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Electro-optic coefficients of a non-congruent lithium niobate fabricated by vapour transport equilibration: Composition effect

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ABSTRACT

Composition effect on electro-optic (EO) properties of a LiNbO₃ (LN) single-crystal has been investigated in a Li₂O-content range of 47.0–49.95 mol%. Some non-congruent LN crystals with different Li₂O-contents were prepared by performing Li-deficient or Li-rich vapour transport equilibration treatments on as-grown congruent LN crystals. Unclamped EO coefficients γ_{13} and γ_{33} of these samples were measured by a Mach-Zehnder interferometric method. The measurements show that in the Li-deficient regime both γ_{13} and γ_{33} increase by ~8% as Li₂O-content decreases from the congruent 48.6 mol% to the 47.0 mol% in the Li-deficient regime. The feature is desired for the EO application of the Li-deficient crystal. In the near-stoichiometric regime, both γ_{13} and γ_{33} reveal a non-monotonic dependence. As the Li₂O-content increases from the 48.6 mol%, the EO coefficient decreases. Around Li₂O-content 49.5 mol%, a minimum is reached. After that, the EO coefficient recovers slowly. At the stoichiometric composition, it recovers to a value close to that at the congruent point. Comparison shows that different crystal growth methods give rise to different defect structure features and hence different composition effects.

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1. Introduction

LiNbO₃ (LN) is widely investigated due to its good acousto-optic, electro-optic (EO) and nonlinear optical properties. By utilizing its excellent EO property, various passive [1–3] and active devices [1,4–7] have been demonstrated. As the composition approaches stoichiometry, the crystal, called near-stoichiometric (NS) LN (Li₂O-content >49 mol%), exhibits a number of attractive features such as larger nonlinearity, lower coercive field for domain reversal, largely lowered threshold concentration of photorefractive-damage-resistant dopants such as Mg, Zr, Sc, etc. In addition, a non-congruent, Li-deficient LN may be used to develop active waveguide devices as such crystal enables to promote the solubility of rare earth ion and hence the optical gain of an amplifier or laser [8,9]. Alternatively, a Li-deficient/NS LN plate may be fab-

ricated by performing a post Li-deficient/Li-rich vapour transport equilibration (VTE) treatment on a congruent LN crystal.

To design and implement an EO device based on an NS/Li-deficient LN, it is essential to know the composition effect on the EO coefficients of an LN substrate. Over the past years, people has studied the effects of various dopants on the EO coefficients of LN. These include Cr³⁺ [10], Hf⁴⁺ [11], Mg²⁺ [12–14], Ti⁴⁺ [14,15], Zr⁴⁺ [16], Fe³⁺ [17], Zn²⁺ [18], H⁺ [19–21], Er³⁺ [22,23] and In³⁺ [23]. Among them, the Cr³⁺, Zn²⁺ and H⁺ dopants have a relatively large effect while the others have a weak effect. In addition, people have also studied the stoichiometry effect on the EO property of LN (mainly in the NS regime) for the crystals prepared by different growth methods [10,13,24–27]. The study has shown that the crystal composition takes a large effect, and different crystal growth methods cause different crystal composition effects on the EO coefficient. For the Li-rich VTE preparation method, which involves Li diffusion into the LN crystal, the lattice is already present before the VTE treatment. The ion environment in cells generated after the VTE may be distinct from that after the crystal growth. It is unclear what composition effect is caused by the Li-rich VTE. It is necessary to perform an independent investigation on the EO properties

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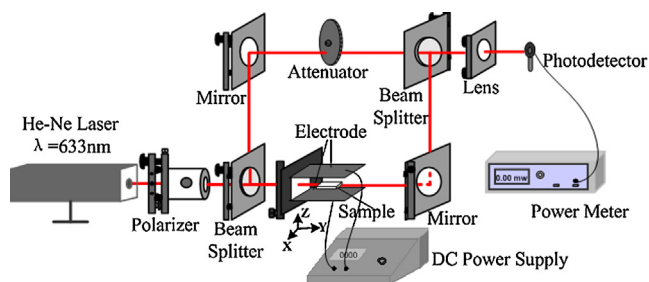


