on necessary depth as a result of the diffusion. As a result, the effect of the ionic stream can lead to amorphization of the crystal structure, in other words to create "swelling" of atomic packing, and to change both the refractive index and the optical absorption.
3. Process modelling

3.1. Modelling of the implantation process

The implanted dose of ions of copper $D_{Cu}$ was calculated on a ratio:

$$D_{Cu} = n_{Cu} \cdot N_A / a \cdot b$$  \hspace{1cm} (1)

Where $n_{Cu}$ is the quantity of copper mole; $N_A=6.02 \cdot 10^{23}$ is the Avogadro number; $a=2.5$ cm is the length of a sample; $b=0.8$ cm is width of a sample; $d$ is the thickness of a film.

$$n_{Cu} = \rho_{Cu} \cdot a \cdot b \cdot d / M_{Cu} ,$$  \hspace{1cm} (2)

$\rho_{Cu}=8.9$ g/cm$^3$ is the copper density; $M_{Cu}=81.72$ g/mole is the molar mass of copper.

At the calculated thickness of the film $d=50 \cdot 10^{-9}$ m the calculated implanted dose makes: $D_{Cu}=3.6885 \cdot 10^{17}$ ion/sm$^2$.

3.2. Modelling of the diffusion process

During the diffusion time considerably exceeding the time needed for the complete intromission of an impurity from the film into the crystal, the regularity of its concentration is featured by the Gaussian function. The thickness $h$ of the wave-guided layer used here is related to the diffusion depth, as [4]:

$$h = 0.892 \sqrt{4Dt} ,$$  \hspace{1cm} (3)

where $D$ is the diffusion constant and $t$ is its duration.

3.3. Modelling of the optics process

The absorption coefficient $k(\lambda)$ was calculated on the ratio:

$$k_\lambda = -\frac{1}{d} \ln \left\{ \frac{- \left(1 - \frac{1}{R}\right)^2}{2T} + \sqrt{\frac{1}{R^2} + \left(1 - \frac{1}{R}\right)^4 \cdot \frac{1}{4T^2}} \right\} ,$$  \hspace{1cm} (4)

where $T(\lambda)$ is the transmittances constant, and $R(\lambda)$ is the reflexions constant. Calculations were carried out with the software package MATCAD.

4. Procedure and technique of the experiment

On fig. 1 the circuit of the ionic source with the microsecond duration with the initiation of a surface breakdown of the dielectric is presented [5]. The source consists of the cathode 1, the dielectric interposing 2, the ignitor electrode 3 and the anode 4 closed by the grid 5. The device of neutralising of the ion charge is fulfilled on the basis of a plasma source of electrons.

At pressure 0.01 Pa switching of size C capacity (5 kV, 100 µF) in the chain of the transformer $T$. The disruption in the dielectric interposing between the cathode and the ignitor electrode takes place. In the source cavity a vacuum arc is formed. When feeding of the positive polarity speeding up impulses (U~20 kV, 250 µs, 1- 50 Hz), the ions extraction with the current under 1000 mA occurs. Ions when transiting the anode 4, the grid 5, and the neutralizer of charge 6 hit the specimen 7 installed on the work table 8. The reduction of the drop wise fraction and the stability of operation was attained by feeding of the plasma gap at the moment of the ionic current takeoff. On the fig. 2(a) the circuit of Hall’s source for the crystal ion clearing of Niobate Lithium and the circuit of the magnetron (fig. 2b) to form the copper film are presented.
Figure 1. Pilot model. 1 – cathode, 2 – dielectric interposing, 3 – ignitor, 4 – anode, 5 – grid, 6 – neutralizer, 7 – specimen, 8 – work table.

Figure 2. a) – Circuit of Hall’s source; b) – magnetron’s circuit, 1 – anode, 2 – box, 3 – target, 4 – magnet.

In the capacity of a dose analyser, the device with the dose increase control during exposure to radiation is used [5].

