# Confocal optical techniques to study channel waveguides in LiNbO$_3$

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**Abstract.** *Optical properties of rare-earth ions incorporated into channel waveguides can be used as optical probes to investigate the characteristics of the waveguides by using sub-micrometric resolved confocal fluorescence imaging. In this work, luminescence together with Raman spectroscopy, have been used to evaluate waveguides fabricated by different procedures in LiNbO$_3$ crystals.*

## Introduction

LiNbO$_3$ is a well established material for integrated optics applications [1] due to its electro-optical, acousto-optical and nonlinear properties and to the availability of well developed techniques to fabricate low-loss channel waveguides. The incorporation of active ions, frequently rare-earth ions (RE), allows the fabrication of photonic devices such as integrated lasers and amplifiers [2,3]. For those applications, the channel waveguide characteristics and the modifications that the fabrication procedures induce in the LiNbO$_3$ lattice are of great relevance. This has motivated a wide activity in that field throughout the last years, although the details of the lattice changes induced by the different procedures used for waveguide fabrication are not completely understood yet. Along this line, it has been recently demonstrated that confocal micro-luminescence can provide direct and precise information on the local properties of the material [4-6].

In this work, different optical techniques (continuous wave, time resolved luminescence, as well as Raman spectroscopy) have been used in a confocal configuration, in order to characterize the diverse micro-structural modifications induced by the different waveguide fabrication procedures. As optical probes, Tm$^{3+}$ and Nd$^{3+}$ ions have been selected because of their intrinsic interest as active photonic ions with applications in fields such as surgery, optical communications or remote sensing, as well as due to the availability of suitable optical transitions highly sensitive to the host surroundings.

## Experimental

Z-cut samples of LiNbO$_3$ doped with rare-earth ions (Tm$^{3+}$ or Nd$^{3+}$) have been used in this work as substrates to create channel waveguides. Different channel widths (*w*) are defined by a previous standard ultraviolet lithography, using a suitable silica mask.

Two fabrication procedures have been used: reverse proton exchange (RPE) and Zn-indiffusion. The RPE waveguides are obtained by a proton exchange process in benzoic acid at 300 ºC during 14.5 hours in a sealed ampoule, followed by a reverse ionic exchange, in which the sample was immersed in a mixture of Li/Na/K nitrides at 350 ºC during 38.5 hours. This procedure creates two different waveguides, each one confining a different propagating mode: an ordinary waveguide (TE propagating mode) close to the

surface and an extraordinary waveguide (TM propagating mode) buried below the ordinary one, in the region where protons remain after the whole process.

In the Zn-diffusion technique the substrate is heated to 500 ºC during 2 hours in a metallic Zn atmosphere, creating a rich-Zn layer on the sample surface. Then the sample is heated to 850 ºC for 4 hours. This facilitates the diffusion of $Zn^{2+}$ ions from the surface to the bulk creating a waveguide that confines both, extraordinary and ordinary, polarization modes.

To investigate the changes that the index modifiers, namely protons or $Zn^{2+}$ ions, are causing to the $LiNbO_3$ substrate, confocal micro-luminescence experiments were performed using an Olympus BX41 fiber coupled confocal microscope. Excitation of the dopant ions is performed using either a continuous argon laser or an 808 nm diode laser (LIMO GmbH). The excitation beam was focused on the sample surface by means of an x100 achromatic microscope objective (NA 0.9) down to a $\approx 0.6$ and $\approx 0.8$ µm spot size for the case of Argon and diode pumping, respectively (Figure 1). To separate the excitation beam and the sample luminescence, a Notch filter was used. The fluorescence was focused into a fiber-coupled high resolution spectrometer (SPEX 500M).

The reference system used all along this work is sketched in Figure 1. The x-axis stands for the direction parallel to the sample surface, while the y-axis stands for the depth, being the origin of the coordinates located on the $LiNbO_3$ surface, in the middle of the mask opening. To control the samples position, they were situated onto an XY motorized with 0.1 µm spatial resolution stage that allows the possibility of mapping fluorescence properties such as intensity, spectral position and bandwidth of the emission bands.

For lifetime measurements, the collection fiber was connected to a photomultiplier tube and the generated signal was averaged and recorded by a 500MHz Lecroy digital oscilloscope.
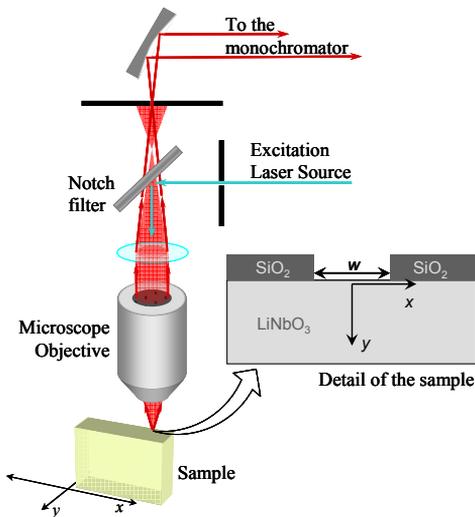


Fig. 1: Confocal microscope schematically represented showing the experimental setup and a cross section view of the sample with the silica mask channel opening. The reference axes used in this work, x and y, are represented.

