LiNbO$_3$ Optical Waveguide Fabrication by High-Temperature Proton Exchange

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Abstract—An efficient method, the high-temperature proton exchange (HTPE), to fabricate high-quality LiNbO$_3$ optical waveguides is studied. The new proton exchange source, the stearic acid diluted by lithium stearate, is proposed for HTPE process. The known soft proton exchange (SPE) process can be realized by HTPE. There are no phase transitions when the α-phase waveguides are fabricated by SPE. This phase presents the same crystalline structure as that of LiNbO$_3$ and, as expected, maintains the excellent nonlinear and electrooptical properties of the bulk material. The kinetics of HTPE is studied.

Index Terms—LiNbO$_3$, optical waveguides, proton exchange.

I. INTRODUCTION

LITHIUM niobate (LiNbO$_3$) is a very important integrated optical material owing to its electrooptical and nonlinear optical properties. One of the basic methods to make optical waveguides in this crystal is proton exchange (PE). The use of proton exchange was started from work of Jackel *et al.* [1]. They described and fabricated optical waveguides in LiNbO$_3$ by PE in benzoic acid melt at 200–250 °C. The step-like index profiles with a surface refractive index increase of 0.12 for extraordinary polarization have been observed. Since then a lot of organic and inorganic melts used as a source of proton exchange were reported [2].

Despite the simplicity of the proton exchange technique, the PE LiNbO$_3$ waveguides exhibit very complex structural chemistry. Our work [2]–[5] has allowed us to identify seven different crystallographic phases H$_x$Li$_{1-x}$NbO$_3$ which can be realized in the PE LiNbO$_3$ waveguides. There is a phase jump when gradually varying the proton concentration in the exchange layer. This causes a sudden variation of the cell parameters, even if the crystallographic system is conserved. The most interesting for waveguide fabrication is that most of these discontinuities and phase jumps can already be seen on a rather simple diagram, which we called the *structural phase diagram*, where the surface index increase Δn_e is plotted as a function of the strain normal to the substrate surface, ε''_{33}. The β_i and α phases can be fabricated by direct proton exchange or by annealing of

H$_x$Li$_{1-x}$NbO$_3$ phase with higher proton concentration, as following: $\beta_4 \to \beta_3 \to \beta_2 \to \beta_1 \to \alpha$. The κ_i phases cannot be formed by direct PE and are fabricated by postexchange annealing of the β_i phases only. Annealing is an important parameter in the fabrication of PE waveguides. Actually, most of the published results were obtained using heavily annealed PE waveguides (APE [6]), whose index profiles indicate that they are in the α-phase. However, APE process, as was reported recently [7]–[11], can reduce the nonlinear coefficients or even destroy the periodic domain orientation in a portion of the waveguiding structure.

The α-phase waveguides can also be created using a direct proton exchange process without postexchange annealing in a highly diluted melt of benzoic acid which has been called recently as soft proton exchange (SPE) [12]. The use of proton exchange in dilute benzoic acid has been proposed for the first time in [13]. SPE method gives rise to an exchanged layer in the α-phase of better quality than the more popular APE method. The main reason for this is that SPE indicates no phase transitions. This allows preserving both the nonlinear coefficient and the domain orientation while fully benefiting from the power confinement associated with the guided wave configuration [8], [9]. Despite that the α-phase waveguides prepared by APE and SPE methods have similar refractive indexes and strains. However, there is probably a principal difference in basic properties of the α-phase waveguides prepared by these techniques. Indeed, El Hadi *et al.* [14] reported recently that the dispersion of extraordinary refractive-index increase in the α-phase waveguides prepared by SPE method is high compared with that in the APE waveguides [15].