5. Preparation of samples
To increase a film adhesion the gas emission mechanism was estimated [6]. For this purpose the crystal raying is made in the low-energy plasma, and the monochromator DMR 2 with the photoreceiver FSK-1 recorded the intensity of the oxygen lines under different discharge rates. The crystals with the minimal gas extraction were chosen when the intensity of the oxygen lines was minimal and almost did not change while alternating the current voltage characteristics of the discharge.

6. Forming the wave-guided layer
The usage of the constant vacuum system wash with argon at the rate of flux of 30 sm$^3$ atm/hour according to mass-spectrometry by a measurer of partial pressures IPDO-1 allows decreasing the quantity of such optical modes quenches as hydrocarbons. The direction of the synthesis reaction was estimated by the pressure change during a period of time. During the experiment, the following condition was provided: the inflow stream of inert gas $Q$ did not exceed the pressure change of the gas component $D P$ for a period of the implantation time $D t$ (~ 20 min).

$$Q = V (\Delta P / \Delta t),$$

where $V$ is the size of the evacuated chamber.

Implantation was carried out under the speeding up voltage 20 kW in the argon medium under a pressure 0.01 Pa at arc current less than 100 A. The ionic current impulse was 25-1000 mA at the duration 250 µs. The impulse repetition frequency was 1- 50 Hz. The measured implanted dose was $3 \cdot 10^{17}$ ion/sm$^2$ which is close to the calculated value for the film with the thickness of 50 nm.

The crystal annealing with the size of 25x8x2 mm was made in the furnace SUOL 025 with the speed of the temperature increase 2 K/s and with the temperature endurance 1350 K within 4 hours. Then, the obtained waveguide was cooled within 8 hours with the velocity of ~2 K/s.

Then the forming process of copper scum having thickness 0.5 micron on Niobate Lithium was carried out by means of the small-scale pulsed magnetron which works on a frequency of 50 kHz under a pressure of 0.1 Pa. The analysis carried out on the focus beam microscope showed that the quantity of copper in the scum amounts 4% under the thermal forming of covering in the vacuum, and it calculates 36% in argon. While forming by means of the magnetron the part of copper compounds 40% total.

The diffusion of copper is realized in the air atmosphere at a temperature of 1050 K during 2-20 hours.
7. Undertaken researches
The figure 3 presents a circuit for optic tests. The reference beam of laser 1 (LCS-DTL-317) with the power 40 mW on the wave length 532 nm through the collimator 2 falls on the beam splitting cube 3 where divides into two beams with equal intensities on 390 mW /sm². Then one part of the beam is aimed to the crystal 6 at Bragg angle (12,5° for the emission with the length wave 532 nm). The other part of the beam from the beam splitting cube through the prism 4 is also directed to the crystal symmetrically to the normal by its entry edge. Reading of the photorefractive diagram in the process of its forming is carried out according to the Bragg angle diffraction method of the light beam with the length wave 655 nm radiated by the laser diode 5 with the power output 6 mW. The angle of descent of the reading beam on its entry edge is equal to the Bragg angle which amounts 7,7°. Power timing dependencies of the beam passed the diffraction and the transmitted beam 7, 8 are recorded by means of photodiodes 9 (FD-24 K). The data processing system 10 included two digital microampermeter as V7-40/1, an interface and personal computer. It is worth to point out, that the readout radiation of the wave length 655 nm weakly influences the formation of photorefractive holograms by writing beams on the wave length 532 nm due to the strong decrease of photovoltaic constants of crystals with the wave length. The system is set up for the minimal discrepancy of beams in the plane XZ. The test sample of crystal was exposed to annealing under the temperature 190°C or is kept in the darkness in order to delete recorded photorefractive holograms. On the fig. 4 specific timing the dependencies of relative powers of the beam after the diffraction (the curve 2) and the transmitted (reading) beam (the curve 1) are presented.