Fig. 1. Schematic of a Mach-Zehnder interferometer for the EO coefficient measurement of LNs.

of NS LN prepared by Li-rich VTE. In addition, it is also interesting and crucial to carry out a study in the Li-deficient regime (defined as Li_2O -content < 48.4 mol%) by using the Li-deficient crystal prepared by Li-poor VTE. Present work aims at these studies.

2. Experiment

Some LN plates with different Li_2O -contents ranging from 47.0 to 49.95 mol% were fabricated by performing Li-deficient or Li-rich VTE treatments on 1.0 mm thick congruent LN plates (Z-cut). Table 1 summarizes the sample plates and their Li_2O -contents, determined from the birefringence measured at the wavelengths of 0.633 and 1.553 μm using prism coupling technique [28].

Interferometric technique has been used to measure many characteristic parameters of the pure or doped LN, such as the EO coefficients [22,23,26,29], elastic constants and photoelastic coefficients [30], and the piezo-optic coefficients [31,32]. Here, the Mach-Zehnder interferometry was applied to study the EO properties of LN crystals. The schematic of measurement is sketched in Fig. 1. The working light source employed a helium-neon laser with an output wavelength of 0.633 μm . To modulate the light phase, a direct-current (DC) voltage was applied to the crystal along its optical axis direction. The voltage was applied by two different schemes A and B. The scheme A used two copper slabs as the electrodes, as shown in Fig. 1. In the scheme B case, two aluminum films thermally evaporated onto the crystal surfaces were used as the electrodes. In both cases of schemes of A and B, a Teflon holder was used to fix the sample to be measured. Since a DC voltage was used to drive the crystals, the γ_{13} and γ_{33} measured from both schemes A and B are unclamped according to the definition [26]. The DC voltage dependence of optical power of the zero-order interference fringe was monitored. Based on the dependence we can derive the half-wave voltage, named V_π .

3. Results and discussion

First of all, we give a brief description for the measurement principle. As described above, by using the Mach-Zehnder interferometry we can measure the half-wave voltage V_π , defined as the voltage under which the optical phase changes π over the length y of interaction between the light wave and the electric field, i.e.,

$$\Delta\Phi = (2\pi/\lambda_0)\Delta n_{o,e}y = \pi, \quad (1)$$

where λ_0 is the working wavelength, y is actually the size of sample along the Y direction. $\Delta n_{o,e}$ is the corresponding index change associated with the EO effect:

$$\Delta n_o = (1/2)\gamma_{13}n_o^3V_\pi/z, \quad \Delta n_e = (1/2)\gamma_{33}n_e^3V_\pi/z, \quad (2)$$

where n_e (n_o) is the extraordinary (ordinary) index of refraction, z is the separation of the two electrodes. Table 1 brings together the y and z values for each sample. In Eq. (2), V_π/z denotes the electric field strength, and Δn_o and Δn_e correspond to the two

cases that the light wave is ordinarily and extraordinarily polarized, respectively. Inserting Eq. (2) into (1), one can find

$$\gamma_{13} = \lambda_0 z / (y n_o^3 V_\pi), \quad \gamma_{33} = \lambda_0 z / (y n_e^3 V_\pi). \quad (3)$$

With the known half-wave voltage V_π one can obtain the EO coefficients γ_{13} or γ_{33} .

