

# Confocal Luminescence Microscopy: Application to Periodically Poled LiNbO<sub>3</sub> Devices

Volkmar Dierolf and Christian Sandmann

*Physics Dept., Lehigh University, Bethlehem, PA 18015, USA*

*vod2@lehigh.edu*

Using the recently developed imaging technique of confocal defect-luminescence microscopy, we investigated Er<sup>3+</sup>-doped periodically-poled LiNbO<sub>3</sub> waveguide devices and found that the Er<sup>3+</sup> ion can be used effectively as a local probe. Scanning over the sample, and evaluating the emission spectra, high resolution three dimensional images of the ferroelectric domain structure can be obtained non-destructively and free of topographical artifacts. The achieved contrast is based on the observation that even in well tempered samples, differences in intrinsic electric (strain) fields and defect arrangements exist for regions which have been domain inverted, are as grown, and most notably, are in the  $\approx 1 \mu\text{m}$  wide region between oppositely poled domains.

**Keywords:** waveguide imaging, PPLN, optical spectroscopy, Er<sup>3+</sup>

## Introduction

Nonlinear light sources based on periodically poled LiNbO<sub>3</sub> have gained a great deal of attention due to low thresholds, ease of operation, and wide tuning range. In particular, the integrated optical versions of them, achieve, due to the confinement of light over long interaction lengths very high conversion efficiencies (see e.g.: [1, 2]). In order to optimize the latter, both the Ti-profile and the domain structure have to be engineered precisely and effects of interaction have to be under control. In order to achieve this goal we developed a novel characterization tool based on the luminescence of well characterized designer defects that we use as probes for the local environment. For our work we chose Er<sup>3+</sup> ions because, through many studies [3, 4, 5, 6, 7] of this rare earth ion in LiNbO<sub>3</sub>, detailed information about the optical transitions and their dependence on the local lattice environment are known. For instance, the spectral shifts, which are a signature for changes in the local electric fields in z-direction and of rearrangement of the surrounding oxygen ions, were characterized by application of electric fields and hydrostatic pressure[8]. In this paper we report how the spectral information can be used to create images of waveguides and periodically poled domain structures with sub- $\mu\text{m}$  spatial resolution.

## Experimental techniques and results

*Samples.* The periodically poled waveguide LiNbO<sub>3</sub> sample was obtained from the group of Prof. Sohler. It was produced in the "standard" way starting from commercial z-cut LiNbO<sub>3</sub> wafers. After Er<sup>3+</sup> and Ti<sup>4+</sup> indiffusion producing a Er<sup>3+</sup>-doped planar layer and Ti:LiNbO<sub>3</sub> channel waveguides, the sample was periodically poled (period 31.42  $\mu\text{m}$ ) by electric field application at room temperature. To remove intrinsic stress, the sample was tempered at  $T > 120^\circ\text{C}$ , such that the domains were no longer visible in a polarization-contrast microscope. Details see Ref. [2].

*Confocal defect luminescence microscopy.* This technique combines the atomic-level information contained in optical spectra with the spatial resolution of a optical microscope. In our set-up the waveguide samples were mounted in a He-microscope cryostat, which allows temperatures down to 4K. The Er<sup>3+</sup> ions were excited by a laser (dye-lasers or 488nm argon laser) through a home-built microscope (NA up to 0.7) operated in the confocal mode. The pinhole usually required in this mode was replaced by a single mode fiber allowing easy change of the excitation source. The resulting emission was detected in the reverse direction through the same microscope, coupled back into the fiber, and separated from the excitation light by a

dielectric mirror. Finally, the emission spectra are measured in a 0.5m-spectrometer equipped with a LN-cooled CCD array allowing the measurement of 10 spectra/s. For imaging purposes, the microscope is scanned in three dimensions over the samples in fine steps (200nm) and the first and second moments of the recorded spectra ( $\approx 10^5$ ) are determined and displayed as a function of the probing position.