![Figure 3. Circuit of optic tests. 1 – laser, 2 – collimator, 3 – splitting cube, 4 – prism, 5 – laser diode, 6 – crystal, 7, 8 – transmitted beam, 9 – photodiodes, 10 – processing system]

The photo induced absorption of the non-object beam and the transfer of the optical power from the beam 7 (figure 3) into the beam 8 was discovered and in reverse. Oscillographic measurements made by the stand-by oscillograph S8-12 within the microsecond interval show discontinuous variations of intensity patterns are observed when the disruption occurs on the crystal surface. In this case, the field amplitude of the spatial charge on the crystal frontier reaches the value 30 kV/sm what exceeds the electrical surface stability of the piezoelectric.

8. Results and outputs
The biggest alteration of the diffraction pattern is observed under the maximal copper doping of the crystal. Due to the optical absorption coefficient on the waves length 477 and 1040 nm, according to the methodic [1] the concentration $C$ of ions $Cu^{2+}$ and $Cu^+$ responsible for electro-optical characteristics of copper doping crystals was determined:

$$C_{Cu^+} = 2 \times 10^{21} a_{477}; \quad C_{Cu^{2+}} = 6.8 \times 10^{22} a_{1040} \text{[m}^{-3}] ,$$

(6)
where $\alpha$ is the absorption coefficient (sm$^{-1}$). The index points out the wavelength 477 and 1040 nm.

The table 1 presents parameters of formed wave-guided layers in a time of the diffusion $t$ (hour) for the films of copper having thickness $\delta$ (nm).

| № | $\delta$ (nm) | $t$ (h) | Cu$^+$ (m$^{-3}$) | Cu$^{2+}$ (m$^{-3}$) | $\alpha_{532}$ (sm$^{-1}$) |
|---|---|---|---|---|---|
| 1 | 100 | 2 | $7.9 \times 10^{22}$ | $8.7 \times 10^{22}$ | 0.063 |
| 2 | 200 | 3 | $2 \times 10^{23}$ | $1.4 \times 10^{24}$ | 0.615 |
| 3 | 260 | 20 | $7.8 \times 10^{24}$ | $6 \times 10^{24}$ | 0.88 |
| 4 | 900 | 9 | $6 \times 10^{24}$ | $2.8 \times 10^{25}$ | 7.9 |

Calculations show that perhaps changes of diffraction efficiency are connected with the magnitude variation of the crystal spatial field.

9. Conclusion

The process to form wave-guided layers on the typical vacuum sets requires computer maintenance to define the order and parameters of producible process. The doze of copper ions $\sim 5 \times 10^{17}$ sm$^2$ is minimal to create an active wave-guided layer. The next increasing of the concentration is attained by the diffusion of copper from the scum formed in the inert atmosphere.

Powers changes of the transmitted beam and the beam after the diffraction are connected with the alteration of the crystal electric field caused by cracking on its surface.

The availability to conduct the intensity of photo induced absorption using the change of copper concentration in the crystal offers the challenge to create light-controlled active optic wave-guided layers.

On figures 5, 6 dependences of transmittances $T(\lambda)$, absorption $k(\lambda)$ and reflexions $R(\lambda)$ on the wave length through the waveguide nanolayer before and after implantation are presented.

![Figure 5](image)

**Figure 5.** Dependences of transmittances $T(\lambda)$, absorption $k(\lambda)$ and reflexions $R(\lambda)$ on the wave length before implantation.

![Figure 6](image)

**Figure 6.** Dependences of transmittances $T(\lambda)$, absorption $k(\lambda)$ and reflexions $R(\lambda)$ on the wave length after implantation.

We can see that before the implantation the following effects are present: transmittance of the waveguide layer on the wave length almost does not vary, and the reflectivity increases with the wave length growth. After implantation and annealing the transmittance decreases; (the layer "clarifies"), and the reflectivity has a minimum on the wave length of 800 nm.

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