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Erbium diffusion from erbium metal or erbium oxide layers deposited on the surface of various $LiNbO_3$ cuts

P. Nekvindova [a],*, J. Cajzl [a], B. Svecova [a], A. Mackova [b,c], P. Malinsky [b,c], J. Oswald [d], J. Vacik [b], J. Spirkova [a]

[a] *Department of Inorganic Chemistry, Faculty of Chemical Technology, Institute of Chemical Technology, Technicka 5, 166 28 Prague, Czech Republic*
[b] *Nuclear Physics Institute, Academy of Sciences of the Czech Republic, v.v.i., 250 68 Rez, Czech Republic*
[c] *Department of Physics, Faculty of Science, J.E. Purkinje University, Ceske mladeze 8, 400 96 Usti nad Labem, Czech Republic*
[d] *Institute of Physics, Academy of Sciences of the Czech Republic, v.v.i., Cukrovarnicka 10, 162 53 Prague, Czech Republic*

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ABSTRACT

In this paper the doping of erbium from thin layers deposited on the $LiNbO_3$ surface with an emphasis on the relations between changes in the $LiNbO_3$ structure and the luminescence properties of Er:$LiNbO_3$ have been studied. The erbium-containing layers were primarily characterised in terms of the luminescence at 1.5 μm. The obtained results showed a high level of structural ordering in the samples after indiffusion of Er at high-temperature. This was apparent from the luminescence results, where more than 6 quite narrow luminescence bands were found, and also from the RBS/channeling analysis which showed, that the doped structure has similar degree of ordering as the bulk crystal $LiNbO_3$. In terms of luminescence intensity at 1.5 μm a better source for the erbium doping seems to be the erbium oxide layer. The highest luminescence intensity values were among the cuts achieved in the *Z* cut.

1. Introduction

Lithium niobate ($LiNbO_3$, LN) is a well-known material in the field of photonics because of its ability to combine active and passive optical beam control [1]. In order to create an optically active material that could be used as an optical amplifier or waveguide laser, the LN is doped with laser-active ions such as erbium to form erbium-doped lithium niobate (Er:$LiNbO_3$ or Er:LN) [2–5].

Currently, there are several methods used for erbium doping [6–8]. The doping from a thin erbium metal film, deposited on the substrate surface, is the first and thus far the most common approach for introducing erbium into the lithium niobate. Another suitable source of doping has been found in erbium oxide [7]. Thin layers prepared in this way were investigated in detail; it was found that the concentration of erbium reached approximately 0.1–0.2 at.% at a depth of several microns (i.e., up to 6 μm) and such erbium-rich layers displayed high-intensity luminescence at 1.5 μm [8]. A study of the structure of the doped layers revealed that erbium atoms preferentially occupied the lithium sites [9]. The implantation of erbium ions into the lithium niobate [10,11] and the diffusion of erbium ions from a melt of erbium containing salts [12,13] are also known.

For several years, we have utilised an alternative method of Er:LN-layer fabrication with emphasis on the relations between the changes in the LN structure and the luminescence properties of Er:LN. We have also considered possible ways to influence the luminescence properties by modifying the crystal field surrounding the erbium ions. We have already reported the doping from the melt [14,15] and the techniques of ion implantation [16–18] into various LN cuts. In order to make the subject of our interest more complete, we have performed some experiments using the techniques of doping from a thin Er metal film deposited on various LN cuts.

In this paper, we present the results of our experiments carried out on the doping of erbium from the thin layers deposited on the LN surface, the layers being either Er metal or Er_2O_3. The layers used as the doping source had a thickness of 10 and 20 nm. The erbium-containing layers were primarily characterised in terms of their luminescence at 1.5 μm. The concentration depth profiles of erbium and lithium in the doped samples and the changes of their structure were also investigated. An entirely new idea was to compare the results of such doping into commonly used and specially designed lithium niobate substrate cuts to see the possible differences arising from the migration of the incorporated erbium ions through a differently oriented crystalline matrix.