*Waveguide imaging.* In earlier studies [9, 10] we have shown that the optical spectrum of  $\text{Er}^{3+}$  depends strongly on the concentration of the ions  $\text{Ti}^{4+}$  or  $\text{Zn}^{2+}$  which are used to define the waveguide. Most notably, we found a strong inhomogeneous broadening of the spectral lines and a small spectral shift. Due to the nature of these changes, we obtain the highest contrast if the second moment is evaluated. An example of an image is shown in Fig. 1. For this image we excited the  $\text{Er}^{3+}$  ions at 652nm and detected their emission at around 980nm. However, other wavelength combinations work as well. The image was recorded by scanning the front surface of the waveguide focussing about  $10 \mu\text{m}$  inside the sample. The best contrast is achieved when the spectra are recorded at low temperature.

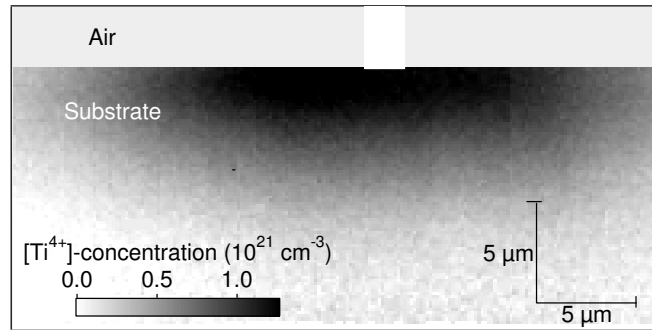


Fig.1. Image of the  $\text{Ti}^{4+}$  profile defining a single mode waveguide. The image is obtained by evaluating the emission spectra while probing over the front face of the sample

*Imaging periodically poled domain structures.* Figure 2 shows one out of the five measured emission bands in the green which were recorded under 488nm excitation for regions with different domain orientation (1,2) and between the domains (3). The differences are very small but notable. It has been found earlier that domain inversion changes the distribution of defect sites[11] leading to different relative emission intensities of the corresponding emission peaks. Comparing region (1) and (2), we find a small decrease on the high energy side and an increase on the low energy side which is consistent with this interpretation. More pronounced is the difference for region (3), for which a clear spectra shift is observed, which can be attributed, at least partially, to a decreased electric field in z-direction experienced by the  $\text{Er}^{3+}$  ion. The spectral changes can be quantified more precisely by evaluating the first moment for the complete spectrum. Figure 3b shows a scan along the horizontal direction covering several changes in the domain orientation. Especially for the first moment (dotted line) clearly different values are obtained for the three regions making it very suitable for imaging as shown in Fig. 3a. The image has good contrast and shows fine details of the structure. It was taken mainly in regions without waveguides. On the bottom of the image a  $\text{Ti}^{4+}$  diffused waveguide region starts, leading to a gradual change of the emission spectra and the corresponding moments.

The second moment (solid line), which characterizes the spectral width or spread of the spectra, emphasizes very strongly the change occurring in-between domains. Indeed, it is expected that changes in the intrinsic electric field, obviously occurring in these regions, will change the Stark splitting of the 4f-levels and hence the spread of the spectrum and the second

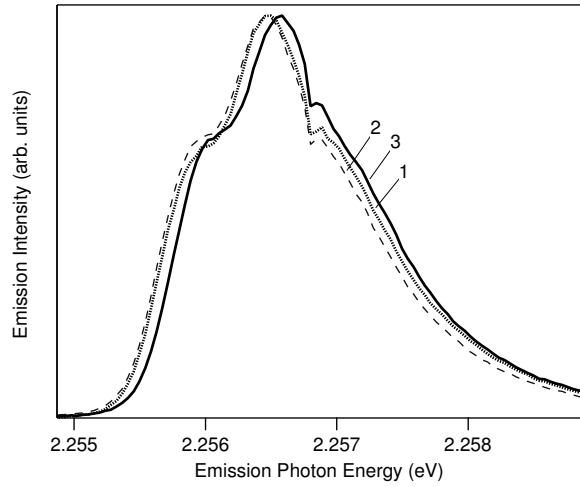


Fig. 2. Expanded view of the measured emission spectra for the regions 1,2, and 3 indicated in Fig. 3 obtained under excitation with 488nm. Only one out of the 5 measured emission bands is depicted. Changes in the other peaks are even less pronounced.