The known SPE processes were performed in benzoic acid diluted by lithium benzoate [8], [9], and [12]. The proton–lithium interdiffusion coefficient for the α-phase is orders of magnitude smaller than that for the β_i phases [16], and one has to use very long processes to make the α-phase single mode waveguides operating at 1.55 μm wavelength (three days at 300 °C [12]). Therefore, it is important to increase the rate of the SPE process. This process rate can be increased by increasing the exchange temperature, however, the low boiling point of benzoic acid (249 °C [17]) and its high vapor pressure limit the temperature that can be used for SPE. What would be more convenient is a source with a higher boiling point and lower vapor pressure. Possible candidates include stearic (boiling point 374 °C [17]), oleic (369 °C), palmetic (360 °C), adipic (338 °C), and cinnamic (300 °C) acids [17]. Fig. 1 shows the dependences of vapor pressure on temperature for these acids [18]. One can see that widely used benzoic acid has the highest vapor pressure and

Fig. 1. Plot of vapor pressure versus temperature for some organic acids used as sources of PE [18].

Fig. 2. Container used during HTPE process.

Fig. 3. Surface value of strain, ε_{33}'', versus composition of melt, ρ.

therefore is most inconvenient for high temperature processing. In this study we used stearic acid, the acid with the highest boiling point (374 °C [17]) and lowest vapor pressure (Fig. 1, [18]) to fabricate LiNbO$_3$ optical waveguides by high-temperature proton exchange (HTPE).

As an alternative, so-called graded proton exchange (GPE) [19] could be also used for HTPE in a sulphate mixture with a relatively low acidity. This can be realized by adding small amount of hydrogen containing salt. However, the relatively high melting temperature for the sulphate mixture makes it inconvenient to use for practical waveguide designs.

In this paper we present the results of fabrication and characterization of LiNbO$_3$ optical waveguides prepared by HTPE process with a new proton exchange source, the mixture of stearic acid and lithium stearate. The pure stearic acid has been used in reports [20]–[25] as a proton source for fabrication of optical waveguides at temperatures from 220 to 270 °C. This acid is nontoxic, noncorrosive and is safe to use. In this paper we focus on high-temperature proton exchange (PE at temperatures higher than 300 °C), as a perspective method of deep α-phase waveguide fabrication.

II. SAMPLE PREPARATION AND CHARACTERIZATION

PE waveguides were fabricated in integrated optical grade virgin X-cuts LiNbO$_3$ substrates supplied by Crystal Technology, Inc. Despite the high boiling point of stearic acid the melt must be sealed, since the melt composition would otherwise change during exchange, due to differential evaporation of the melt constituents. The container design used is shown in Fig. 2. Before exchange the mixture of stearic acid and lithium stearate was melted to become homogeneous.

At temperatures higher than 400 °C the stearic acid is decomposed during the exchange process even when a hermetic container is used. Therefore, to study PE processes at temperatures above 400 °C, a eutectic mixture of sulphate salts with a small amount of KHSO$_4$ added was used as the proton exchange source [19]. The melt consisted of ZnSO$_4$ (41.1 mol.%), K$_2$SO$_4$ (32.9 mol.%), Na$_2$SO$_4$ (25.9 mol.%) and KHSO$_4$ added with concentrations from 0.01 to 0.2 mole per kg of this eutectic. The melting point of this mixture was 384 °C [26].
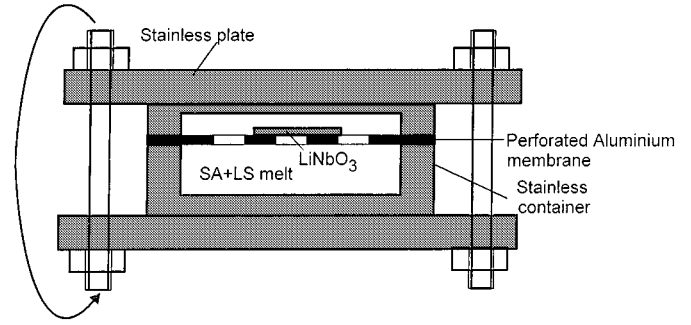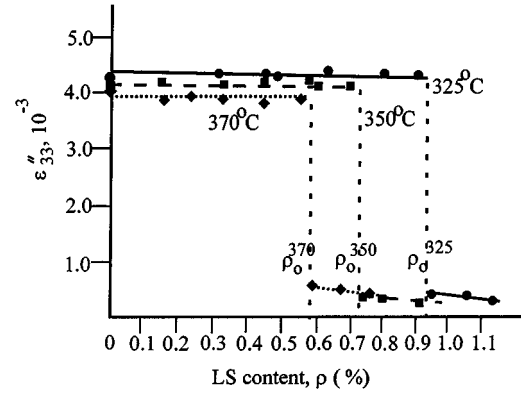
The depth index profiles of the uniform (single phase) waveguides were reconstructed by the IWKB technique proposed by White and Heidrich [27] and improved by Dikaev *et al.* [28]. All the fabricated waveguides were multimode at the 633 nm wavelength used throughout our studies. Therefore, we could determine the effective indexes of guided modes that are necessary for the IWKB technique by measuring the excitation angles of dark m-lines with a one-prism coupler setup. The effective indexes ranged from 2.21 to 2.30. The procedure for obtaining refractive index profiles for two-layer waveguides will be discussed in Section III.