2. Experiments

For the experiments, we used wafers of LN with various crystallographic orientations (pulled by the Czochralski method) supplied by Crytur Turnov (Czech Republic). The wafers were oriented and

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* Corresponding author. Tel.: +420 220 444 003; fax: +420 224 311 010.
E-mail address: pavla.nekvindova@vscht.cz (P. Nekvindova).

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labelled as follows: standard *X* cut $\langle 1\,1\,-2\,0\rangle$ and *Z* cut $\langle 0\,0\,0\,1\rangle$, and specially executed *Y* cuts with respect to the cleavage plane of the LN crystal, namely a parallel Y_{II} cut $\langle 1\,0\,-1\,2\rangle$ and perpendicular $Y_{\perp}$ cut $\langle 1\,0\,-1\,4\rangle$. All wafers were polished on both sides, with one side polished to optical quality. The dimensions of the wafers were 12 × 7 mm with a thickness of 0.7 mm.

The experimental conditions of the erbium doping from thin erbium metal and oxide films deposited on the crystal surface were chosen according to an already published experiment [7]. The thicknesses of the deposition source layers were 10 nm and 20 nm for both Er metal layers and Er_2O_3 layers (custom-made by Crytur Turnov – Czech Republic by magnetron sputtering on the crystal surface). For these experiments, only *Z* cuts were used. The diffusion of erbium from both kinds of layers was carried out at 1060 °C for 100 h in a tube furnace in a dry O_2 atmosphere. These experiments were repeated in order to verify the results. Based on previous results, further studies were conducted only with samples doped from 20-nm thick layers. Using these samples, we focused on the behaviour of the different cuts. The experiments were performed with the *X*, *Z*, Y_{II}, $Y_{\perp}$ cuts of $LiNbO_3$.

The concentration depth profiles of the incorporated erbium atoms were studied by Rutherford Backscattering Spectroscopy (RBS) at the Nuclear Physics Institute in Řež, Czech Republic. The analysis was performed by the Tandetron 4130 MC accelerator using a 2.0 MeV He^+ ion beam. He^+ ions incoming at normal incidence were backscattered and detected at a laboratory angle of 170°. The RBS spectra were evaluated using the GISA 3.99 [19] code utilising cross-section data from IBANDL [20]. The RBS depth resolution is better than 10 nm and the detection limit of Er concentration in this case is about 0.1 at.%. The detection limit mentioned above can be achieved for the region of backscattered He^+ ions, where the Er signal is not overlapped by the Nb signal from the LN substrate. Consequently, this detection limit for Er is implementable up to an analytical depth of about 150 nm. In order to study the changes in the lithium niobate structure caused by the erbium in-diffusion, the RBS/channelling measurements, using a 1.7 MeV He^+ beam from the Van de Graaff accelerator in the Helmholtz-Zentrum Dresden Rossendorf, Germany, were performed. The value of the minimal yield χ_{min} was determined by the integration of the regions of interest in random and aligned spectra using the usual procedure reported, e.g., in [21].

The lithium concentration depth profiles in the prepared samples were measured by Neutron Depth Profiling (NDP). The method is based on a reaction of a thermal neutron with ^6Li:$^6Li(n,\alpha)\,^3H$. The fabricated samples were irradiated with a thermal neutron beam from a 6-m long neutron guide (the neutron intensity was 10^7 $n\text{th}\,\text{cm}^{-2}\,\text{s}^{-1}$) and the charged reaction products were recorded by a Hamamatsu PIN diode detector. The nominal accuracy of the NDP method is ~5% of the concentration value of Li, c_{Li}; the nominal depth resolution is ~10 nm.

