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Refractive index change in Ti-diffused near-stoichiometric LiTaO₃ waveguide and its relation to Ti-concentration

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- Near-stoichiometric (NS) Ti:LT multimode planar waveguide was fabricated.
- Both index change and Ti-concentration in the waveguide follow Gauss profile.
- Extraordinary and ordinary index change $\Delta n_{e,o}$ follows an almost same profile.
- Δn_e and Δn_o have an almost same
exponential relation to Tiexponential concentration.
- Congruent or stoichiometric Ti:LT and Ti:LN waveguides show different properties.

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ABSTRACT

Ti-diffused near-stoichiometric(NS) LiTaO₃ multimode planar waveguide was fabricated by diffusion of Ti-film coated onto a congruent LiTaO₃ substrate and post Li-rich vapor transport equilibration process. The crystalline phase in the Ti-diffused layer was analyzed by powder x-ray diffraction and the Li₂Ocontent was evaluated from the measured birefringence. The Ti-concentration profile was analyzed by secondary ion mass spectrometry. The modes guided in the planar waveguide were characterized by prism-coupling technique. The refractive index profile was constructed from the measured mode indices, and correlated with the Ti-concentration profile. The results show that the waveguide retains the LiTaO₃ phase and is in an NS composition environment. Both the refractive index and Ti-concentration follow Gaussian profile. The extraordinary and ordinary index change profiles are almost same, implying that the electric field profile of the mode guided in the NS Ti:LiTaO₃ waveguide would be polarizationinsensitive. The ordinary and extraordinary index changes and the Ti-concentration follow a similar exponential relationship with a power index \sim 1.3. Comparison shows that the Ti:LiTaO₃ waveguide

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<https://doi.org/10.1016/j.matchemphys.2017.10.018> 0254-0584/© 2017 Elsevier B.V. All rights reserved. shows considerable differences from the Ti:LiNbO3 in Ti diffusivity, index change profile and its relation to Ti-concentration, and polarization dependence of guided mode.

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1. Introduction

 $LiNbO₃$ (LN) and LiTaO₃ (LT) have similar crystal and defect structures, and similar applications. For the LT, most of its properties are as favorable as those of LN and some are even advantageous, such as two orders of magnitude higher resistance to optical damage, higher quasi-phase-matching frequency conversion efficiency, and better candidate for UV radiation generation. Moreover, a near-stoichiometric (NS) LT displays a number of advantages over the congruent LT, such as larger electro-optic and nonlinear optical effects, two orders of magnitude higher resistance to optical damage, one order of magnitude lowered coercive field for ferroelectric microdomain reversal $[1-3]$ $[1-3]$. Like the LN, either proton exchange (PE) or Ti diffusion technique can be used to fabricate an LT waveguide. It is well known that the PE LN or LT waveguide suffers from many problems such as electro-optic coefficient degradation, complicated crystalline phases, larger loss, low stability due to high H^+ mobility, and lifetime shortening of electronic transition of rare-earth dopants such as Er^{3+} , Nd³⁺ [\[4\]](#page--1-0). In contrast, Ti-diffused LN (Ti:LN) waveguide is of a number of merits such as lower loss (as low as 0.1 dB/cm [\[5\]](#page--1-0)), higher thermal, electric and chemical stabilities, retained crystal structure, crystalline phase and hence the electro-optic property [\[6\]](#page--1-0) and spectroscopic feature of doped rare-earth ion. In words, Ti-diffused NS LT (Ti:LT) waveguide is more promising than the NS Ti:LN. Nevertheless, due to the low Ti diffusivity, less work has been done on the Ti:LT waveguide. Some results concerning with Ti diffusivity, optical and electrooptic properties of Ti:LT have been reported previously $[7-12]$ $[7-12]$. Ti-induced refractive index change and its relation to the Ticoncentration are crucial to the related devices. The relevant knowledge could not be found for the NS Ti:LT waveguide. Present work focuses on the study of the NS Ti:LT waveguide that is fabricated by Ti-diffusion and post vapor transport equilibration (VTE) process.