For X-ray characterization, the strained state in the proton-exchanged layers was determined by analyzing the rocking curves obtained by a double-crystal X-ray diffractometer DRON-3 (Cu Kα_1 radiation) in accordance with the method developed in our previous work [29].

III. EXPERIMENTAL RESULTS AND DISCUSSION

The melt used is characterized by the mass ratio of lithium stearate (LS) and stearic acid (SA): $\rho = (m_{\text{LS}}/(m_{\text{SL}} + m_{\text{SA}})) \cdot 100\%$. Varying ρ between 0–1% strongly modifies the phase of the exchanged layer. The surface value of strain, ε_{33}'', has been found to be independent of the exchange time.

Fig. 3 shows the variation of the H$_x$Li$_{1-x}$NbO$_3$ surface value of strain, ε_{33}'', as a function of the LS content and temperature of the melt. Two regions can be clearly recognized. For the PE in the melts with values of ρ lower than the threshold ρ_o, the exchanged structure is then stratified in two phase layers presenting two different indexes and strains. This has been verified
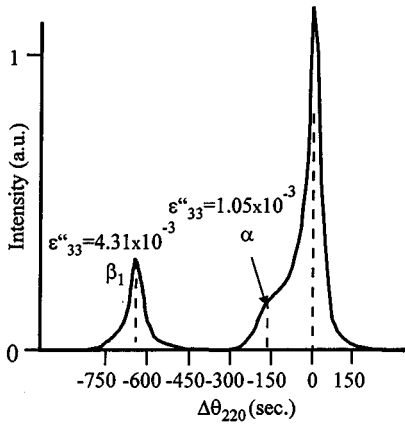
Fig. 4. X-ray rocking curve for (220) reflection from HTPE LiNbO$_3$ sample prepared on X-cut lithium niobate under following conditions: $\rho = 0.4\%$, T= $350\,^{\circ}$C, $t = 8$ h.

by slowly removing the surface layer by successive polishing. The rocking curves show two different peaks, corresponding to the β_1 and α-phases (Fig. 4). The surface value of strains (highest value for the β_1-phase) is found to be largely independent of melt dilution for compositions up to threshold ρ_o (Fig. 3). With ρ higher than threshold ρ_o, which has been determined with a precision of 0.02%, the uniform α-phase waveguides are formed, which present graded index profiles with a maximum index increase $\Delta n_{\alpha}^{o} = 0.02$.

The situation with refractive index measurements for samples prepared under conditions $\rho < \rho_o$ is more complicated. The effective indexes, n_m, of all the modes of planar multimode waveguides can be experimentally determined using the prism coupling technique. Then feeding these data into an Inverse WKB [27], [28] reconstruction program, allows one to calculate the refractive index profile. However, the IWKB method assumes a continuous refractive index profile, but, as has been mentioned above, the real waveguides fabricated by HTPE with $\rho < \rho_o$ are two-layer and standard IWKB method cannot be applied in this case.

Here we describe an algorithm, based on the IWKB method, which has been used to obtain the refractive index profile of two-layer PE planar waveguide.

