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Influence of post-growth treatment on the optical properties of In:Ce:Cu:LiNbO<sub>3</sub> crystals

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# Abstract

Congruent In (3 mol%):Ce:Cu:LiNbO<sub>3</sub> crystals have been grown by the Czochralski method in air. Some crystal samples were reduced in Li<sub>2</sub>CO<sub>3</sub> power, and others were oxidized in Nb<sub>2</sub>O<sub>5</sub> power. The structure of crystals was studied by an infrared transmittance spectrum. The resistance ability to optical damage and the photorefractive properties were measured by light-induced scattering experiments and two-beam coupling, respectively. It has been found that the reduction treatment increased the photoconductivity , which resulted in decreased erasure time and diffraction efficiency, but higher light-induced scattering resistance ability. The oxidation treatment caused the inverse affect. Finally, the nonvolatile holographic recording in In:Ce:Cu:LiNbO3 crystals is realized.  $\bigcirc$  2008 Elsevier GmbH. All rights reserved.

Keywords: In:Ce:Cu:LiNbO3; Post-treatment; Diffraction efficiency

# 1. Introduction

LiNbO<sub>3</sub> (LN) crystals were widely used in many areas because of their excellent piezoelectric, electro-optic and nonlinear optic properties [1,2]. Because of its excellent photorefractive properties, LN crystals can be applied in many areas, such as piezoelectric, electro-optic, surface acoustic wave, waveguide and nonlinear optical devices [3–6]. However, optical damage and long response time severely limited its applications in practice. The essential way to solve these problems is to optimize the crystal itself by doping with damage-resistance elements (Zn, In, Mg, Sc, etc.) [7–11] and photorefractive sensitivity elements (Fe, Ce, Mn, Cu, etc.) [12–15]. In addition,

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moderate post-treatment processes of oxidation and reduction can also change the photorefractive properties of the crystals. To obtain good photorefractive properties by different dopants and post-treatment have attracted much attention in holographic optical data storage.

Liu [16] et al. found that nonvolatile holographic recording can be realized in Ce:Cu:LN crystals. But the recording time was long, doped In ions can improve these properties and moderate post-treatment processes may obtain good photorefractive properties. We obtained a series of high-quality doped In:Ce:Cu:LN crystals grown from the congruent composition, and studied their photorefractive properties after different treatments (oxidation, reduction).On the basis of experimental results, the dependence of the optical damage resistance and photorefractive response time on the defect structure are discussed.

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### 2. Experimental procedure

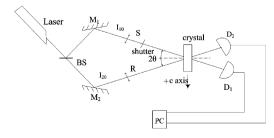
## 2.1. Crystal growth and sample preparation

In(3 mol%):Ce(0.1 wt%):Cu(0.02 wt%):LiNbO<sub>3</sub> single crystals were grown in an automatic diameter control system by the Czochralski method using an intermediate frequency (IF) furnace. The raw materials used to grow the crystals were Li<sub>2</sub>CO<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub>, In<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub> and CuO. All raw materials were of 99.99% purity. The Li/Nb ratio of the initial melts to grow the congruent LiNbO<sub>3</sub> is 0.946. The crystals were grown along the *c*-axis in air from the polycrystalline material in a diameter-controlled equipment at a rate of 2 mm/h and a rotating rate of 15–25 rpm. The growth temperature gradient of the IF furnace was 35-40 °C/cm. After growth, the crystals were cooled down to room temperature at a speed of 50 °C/h. They were then polarized in another furnace, where the temperature gradient was almost zero with a DC electric density of  $5 \text{ mA/cm}^2$  for 30 min. Finally, the crystals were v-cut into slices with the size of  $10 \times 2 \times 10 \text{ mm}^3$  (a × b × c). Some of the samples were buried in Li<sub>2</sub>CO<sub>3</sub> power to be reduced at 550 °C for 30 h, and some were buried in Nb<sub>2</sub>O<sub>5</sub> power to be oxidized at 1150 °C for 15 h. All the samples were polished by SiC power to optical-grade smoothness.

#### 2.2. Measurements

The IR transmittance spectra of the crystals were recorded with a Fourier spectrophotometer at room temperature. The measurement wave number range was from 3300 to  $3600 \text{ cm}^{-1}$ .

To measure the diffraction efficiency and the erasure time, the two-wave coupling method was carried out. A scheme of experimental set-up is shown in Fig. 1. The holographic gratings were written by two extraordinarily polarized beams, the wavelength of the laser is 532 nm. After the grating was built, the S beam was blocked and we obtained the



**Fig. 1.** Experimental set-up of detect diffraction efficiency: BS: beam splitter; M1, M2: mirrors; D1, D2: detector; S: signal beam; R: reference beam; PC: computer.

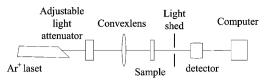


Fig. 2. Experimental set-up for photo scattering resistance ability.

diffraction efficiency:

$$\eta = \frac{I_{\rm d}}{I_{\rm d} + I}$$

where  $I_t$  is the transmitting intensity of the signal beam before the grating was built, and  $I_d$  is the diffractive intensity of the signal beam after the grating was built.

Light-induced scattering was used to characterize the resistance ability to the optical damage for crystals. It was performed with a He–Ne laser at the wavelength of 632.8 nm. An attenuator could adjust the laser incident power, and the beam polarizing direction was parallel to the *c*-axis. The crystals were placed on the focal plane of the convex lens; the central power density of the transmitted beam was record with a detector. Fig. 2 shows the experimental set-up.

# 3. Results and discussions

## 3.1. Infrared transmittance spectra

Because of  $H_2O$  vapor in the air,  $H^+$  ions were introduced into the crystals during the growth process and formed the O-H band. We can analyze the change of the OH<sup>-</sup> absorption peak to conjecture the position of doped ions. The infrared transmittance spectra of crystals are shown in Fig. 3. The OH<sup>-</sup> absorption peak of the Ce:Cu:LiNbO<sub>3</sub> crystal is located at  $3484 \text{ cm}^{-1}$ [17]. When the  $In^{3+}$  concentration is above its threshold concentration, a part of  $In^{3+}$  ions begin to occupy Nb sites and exist in the form of  $In^{2-}_{Nb}$ . Because  $In^{2-}_{Nb}$  had a higher ability to attract H<sup>+</sup> than that of Li vacancies, OH<sup>-</sup> vibration needs more energy, the absorption edges of the crystal shift to short wavelength, which is responsible for the absorption peak at  $3508 \,\mathrm{cm}^{-1}$ . After treatment, the position of the absorption peaks of In(3 mol%):Ce:Cu:LiNbO<sub>3</sub> is just equal to that of asgrown. The treatments seem to have little influence on the position of absorption peaks; this indicates that the post-grown treatments do not influence the defect structure of In(3 mol%):Ce:Cu:LiNbO<sub>3</sub>. This indicates that the In concentration in crystals is over the threshold and the post-grown treatments do not influence the defect structure.

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