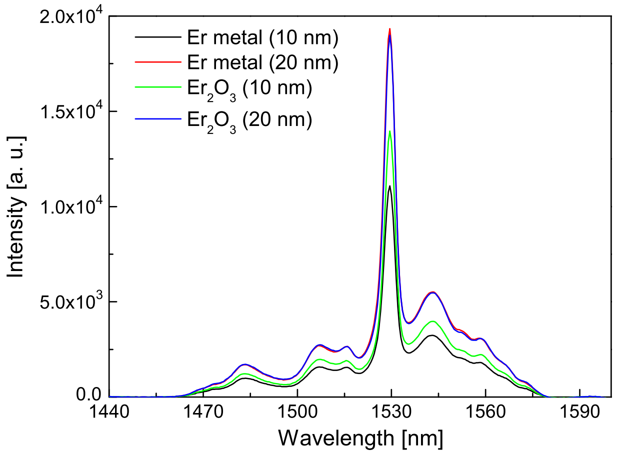

Fig. 1. The luminescence spectra of the erbium-doped LN samples prepared by doping from the erbium metal and erbium oxide layers deposited on the *Z* cut of LN. (*Experimental conditions*: 1060 °C; 100 h; atm. O_2.)

Table 1
The comparison of luminescence intensity at 1529 nm of the erbium-doped samples prepared from the 20-nm thick layers of Er metal or Er_2O_3 deposited on the surface of various LN cuts. (*Experimental conditions:* 1060 °C; 100 h; atm. O_2.)

Er sources	*Z* cut	*X* cut	$Y_{\perp}$ cut	Y_{II} cut
Luminescence intensity (rel.%)				
Metallic Er	100	16	81	43
Er_2O_3	100	10	60	40

The photoluminescence spectra of the prepared samples were collected within the range of 1440–1600 nm at room temperature. A pulse semiconductor laser POL 4300 emitting at 980 nm was used for the excitation of the electrons. The luminescence radiation was detected by a two-step-cooled Ge detector J16 (Teledyne Judson Technologies). To scoop specific wavelengths, a double monochromator SDL-1 (LOMO) was used. For the evaluation, all of the luminescence spectra were transformed to the base level and the baseline was abstracted.

3. Results

3.1. Luminescence properties

Fig. 1 shows a comparison of the luminescence spectra in the region around 1.5 µm for samples prepared by erbium diffusion from an Er metal layer and an Er_2O_3 layer of various thicknesses. It is obvious that all samples revealed the highest intensity at a wavelength of 1529 nm. Moreover, there are evident 6 emission bands with a narrow spectral width at wavelengths of 1483 nm, 1507 nm, 1516 nm, 1529 nm, 1543 nm and 1559 nm. The measured spectra are similar to the spectra obtained from a bulk-doped Er:LN crystal [14,22]. As expected, the luminescence intensity corresponded well with the thicknesses of the deposited source layers, i.e., the intensity was always higher for the 20-nm thick source layers. The comparison of both erbium sources is not so ambiguous. The luminescence intensity at 1529 nm was higher for the 10-nm thick Er_2O_3 deposited layers than for the 10-nm thick metal deposited ones. No differences between the luminescence intensity of the two erbium sources were observed for the 20-nm thick source layers. To verify the results, we repeated the experiments with the second set of the LN wafers, which proved the results described above.

The behaviour of variously oriented LN cuts was studied using the samples that were doped from 20-nm thick films of both erbium metal and erbium oxide; the doping conditions were otherwise identical. The resulting luminescence spectra showed similar shapes of the luminescence spectra with the 6 above-described narrow luminescence bands for all the samples. The spectra of the various cuts differed especially in the intensity of the 1529 nm band, which was the highest in the *Z* cuts for both types of the doping sources. Values of the intensity of luminescence at 1529 nm are stated in Table 1. Data are compared to the *Z* cut which luminescence intensity was considered to be 100%.

3.2. Erbium concentration in the prepared layers

In Fig. 2, the RBS spectrum of the Er metal layer deposited before the actual diffusion was compared with that of the erbium-containing layer after the diffusion. The random RBS spectrum of the sample with the deposited erbium layer clearly