The accuracy of the measurement system has been confirmed by using it to measure a standard congruent LN and comparing the results with those values reported previously. This is the case for the results measured from both schemes A and B [13], indicating that the use of the Teflon sample holder has little influence on the measurement and the results measured using our system are convincing. The reliability of the system of the scheme A was further confirmed by doing the measurements on a sample that was placed at different positions within the Cu slab electrodes [13]. The measurements show that the EO coefficients do not change with the position where the sample was placed, further verifying the measurement system. The EO coefficients measured using our system have a relative error of $\pm 3\%$ in total, which includes the uncertainties of 0.3%, 1.0%, 0.2% and 1.5% for the relevant parameters y , z , $n_{o,e}$ and V_π in Eq. (3), respectively.

In addition, it is well known that the LN crystal suffers from serious photorefractive damage in visible and near infrared regions. As the photorefractive effect is actually caused by the electro-optic effect of the LN crystal, it is unclear if the photorefractive damage affects the experimental results for the working light intensity (10^2 W/m^2) adopted in the measurements. In our previous work [15], we exemplified a pure congruent LN to demonstrate the working light intensity and hence the photorefractive effect on the EO coefficients γ_{13} and γ_{33} . The results show that the EO coefficients do not change as the working light intensity is below 10^2 W/m^2 . As the light intensity adopted in our measurements is 10^2 W/m^2 , the optical damage should not take an effect on the result. In words, the EO coefficients measured using our experimental system are reliable and convincing.

The obtained γ_{13} and γ_{33} values of the LNs with different Li compositions are brought together in Table 1. The values outside/inside the parentheses corresponds to the EO coefficient measured from the voltage-applying scheme A/B. The Li_2O -content dependence of the EO coefficients is more clearly illustrated in Fig. 2 with the red/blue balls corresponding to the scheme A/B.

We note from Fig. 2 that the EO coefficients γ_{13} and γ_{33} obtained from the schemes A and B show a systematic error and the results measured from the scheme A are larger. This is true not only for the congruent LN but also for the non-congruent LN. The systematic error between the schemes A and B accounts for 7.5% and 5.3% in the cases of γ_{13} and γ_{33} , respectively, and definitely exceeds the experimental error of 3%. We consider that the inhomogeneity of externally applied field accounts for the systematic error. In the scheme A case, the largest size of the measured sample $x \times y = 10.2 \text{ mm} \times 13.7 \text{ mm}$ is considerably smaller the geometry of the electrodes ($50 \text{ mm} \times 41 \text{ mm}$), implying that the samples in this case are in an approximately uniform environment of electric field. In the scheme B case, however, the aluminum film is thermally evaporated onto the sample surface. In this case, the electric field at the sample edge should be different from that at the sample center. The difference of electric field homogeneity between the cases A and B consequently leads to different values of V_π , which yield different EO coefficient values. It is no doubt that the coefficient measured from the scheme A is closer to the practical scenario. The voltage-applying scheme B is usually adopted to realize a device [4–7].

We also note from Fig. 2 that in the Li-deficient region both γ_{33} and γ_{13} increase as the Li_2O -content is decreased. The increase is of $\sim 8\%$ as Li_2O -content decreases from the congruent 48.6 mol% to the 47.0 mol% in the Li-deficient region. The increase consider-

Table 1

Summary of Li_2O -content, interaction length y between applied electric field and light wave propagated in crystal, spacing z of electrodes, and unclamped EO coefficients γ_{13} and γ_{33} in two measurement cases (data outside/inside the parentheses correspond to the case A/B).

