Method for the local incorporation of Er into LiNbO₃ guided wave optic devices by Ti co-diffusion

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Co-diffusion of Er with Ti into LiNbO₃ produces more than an order of magnitude enhancement in Er diffusivity over that achieved by ion implanting and thermal diffusion. The enhancement is dependent on the amount of Ti present. Er doped channel waveguide fabrication is completely compatible with standard Ti:LiNbO₃ technology. Fluorescence measurements from co-diffused and bulk grown Er doped LiNbO₃ show that the co-diffused Er³⁺ is in the LiNbO₃ phase.

LiNbO₃ guided wave optics has produced the most sophisticated integrated optic structures to date, but the technology lacks the ability to produce or amplify light. Guided wave lasing and amplification have been reported in channel waveguides fabricated in bulk-grown Nd: LiNbO₃.[1,2] Bulk-doped materials are, however, not the optimal solution for guided wave integrated optics since absorption will occur in every section of the waveguide which is not optically pumped to transparency. Lateral confinement of the gain medium is also necessary to avoid absorption of the evanescent field. From a telecommunications perspective, Er is a more interesting rare-earth dopant due to its long lived ~1.5 μm radiative transition. Previous attempts to develop techniques which could be used to locally dope LiNbO₃ with Er have met with limited success. Ion implanting (10^{16} cm⁻² dose of 200 keV Er ions) followed by a thermal annealing at 1050 °C for 45 h produced a $1/e$ depth of 1.8 μm.[3] A modest optical gain (0.75 dB) has been recently reported[4] from similarly prepared channel waveguides. Guided wave stimulated emission at 1.53 μm has also been observed[5] after extended heating (80 h at 1060 °C) of an Er coated LiNbO₃ substrate. In this letter we demonstrate that the diffusivity of Er in LiNbO₃ can be dramatically enhanced by co-diffusing Ti with Er. Fluorescence spectra from co-diffused Er:Ti:LiNbO₃ and bulk doped Er:LiNbO₃ are nearly identical.

Channel waveguides were photolithographically defined on z-cut LiNbO₃. Er and Ti/Er thin films were deposited by ion beam sputtering onto a photoresist patterned substrate or directly onto LiNbO₃ (planar diffusions). Er features could not be produced with this (liftoff) technique due to the poor sticking coefficient of Er on LiNbO₃. However, the films were easily processed if the Er was over-coated with Ti. Diffusions were carried out at 1050 °C under a flowing oxygen atmosphere containing water vapor. To characterize diffusion profiles, samples were mechanically milled at an angle with a rise-to-run of ~1:30 (Fig. 1). Two-dimensional Er concentration depth profiles were made by electron microprobe (EMP) scans of the milled region (inset, Fig. 1). One-dimensional pro-

files were made by averaging successive scans (Fig. 2). Due to secondary x-ray excitations the EMP results represent the average Er content within the volume of the x-ray plume (~1 μm diam). An underestimation of the Er penetration is expected because of the plume effects.

One-dimensional depth profiles from four Er diffusion experiments are shown in Fig. 2. In each case the Er film can be considered to be an infinite source since an Er-rich surface layer (<200 nm) always remained after diffusion. Without Ti, Er is observed to diffuse to a depth of $D \sim 1$ μm (curve a) after a 20 h diffusion. With an 80 nm Ti overcoat, the effective diffusivity, $D = D^2/4t$, is more than doubled (compare curves b and c). Curve d is an Er co-diffusion with an effectively infinite Ti source (since after diffusion a thin Ti-rich layer is present). The Er diffusivity is strongly dependent on the amount of Ti present as evi-
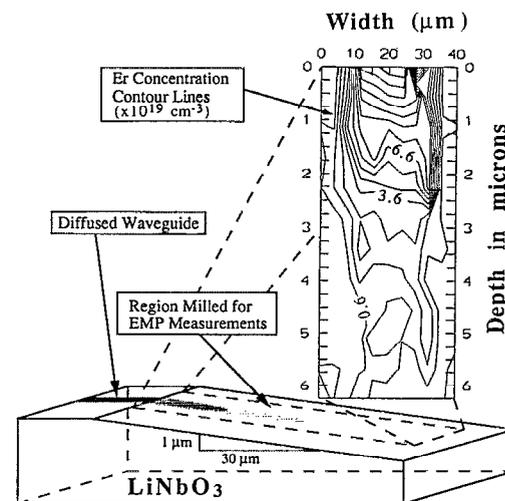