## Results

Figure 2 shows the micro-fluorescence spectra of the $Tm^{3+}$:$^3H_4 \rightarrow {}^3H_6$ and the $Nd^{3+}$:$^4F_{3/2}$ $\rightarrow {}^4I_{9/2}$ transitions. Those transitions have been selected as probes to characterize the

possible local lattice changes of the sample, produced during the waveguide fabrication, due to their high intensity and high sensibility to the host surroundings.

Fig. 2: Micro-fluorescence spectra of the $Tm^{3+}$: $^3H_4 \rightarrow$ $^3H_6$ and the $Nd^{3+}$:$^4F_{3/2} \rightarrow$ $^4I_{9/2}$ transitions. The arrows point to the peaks selected to be used as probes for the local changes induced in the material.
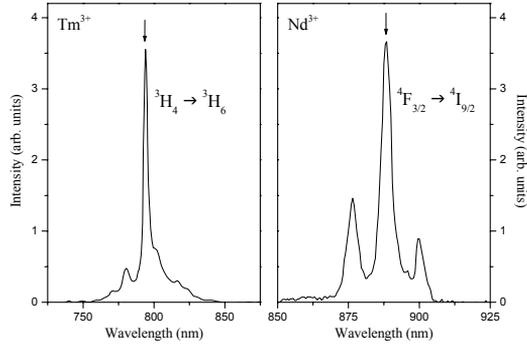
Reverse proton exchange waveguides:

Previous works ensure that proton exchange waveguides show a noticeable change in the luminescence lifetime of the dopant levels, being the de-excitation faster in the waveguide than in the bulk [7]. Confocal experiments in $Nd^{3+}$-doped $LiNbO_3$ RPE waveguides confirm those results (Figure 3a), and add the possibility of monitoring the lifetime value at any position within the waveguide and its surroundings, which could bring a further understanding of such lifetime reduction. A scanning through the sample edge confirms the correlation between lifetime reduction and the density of protons in the waveguide. The buried waveguide defines a minimum lifetime, located 6 μm below the surface and a Gaussian-shaped distribution along the x-axis as depicted in Figure 3b (full circles).
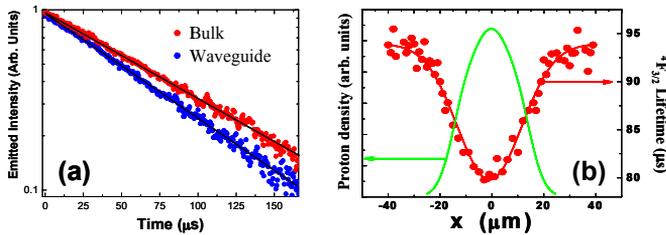
Fig. 3: (a) Lifetime value of the $^4F_{3/2}$ energy level of $Nd^{3+}$ ions inside and outside the RPE waveguide ($w = 17$ μm). (b) $^4F_{3/2}$ lifetime as function of x position (full circles) and calculated proton density in the same positions.

This mapping and modifications can also be tracked by following the luminescence associated to the $^4F_{3/2} \rightarrow$ $^4I_{9/2}$ transition where protons induce a blue-shift of the main peak energy position (about $\Delta E \approx 0.8$ $cm^{-1}$ [5]). Both spectral shift and lifetime reduction are in complete agreement with proton distribution.

Zn-diffusion waveguides:

The energy shift of RE luminescence transitions have been also detected following the luminescence of $Nd^{3+}$ and $Tm^{3+}$-doped $LiNbO_3$. In this case it has been observed that the luminescence peak is shifted to higher or lower energies depending on the depth (y distance). Figure 4 shows the results obtained for a waveguide ($w = 10$ μm) in $Tm^{3+}$:$LiNbO_3$, measured along x-axis and y-axis directions, and quantifying different

spectroscopic characteristics. It can be seen how the luminescence intensity (a), the energy position of the peak (b) and its width (c) have a noticeable spatial dependence in the waveguide region. The energy position of the luminescence peak (Figure 4b) is blue shifted in a buried region (around 3 μm depth) surrounding a central section in which the shift is in the opposite direction. According to previous works [4], this can be understood in relation with the lattice modification of the substrate, indicating a contraction of the lattice in the waveguide surface, surrounded by a lattice-dilatation zone in the deeper regions. In Figure 4c, the width of the main luminescence peak is mapped, showing that in the red-shifted region there is also a broadening of the peak, indicating a higher disorder in that region.
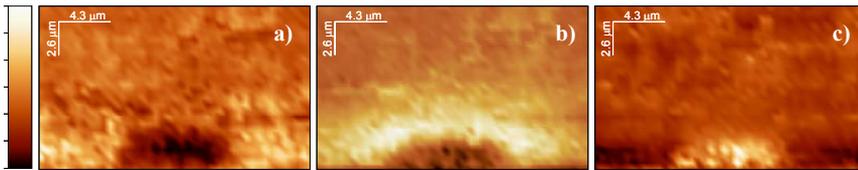


Fig. 4:  $Tm^{3+}$ luminescence dependence on position of: a) the luminescence intensity at 795 nm, b) energy position of the peak and c) peak width.

Those lattice modifications can be also detected by means of Raman spectroscopy, using the same confocal geometry. In particular, the 504 nm $LiNbO_3$ Raman peak, measured under ZZ polarization, has been used for that purpose.

## Acknowledgements

## References

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