Li_2O -content (mol%)	y (mm)	z (mm)	γ_{13} (pm/V)	γ_{33} (pm/V)
(1) 46.9 ± 0.1	7.1 (7.1)	3.4 (1.0)	11.3 ± 0.3 (10.4 ± 0.3)	34.8 ± 0.8 (33.0 ± 0.8)
(2) 47.4 ± 0.1	8.0 (8.0)	3.4 (1.0)	11.0 ± 0.3 (9.9 ± 0.3)	33.9 ± 0.8 (32.0 ± 0.8)
(3) 48.0 ± 0.1	12.9 (12.9)	3.4 (1.0)	10.8 ± 0.3 (9.8 ± 0.3)	33.3 ± 0.8 (31.3 ± 0.8)
(4) 48.6 ± 0.1	13.0 (13.0)	3.4 (1.0)	10.4 ± 0.3 (9.7 ± 0.3)	32.2 ± 0.8 (30.7 ± 0.8)
(5) 49.5 ± 0.1	13.7 (13.7)	3.4 (1.0)	10.1 ± 0.3 (9.3 ± 0.3)	31.2 ± 0.8 (29.9 ± 0.8)
(6) 49.95 ± 0.1	12.9 (12.9)	3.4 (1.0)	10.5 ± 0.3 (9.6 ± 0.3)	32.3 ± 0.8 (30.6 ± 0.8)

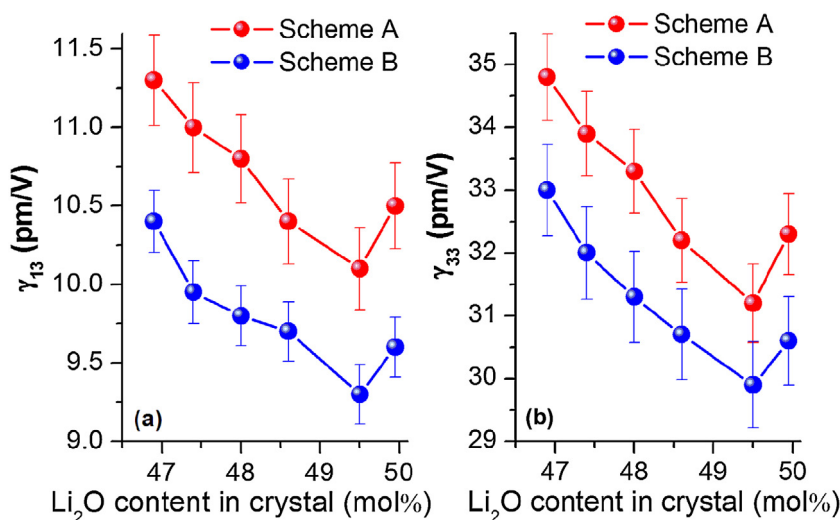


Fig. 2. Unclamped (a) γ_{13} and (b) γ_{33} versus Li_2O -content for two voltage-applying schemes A (red balls) and B (blue balls).

ably exceeds the error of 3%. This observation is favourable for the EO application of the crystal. In the NS regime, both γ_{13} and γ_{33} reveal a non-monotonic dependence on the Li_2O -content for both cases of schemes A and B. As the Li_2O -content increases from the 48.6 mol%, the coefficients continue to decrease. Around the Li_2O -content 49.5 mol%, a minimum is reached. As the Li_2O -content is further increased from 49.5 mol%, the coefficient recovers slowly. At the stoichiometric composition, it recovers to a value close to that at the congruent point.

It is essential to compare the composition effect on the EO coefficient in the NS/Li-deficient regime for the NS/Li-deficient LNs prepared by different methods of growth or post-growth treatment like the Li-rich/Li-deficient VTE adopted here.

In the Li-deficient region, present result is in qualitative agreement with the previous study on MgO-doped LN [13], further confirming the observed effect that the EO coefficient increases as the crystal composition is decreased.

In the NS region, several research groups have studied the effect [10,24–27] and the NS crystals were grown by different methods such as double crucible Czochralski growth (DCCG), K_2O flux and top seeded solid-solution growth (TSSG).

- a In 1999, Kitamura group [24] grew an NS LN with a Li_2O -content 49.8% using the DCCG method, and measured its unclamped EO coefficients γ_{13} and γ_{33} . They found that while γ_{13} remains unchanged γ_{33} increases by >20% (4% error).
- b In parallel, Cabrera et al. grew and measured an NS crystal with an Li_2O -content $49.8 \pm 0.03\%$ by K_2O flux method, and found a reversed effect: both (unclamped) γ_{13} and γ_{33} reduce by 7% (1% error) compared to the congruent LN [25].
- c In 2003, Abarkan et al. [26] have measured γ_{22} and γ_c of one NS sample, without the Li_2O -content and preparation method

specified, and reported that γ_{22} increases while γ_c decreases in comparison with the congruent LN.