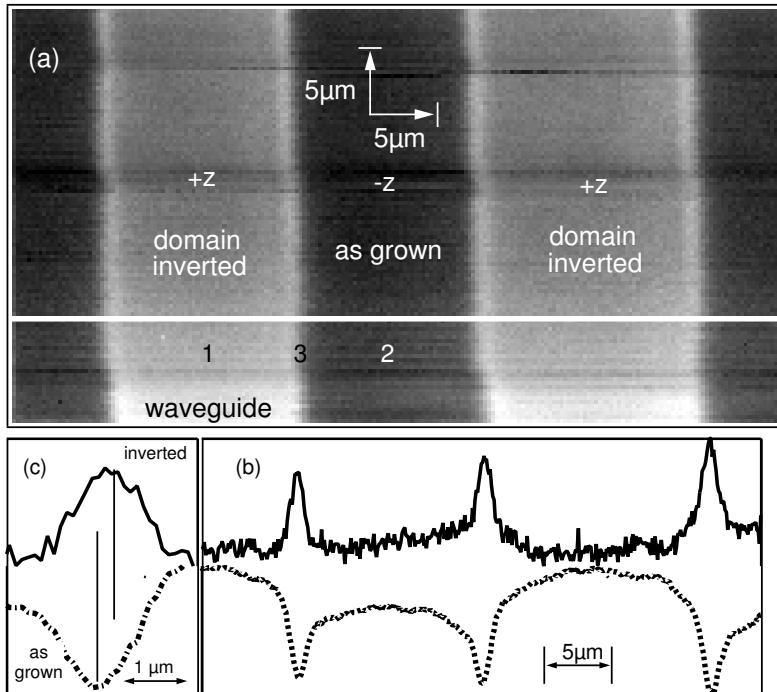


Fig. 3. (a) Image of a periodically poled LiNbO<sub>3</sub> sample obtained by evaluating the first moment of the optical spectra of Er<sup>3+</sup> measured in a scanning confocal luminescence microscope. (b) Line scan of the first (dotted line) and second moment (solid line) of the spectrum. (c) same as (b), but in expanded scale in the region of the domain wall.

moment. Looking even more closely (Fig3c) at the line scans of the first and second moment in the transition regions reveals a small offset indicating that not only one cause is responsible for the change. We assume that we are dealing in the domain wall region with both a change in intrinsic electric field and a redistribution among the possible  $\text{Er}^{3+}$  defect sites. The two obviously do not (and do not have to) coincide with each other spatially.

The rate of change in the moments indicate that our spatial resolution is better than  $1\mu\text{m}$  even though only an objective with numerical aperture of 0.3 was used in these measurements allowing still room for improvement. While all the measurements have been performed at low temperatures (in order to extract details), we found that optical spectra taken at room temperature are sufficient for imaging purposes.

## Summary and Conclusions

We demonstrated the possibility of imaging  $\text{Ti}^{4+}$ -concentration profiles and ferroelectric domain structures in  $\text{Ti}:\text{Er}:\text{LiNbO}_3$  waveguides by defect luminescence microscopy. Although, the underlying mechanism responsible for the contrast, which is an interesting topic all by itself, is not completely clarified, yet, this non-invasive technique may prove especially valuable for development of waveguide technology on  $\text{LiNbO}_3$  material, which has been modified in terms of stoichiometry, co-doping and which underwent different thermal and/or chemical treatments. The concentration of e.g.:  $\text{Er}^{3+}$  probe ions can be kept very low permitting application of the method in samples for which the functionality of the ion is otherwise not required. Traces ( $10^{-3}\text{mol}\%$ ) introduced by doping or diffusion are sufficient. Due to the fact that our imaging technique is based on evaluating the spectral information, it is insensitive to intensity fluctuations and is free of topographic artifacts.

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