The peak on the X-ray rocking curve for the β_1 phase is sharp (Fig. 4) and deformation profile in the β_1-phase can be considered as step-like. Therefore, one can assume that the refractive index in the β_1 phase is characterized by a step-like function and the realized waveguide consists of two layers (Fig. 5). Note, that the refractive index gradient for the α-phase region is very small (less than 0.005 μm^{-1}), and the α-phase region can be considered as an uniform substrate with refractive index n_{α}^{o} for the surface step-like waveguide. The dispersion equation for $n_m > n_{\alpha}^{o}$ can be written as [30]

$$kh_{\beta}\left[n_{\beta}^2 - n_m^2\right]^{1/2} = (m-1)\pi + \phi_a + \phi_s \qquad (1)$$

where n_{β} and h_{β} are refractive index and thickness of the step-like β_1-phase waveguide, respectively; n_{α}^{o} the highest refractive index value for the α-phase, n_m is an effective index of the mth mode, $m = 1, 2, \ldots, N$, where N is the number
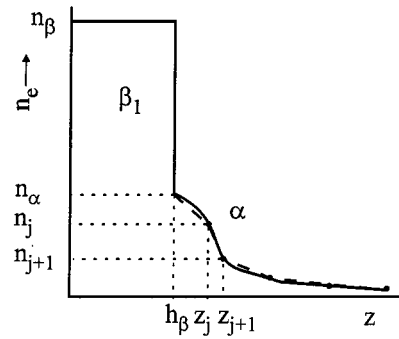

Fig. 5. Refractive index profile for the two-layer HTPE LiNbO$_3$ waveguide.

of modes in the waveguide. The $k = 2\pi/\lambda_0$ is the free-space wavenumber with λ_0 the free-space wavelength. The $2\phi_a$ and $2\phi_s$ are phase changes at the surface and substrate boundaries, respectively and are given by

$$2\phi_a = 2 \tan^{-1}\left\{\xi\left[\frac{n_m^2 - n_a^2}{n_{\beta}^2 - n_m^2}\right]^{1/2}\right\} \qquad (2)$$

and

$$2\phi_s = 2 \tan^{-1}\left\{\xi\left[\frac{n_m^2 - (n_{\alpha}^o)^2}{n_{\beta}^2 - n_m^2}\right]^{1/2}\right\} \qquad (3)$$

where n_a is the refractive index of the superstrate (it was air in our experiments, and $n_a = 1$). The factor ξ discriminates between the transverse electric (TE) and transverse magnetic (TM) modes with $\xi = 1$ for the TE modes and $\xi = (n_{\beta}^2/n_{a,s}^2)$ for the TM modes.

The n_{α}^{o} is known for a given temperature of PE, therefore by solving (1) with measured effective refractive indexes of, at least, two modes with $n_m > n_{\alpha}^{o}$, it is possible to obtain the refractive index n_{β} and thickness h_{β} of the β_1-phase region. The n_{β} is found to be largely independent of melt dilution for compositions up to threshold ρ_o. When n_{β} is known, by solving system (1) with measured effective refractive indexes of even one mode with $n_m > n_{\alpha}^{o}$, it is possible to obtain the thickness, h_{β}, of the thin β_1-phase region.

However, for $n_m < n_{\alpha}^{o}$, the situation is more complicated. For the monotonically decreasing graded-index waveguide, the eigenvalue equation can be written as [28]

$$k \int_0^{z_m} [n^2(z) - n_m^2]^{1/2}\, dz = \left(m - \tfrac{3}{4}\right)\pi + \phi_a$$
$$m = 1, 2, \ldots N \qquad (4)$$

where z_m is the turning point of the mth mode defined by $n(z_m) = n_m$. For the two-layer waveguide with step-like index profile for the β_1-phase and graded profile for the α-phase (Fig. 5), (4) can be written as

$$kh_{\beta}[n_{\beta}(z) - n_m^2]^{1/2} + k \int_{h_{\beta}}^{z_m} [n^2(z) - n_m^2]^{1/2}\, dz$$
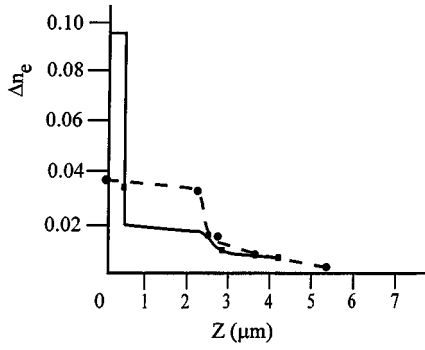$$= \left(m - \tfrac{3}{4}\right)\pi + \phi_a. \qquad (5)$$