FIG. 1. Strip or planar Er or Er + Ti films are diffused into LiNbO₃ and mechanically milled at an angle of about 2° (rise-to-run ~1:30) for electron microprobe measurements. Inset: Er concentration profiles (×10¹⁹ cm⁻³) from a 10 μm wide Er(12 nm)/Ti(80 nm) bilayer, diffused 40 h. The data show ~2.5 μm e^{-1} depth penetration of Er and a maximum measurable penetration of ~4 μm.

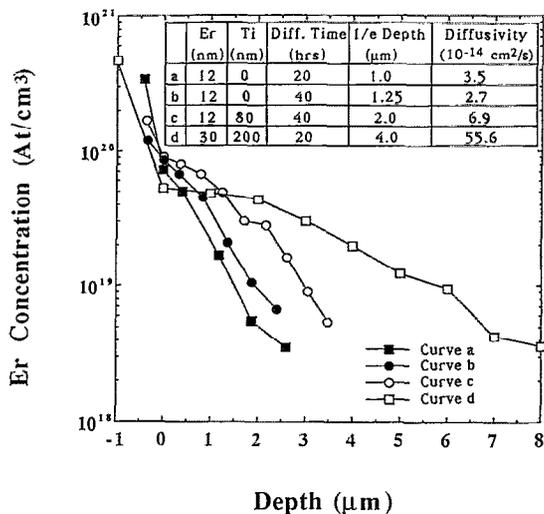|	Er (nm)	Ti (nm)	Diff. Time (hrs)	I/e Depth (μm)	Diffusivity (10^{-14} cm²/s)
a	12	0	20	1.0	3.5
b	12	0	40	1.25	2.7
c	12	80	40	2.0	6.9
d	30	200	20	4.0	55.6

FIG. 2. One-dimensional depth profiles from four Er diffusion experiments (see table inset) produced by averaging adjacent scans. Samples a, b, and d, are planar thin films; sample c is a 10 μm wide bilayer of Er and Ti. The LiNbO$_3$ surface is at $z = 0$.

FIG. 4. Fluorescence spectra ($\lambda_{\text{excit}} = 453.4$ nm) of the $^4S_{3/2} \rightarrow ^4I_{15/2}$ transition at 10 K in (a) co-diffused Er:Ti:LiNbO$_3$ and (b) bulk doped (0.6 mol %) Er:LiNbO$_3$.

denced by curves c and d. In these experiments, a 20 h co-diffusion of Er with Ti produced a maximum 4 μm e^{-1} diffusion depth, corresponding to a 15-fold increase in diffusivity over Er-only diffusion.

For efficient optical amplification the Er doping should be laterally confined to a narrow strip centered on the channel waveguide.[6] Figure 3 shows the two-dimensional concentration profile of what, we believe, is the first locally Er doped Ti:LiNbO$_3$ waveguide. A 3 μm wide bilayer stripe consisting of 12 nm Er and 80 nm Ti was defined using photolithographic liftoff and was diffused for 40 h at 1050 °C. To form the channel waveguide, a second strip of Ti (10 μm wide by 100 nm thick) was photolithographically defined colinearly with the first and diffused for 10 h at 1050 °C. The e^{-1} concentration of Er is $\sim 2.7 \times 10^{19}$ cm^{-3} at a depth of ~ 3 μm with a maximum measurable ($\sim 5 \times 10^{18}$ cm^{-3}) penetration of > 4.5 μm. The e^{-1} diffusion width is ~ 6 μm. By comparison, the e^{-1} width of the Ti diffused waveguide is ~ 18 μm (dashed line).