2. Experiment

The Ti-diffused NS LT multimode planar waveguide was fabricated starting from a congruent Z-cut LT with a technological process in sequence of diffusion of 300 \pm 2 nm thick Ti-film at 1300 °C for 20 h and post Li-rich VTE at 1100 °C for 10 h. To increase the diffusion rate, a higher diffusion temperature of 1300 \degree C was adopted. It is well known that the Curie temperature (T_c) of the congruent LT crystal is ~650 °C, which is far lower than the diffusion temperature adopted, 1300 \degree C. This implies that the ferroelectric domain in the studied sample orients no longer conformably but randomly. The problem may be solved by carrying out uniform ferroelectric poling treatment on the whole sample plate after the waveguide fabrication. After fabrication, the crystalline phase, $Li₂O$ content, waveguide characteristics, refractive index and Ticoncentration profiles in the Ti:LT planar waveguide were characterized.

X-ray powder diffraction experiment was performed on the NS Ti:LT waveguide so as to verify that the waveguide layer retains still the LT crystalline phase. This is not easily realized as the waveguide layer is quite shallow with a thickness of only a few tens of micrometers. If the whole substrate with a thickness of 0.5 mm is directly powdered and used for the x-ray analysis, the obtained diffraction data are contributed mainly from the LT substrate but not from the waveguide layer. To solve the problem and characterize the crystalline phase in the waveguide layer as accurately as possible, we have fabricated another NS Ti:LT planar waveguide sample under the same condition and substantially reduced its thickness by grinding the sample rear where is the clear LT without the waveguide. The final thickness of the thin plate is about 40 um. which is comparable to the thickness of Ti-diffused layer as shown below, around 20 μ m. The thin plate was then fine powdered and used for x-ray analysis. The analysis was accomplished by a Rigaku D/MAX-2500 x-ray diffractometer equipped with a Cu target. The target voltage and current operated at 40 kV and 100 mA, respectively. The scanning range of the 2θ angle was 5-90°. The scanning speed and step were 8 deg/min and 0.02° , respectively. For comparison, the analysis was also performed on the corresponding asgrown congruent LT crystal.

Li2O-content on the crystal surface was estimated from the birefringence measured by prism coupling technique [\[13\]](#page--1-0). To achieve that, the ordinary and extraordinary refractive indices at the undoped part of crystal surface were measured, and the surface $Li₂O$ -content was evaluated from the measured indices. The index measurements were accomplished by a commercial Metricon 2010 prism coupler (Metricon Corp., Pennington, NJ), which has a working principle of measurement of critical angle of total reflection. The 632.8, 1311 and 1553 nm lasers equipped in the system were used as the working sources. The index measured by this method should be the value at the crystal surface because the total reflection phenomenon occurs there. It is convenient to choose a transverse magnetic (TM) or a transverse electric (TE) polarization scheme to measure the ordinary or extraordinary index, depending on the cut of the plate to be measured.

The prism coupling technique was also used to characterize the modes guided in the planar waveguide [\[14\]](#page--1-0). Both TE and TM modes were characterized at the 633 1311 and 1553 nm wavelengths. All measurements were carried out at the room temperature 24.5 \pm 0.1 °C.

The Ti-concentration was profiled by secondary ion mass spectrometry. A time-of-flight secondary ion mass spectrometer (ToF SIMS V, ION-TOF GmbH) was used to analyze the depth profiles of the diffused ⁴⁸Ti and the substrate elements ⁶Li, ¹⁸¹Ta and ¹⁶O. A $Cs⁺$ -beam of 30 nA at 3 keV was used to sputter a crater of $120 \times 120 \mu m^2$ on the waveguide surface and a pulsed bismuth ion beam (pulsed current: 1 pA, pulsed energy: 25 keV) was used to analyze the yields of secondary ions ⁶Li, ¹⁸¹Ta, ¹⁶O and ⁴⁸Ti as a function of time. Positive secondary ions were detected. Ions from a central area of 19 \times 19 μ m² inside the erosion crater rastered on planar waveguide surface were detected. During the analysis, a low-energy pulsed electron gun was used to neutralize the positive charges to degrade the surface charge accumulation. For the same purpose, before the analysis a 45-nm thick Au film was coated on the sample surface. The trace and depth of each erosion crater were measured by a Tencor Alpha Step 200 profilometer. The depth resolution is determined by the roughness of crater and is better than 5 nm in our case.

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