- d In contrast, Chah et al. [10] and Abdi et al. [27] (by the same group) performed a more detailed study in the NS regime. Chah et al. grew a series of NS LNs by K_2O flux method and studied the (unclamped) effective EO coefficient $\gamma_c [= \gamma_{33} - (n_o/n_e)^3 \gamma_{13}]$ in the Li_2O -content range of 48.4–50.1 mol%. Their study has revealed that the γ_c reveals a non-monotonous Li_2O -content dependence similar to that observed in Fig. 2, and the minimum takes place at Li_2O -content 49.5 mol%, too. Similar non-monotonous behaviour was also reported by Abdi et al. [27], who grew NS LNs by TSSG method and measured γ_{61} ($= \gamma_{22}$) against the Li_2O -content. Comparison shows that Abdi et al.'s result shows differences from Chah et al.'s and ours in the following two aspects. First, the minimum takes place at different Li_2O -contents, ~49.0 mol% in the Abdi et al.'s study while ~49.5 mol% in Chah et al.'s and ours. Second, the EO coefficient recovers to different values at the stoichiometric composition: a value considerably larger than that at the congruent point in Abdi et al.'s study while a value close to that at the congruent point in Chah et al.'s and ours.

It is concluded from the preceding comparison that the composition effect on the EO coefficient is different from one preparation method of NS crystal to another. In particular, our result of the NS crystal prepared by VTE is close to the result of the NS crystal prepared by K_2O flux, but is different from the result of the NS crystal prepared by the DCCG and TSSG. As reviewed in Section 1, researchers have studied the stoichiometry and various dopants effects on the EO properties of LN, and the effects were interpreted in terms of defect structure. Fontana group has done more studies on the mechanism behind the observations [10,18,26,27], and proposed a model based on the one of inorganic crystal. The model

assumes that the EO coefficient of LN includes electronic, ionic and piezo-optic components. The LN EO property is governed by the polarizability and deformation of LiO_6 and NbO_6 octahedrons. Speaking simply, the LN electro-optic coefficient is influenced by both Li and Nb contents. As for the crystal composition effect, the composition change can be simply thought as the increase or decrease of Li content. The decrease of crystal composition leads to the increase of Li vacancies. Consequently, the lattice becomes more non-rigid and the deformability of the LiO_6 and NbO_6 octahedrons becomes stronger, resulting in an increase of EO coefficient. The composition effect may be also interpreted from the expression for the piezo-optic contribution [27]. The expression is shown to be associated with the composition-dependent piezoelectric and elastic coefficients, which are responsible for the non-monotonous composition dependence of EO coefficient. It should be stressed that the explanations given above are qualitative. A more convincing explanation needs an in-depth theoretical study in the future.

4. Conclusions

We have demonstrated a composition effect on EO property of LN in NS and Li-deficient regions for the Li-deficient/NS crystals prepared by Li-deficient/Li-rich VTE technique. In the Li-deficient region, both γ_{13} and γ_{33} increase almost linearly by $\sim 8\%$ as the Li_2O -content reduces from 48.6 to 47.0 mol%. The feature of the Li-deficient crystal is propitious to its EO application. In addition, such crystal is also of the merit of higher solubility of Er^{3+} . It is, therefore, a more promising substrate than the congruent material for developing a more efficient active EO device. In the NS regime, both γ_{13} and γ_{33} reveal a non-monotonic dependence with a minimum at the Li_2O -content of 49.5 mol%. Comparison shows that different crystal growth methods cause different defect structure features and hence different composition effects on the EO coefficients of LN.

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