Fig. 6. Refractive profiles for HTPE LiNbO$_3$ waveguide fabricated under following conditions: $\rho = 0.70\%$, $T = 350\,^{\circ}\text{C}$, $t = 4.5$ h. Dashed line: IWKB model. Solid line: Two-layer model.

Fig. 7. Surface index change, Δn_e, versus composition fo the melt, ρ, for different temperatures of HTPE.

Fig. 8. Evolution of surface refractive index changes (dots) and strains (rectangles) for samples exchanged in pure stearic acid with increasing of exchange temperature.

According to White and Heidrich [27] with improvements made by Dikaev et al. [28], (5) is written as a sum of integrals

$$kh_\beta[n_\beta(z) - n_m^2]^{1/2} + k\sum_{j=0}^{m}\int_{z_j}^{z_{j+1}}[n^2(z) - n_m^2]^{1/2}\,dz$$
$$= \left(m - \tfrac{3}{4}\right)\pi + \phi_a \quad (6)$$

where $n_l = h_\beta$, for all values of j with $n_l > n_\alpha^o$.

So as the values of n_α^o and n_β are known, and assuming that $n(z)$ is a piece-wise linear function connecting the measured values of n_m [27], i.e.

$$n(z) \approx n_j + \frac{(n_{j-1} - n_j)}{(z_j - z_{j-1})}(z_j - z) \qquad \text{for } z_{j-1} \leq z \leq z_j \quad (7)$$

the turning points of the m-modes for the α-phase region are determined.

Therefore, we have a simple algorithm for calculating h_β and z_m based only on previously determined values of n_α^o, n_β and n_m.

Fig. 6 shows the refractive index profiles calculated using the two-layer model (5) and with the usual IWKB model [27], [28]. One can see that without X-ray double crystal diffraction measurement, that makes sure that the real structure contains two layers, it is easy to be mistaken and assume that waveguide is in the κ_1 phase [3]. However, the κ_1 phase waveguides, formed by APE process, characterized by graded deformation profiles, and X-ray rocking curves show the graded variation of X-ray intensity with changing angle of diffraction around Bragg angle for (220) plane of LiNbO$_3$ [2], [3].

Fig. 7 shows the variation of the H$_x$Li$_{1-x}$NbO$_3$ surface value refractive index increment, Δn_e, as a function of the LS content and temperature of the melt. Similar to Fig. 3, the two regions can be clearly recognized. The Δn_e, is found to be largely independent of melt composition for dilution up to threshold value, ρ_o (Fig. 7). As was shown above, the strain ε_{33}'' is also independent of the content of LS in the melt up to threshold, ρ_o (Fig. 3). Therefore, the exchange ratio, x in H$_x$Li$_{1-x}$NbO$_3$ is also constant for the fabrication regimes, when the β_1-phase is generated at the surface of waveguide (at $\rho < \rho_o$). Fig. 8 shows the ε_{33}'' and Δn_e as a function of the exchange temperature for the β_1-phase. One can see the monotonic decreasing of strains and index change with increasing temperature, which points to the β_1 phase extending into the low concentration region with an
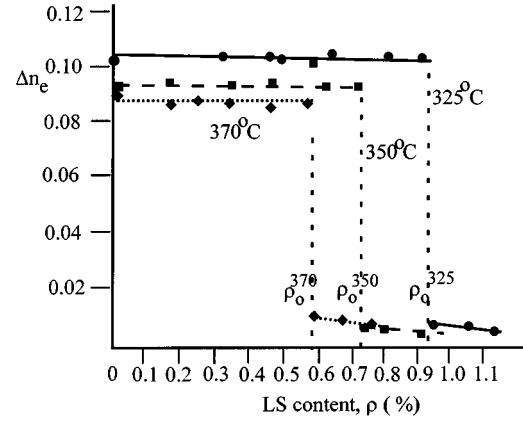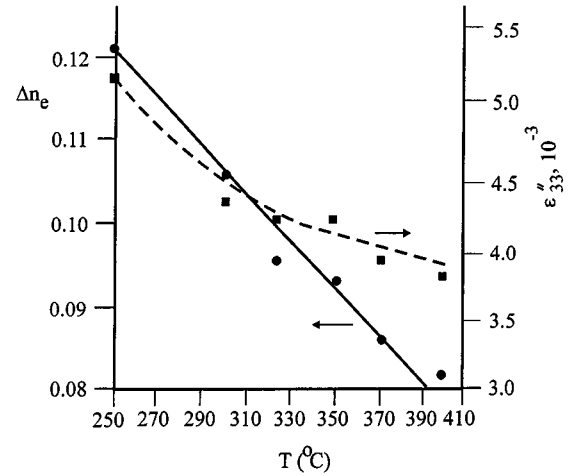
increase in the exchange temperature. The index increment and deformations for the α-phase are very small and the precision of their determination is not accurate enough to make any conclusions about the evolution of boundaries of the α-phase with changing temperature.