Guided wave fluorescence spectra were obtained by focusing light from a tunable dye laser at 655 nm (resonant with the Er $^4I_{15/2} \rightarrow ^4F_{9/2}$ transition) into a 1 mm long end-polished segment of the waveguide. The fluorescence emerging from the waveguide has a strong peak at 1.53 μm from the Er^{3+} $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition with a measured lifetime of ~ 7 ms and a FWHM of 3.3 nm. A number of weaker peaks corresponding to transitions between other crystal field levels were also observed. Bulk doped 0.6 mol % single crystal Er:LiNbO$_3$ at room temperature produced a nearly identical spectrum and lifetime. The broad (3.3 nm) temperature-independent (10–300 K) linewidth suggests that this transition is inhomogeneously broadened. High-resolution low-temperature (10 K) fluorescence scans of the $^4S_{3/2} \rightarrow ^4I_{15/2}$ transition in bulk-doped and co-diffused Er:LiNbO$_3$ (Fig. 4) further confirm that the co-diffused Er^{3+} is in the LiNbO$_3$ phase. Upconverted fluorescence ($\lambda_{\text{fluor}} \sim 550$ nm) from the $^4S_{3/2}$ level was observed from both the Er co-diffused waveguide and the bulk doped samples when the $^4F_{9/2}$ level ($\lambda_{\text{excit}} = 656.5$ nm) was excited. We believe that the Er^{3+} upconversion is caused by efficient energy transfer between two Er^{3+} ions excited to the $^4F_{9/2}$ level as was shown for Er^{3+} doped fluorite.[7]

In conclusion, we have demonstrated an approximately 15-fold increase in the diffusivity of Er into LiNbO$_3$ by co-diffusion with Ti. The improvement is attributed to

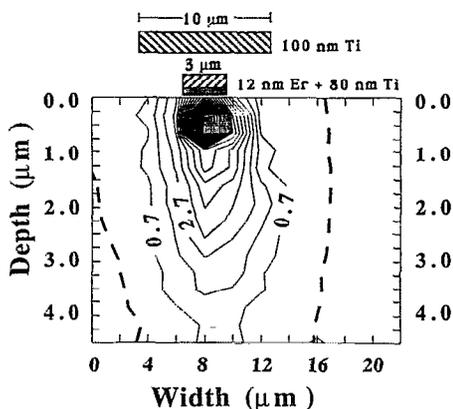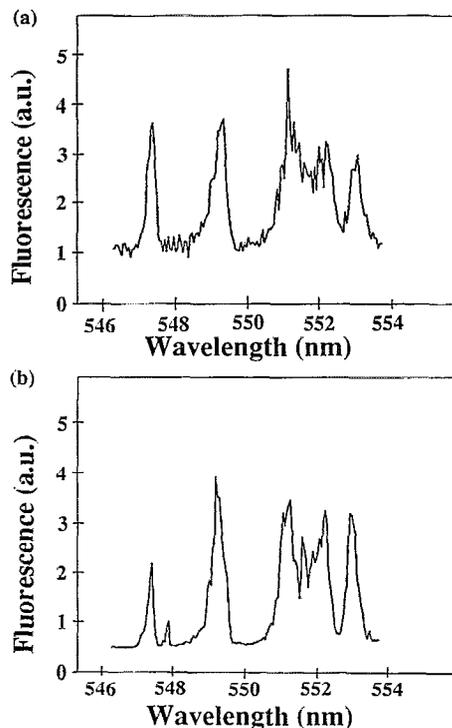FIG. 3. Two-dimensional Er concentration ($\times 10^{19}$ cm^{-3}) profile of a locally Er doped Ti:LiNbO$_3$ channel waveguide. The dashed line shows the e^{-1} Ti concentration contour.

the increased partition coefficient of Er^{3+} when Ti^{4+} is available as a charge compensation mechanism. This technique, which is completely compatible with standard $Ti:LiNbO_3$ processing, has been used to produce for the first time locally doped $Er:Ti:LiNbO_3$ channel waveguides. Detailed descriptions of the spectroscopy will be presented elsewhere but all of our results show that the Er^{3+} within the waveguide structure is in the $LiNbO_3$ phase. We believe that the co-diffusion process will provide a method for producing integrable guided wave optical amplifiers and light sources for $LiNbO_3$ based integrated optics.

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