



# Spectroscopic characterizations and optical damage resistance of Zn:Yb:Er:LiNbO<sub>3</sub> crystals

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## Abstract

Zn:Yb:Er:LiNbO<sub>3</sub> crystals have been grown. The infrared transmission spectra were measured and discussed in terms of the spectroscopic characterizations and the defect structure of the Zn:Yb:Er:LiNbO<sub>3</sub> crystals. The optical damage resistance was characterized by the transmitted beam pattern distortion method. The optical damage resistance of Zn (6.0 mol%):Yb:Er:LiNbO<sub>3</sub> crystal is about two orders of magnitude higher than that of other crystal. The dependence of the optical damage resistance on the defect structure was studied.

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## 1. Introduction

The electro-optic, acousto-optic and non-linear properties of LiNbO<sub>3</sub> (LN) crystals, combined with the laser emission of trivalent erbium ions

(Er<sup>3+</sup>) around 1.55 μm, together with the possibility of producing low-loss waveguides, have led to choose this material in order to develop monolithic integrated devices [1–3]. Also, the energy transfer between Yb<sup>3+</sup> and Er<sup>3+</sup> in LiNbO<sub>3</sub> crystals has been recently characterized under CW and pulsed excitation [4,5], and it has been found that this transfer is highly efficient [6].

It is known that pure LiNbO<sub>3</sub>, as well as Er:LiNbO<sub>3</sub> and Yb:Er:LiNbO<sub>3</sub>, presents a low threshold for photorefractive damage [7]. When

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LiNbO<sub>3</sub> devices are used at high laser intensity, their performance is severely limited by the optical damage effect, which induces birefringence change and distorts the laser beams [8]. Some damage-resistant impurities have been discovered, including divalent (Mg<sup>2+</sup> [9], Zn<sup>2+</sup> [10]) and trivalent (In<sup>3+</sup> [11,12], and Sc<sup>3+</sup> [13]) impurities, which lead to a strong decrease in the photo damage of LiNbO<sub>3</sub> and have been receiving a lot of interest.

In this paper, doping over a range of Zn concentration is used to enhance the optical damage resistance of Zn:Yb:Er:LiNbO<sub>3</sub> crystals. The dependence of optical damage resistance on the concentration of ZnO is discussed. The IR transmission spectra of the Zn:Yb:Er:LiNbO<sub>3</sub> crystals were measured to investigate the structure of the crystals.

## 2. Experimental

### 2.1. Crystal preparation

The congruent LiNbO<sub>3</sub> crystals with 0.5 mol% of Er<sub>2</sub>O<sub>3</sub>, 1.0 mol% of Yb<sub>2</sub>O<sub>3</sub>, and various concentrations (0, 3.0, and 6.0 mol%) of ZnO were grown by Czochralski method. For comparison, Er:LiNbO<sub>3</sub> crystal was also grown. The raw materials used for crystal growth are Li<sub>2</sub>CO<sub>3</sub> (4N purity), Nb<sub>2</sub>O<sub>5</sub> (4N purity), ZnO (4N purity), Yb<sub>2</sub>O<sub>3</sub> (4N purity) and Er<sub>2</sub>O<sub>3</sub> (spectral purity). The melt composition for several crystals is shown in Table 1. To prepare the doped LiNbO<sub>3</sub> polycrystalline materials, the thoroughly-mixed raw materials were put into a platinum (Pt) crucible, and calcined at 750 and 1150 °C for 2 h, respectively. The crystals were grown under the

optimum conditions: temperature gradient of 430–50 °C/cm, rotation rate of 12–20 rpm, and growth rate of 1.0–1.5 mm/h. After growth, the crystals were cooled to room temperature at a rate of 150 °C/h. All of the crystals were clear and transparent.

The crystals were placed in a furnace where the temperature gradient is below 5 °C/cm for polarizing. After being held at a temperature of 1200 °C for 8 h, the crystals were polarized with 5 mA/cm<sup>2</sup> current density. During the cooling process, the temperature was decreased rapidly from 1000 to 800 °C to avoid “dissolve-off” phenomenon from appearing. Test samples were cut from the middle of the crystals and polished to optical grade smoothness. The size of the samples is listed in Table 1.

### 2.2. Measurements

The infrared transmission spectra of the crystals were obtained with a Nicolet Avatar-370 FT-IR spectrometer in the 3000–4000 cm<sup>-1</sup> range at room temperature.

The transmitted beam pattern distortion method [14] was used to study the optical damage resistance of the Zn:Yb:Er:LiNbO<sub>3</sub> crystals. Fig. 1 shows the experimental setup. Light from an Ar<sup>+</sup> ion laser (488.0 nm) was focused by means of a convex lens of focal length,  $f$ , onto a crystal sample placed in the focal plane. The laser beam is polarized parallel to the  $c$ -axis of the crystal. The diameter of the focused laser beam is related to the incident beam diameter,  $d$ , determined by the diaphragm, and yields the focused laser beam area given in Eq. (2) for  $S$ .

$$D = \frac{2f\lambda}{\pi d}, \quad (1)$$

Table 1  
Composition of raw materials of the Zn:Yb:Er:LiNbO<sub>3</sub> crystals and the size of the samples

Crystal	No.1	No.2	No.3	No.4
[ZnO] (mol%)	0	0	3.0	6.0
[Er <sub>2</sub> O <sub>3</sub> ] (mol%)	0.5	0.5	0.5	0.5
[Yb <sub>2</sub> O <sub>3</sub> ] (mol%)	0	1.0	1.0	1.0
Crystal size (mm <sup>2</sup> )	φ35 × 50	φ30 × 40	φ30 × 40	φ30 × 40
Wafer size (mm <sup>3</sup> )	10 × 10 × 3	10 × 10 × 3	10 × 10 × 3	10 × 10 × 3

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