The existence of jump on plot of Δn_e and ε_{33}'' versus content of protons is not a specific feature of SA+LS melt and have been observed in other melts with the small hydrogen concentration. Fig. 9 shows that there are jumps on plots of Δn_e and ε_{33}'' versus content of protons in the GPE melt even when the temperature of proton exchange is 420 °C. In the melts with the concentration of KHSO$_4$ higher then threshold, C_H, the two-layer waveguides with the β_1 phase at the surface and the α-phase, as a bottom layer, are formed. With the concentration of KHSO$_4$ lower than threshold C_H, the uniform α-phase waveguides present a graded index profile with a maximum index increase $\Delta n_\alpha^o = 0.02$.

Our experiments confirm the earlier obtained results for low temperature PE [2]–[5], that neither the κ_1 nor the κ_2 phase can be formed by direct proton exchange.

Proton exchange diffusion is a process which follows the heterogeneous proton exchange reaction H$^+$ →Li$^+$ at the surface of LiNbO$_3$. Both the refractive index and deformation profiles

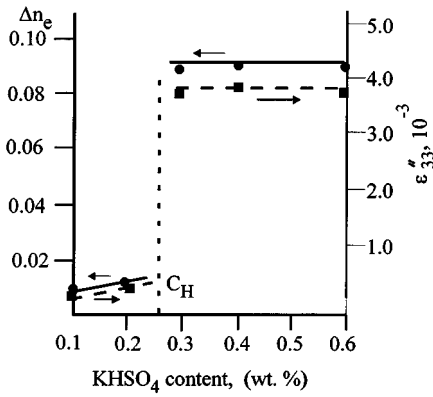Fig. 9. Surface refractive index change (dots) and strains (rectangles) versus content of $KHSO_4$ in the melt $ZnSO_4$–Na_2SO_4–K_2SO_4–$KHSO_4$ for waveguides fabricated by GPE method at the temperature of exchange $T = 420\,°C$.
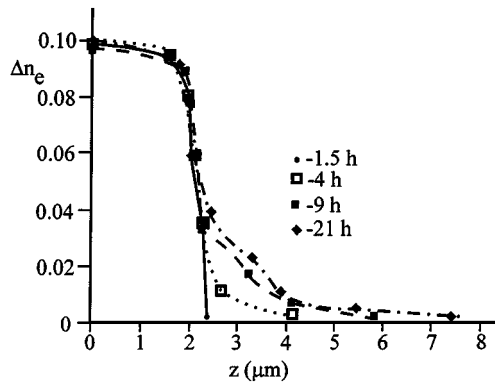

Fig. 10. Evolution of refractive index profile with increasing exchange time for waveguides fabricated by HTPE method in the melt of SA+SL with $\rho = 0.50\%$ at $T = 350\,°C$.

Fig. 11. The depth of the α-phase, $h_\alpha = z_N - h_\beta$, as a function of exchange time t for the two-layer waveguides fabricated at $T = 350\,°C$ in the SA+SL melt with $\rho = 0.50\%$.

Fig. 12. Evolution of the α-phase waveguide, prepared at 350 °C in the SA+SL melt, $\rho = 0.75\%$ with exchange time: (a) refractive index profiles and (b) depth versus square root of exchange time.

for the β_1 region are step-like, and therefore similar to low temperature PE, and so one can expect that the hydrogen concentration profiles are step-like also. The depth of the β_1-phase region in the usual PE process, according to a large number of reports [2], grows proportionally to square root of exchange time, and can be expressed as $h_\beta = \sqrt{2D(T) \cdot t}$, where $D(T)$ is temperature-dependent effective diffusion coefficient and t is the duration of exchange. However, for two-phase structures, realized by HTPE, the situation was determined to be much more complicated. Fig. 10 shows the evolution of refractive index profiles with increasing exchange time. One can see, that the thickness of the β_1-phase, h_β, is not changing during long periods of exchange time. Fig. 10 shows only four representative index profiles, but a large number of experiments has proved the validity of unusual kinetics of this process. However, the thickness of the α-phase region is changing with exchange time. Therefore, flux of protons introduced in $LiNbO_3$ by heterogeneous $H^+ \rightarrow Li^+$ exchange reaction is equal to flux of protons at the β_1/α-phase boundary. Let us define the thickness of the α-phase region as $h_\alpha = z_N - h_\beta$, where z_N is the turning point of last mode. Fig. 11 shows that h_α grows proportionally to square root of exchange time. The explanation of the diffusion kinetics will be reported soon.
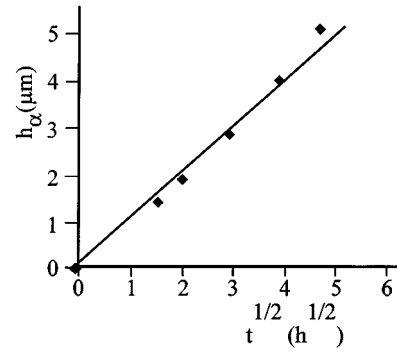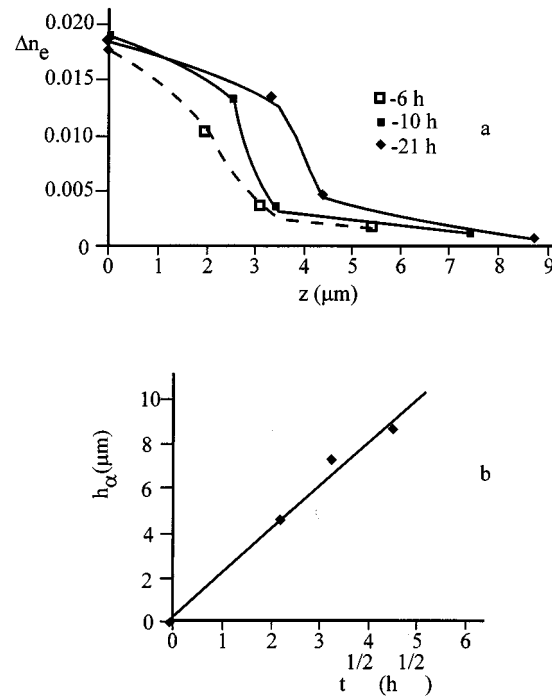
It has been found that for $\rho > \rho_o$, when the uniform α-phase waveguides are formed [Fig. 12(a)], the thickness of the waveguide, defined as a turning point of the last mode, grows proportionally to the square root of the exchange time [Fig. 12(b)].

The uniform α-phase channel waveguides were fabricated by HTPE. Fabrication of single mode channel waveguides was carried out by lift-off photolithographic technique to produce a series of 4-μm (for 850-nm wavelength) and 6-μm (for $\lambda = $ 1550–nm) wide stripes on the sample surface. The pattern was formed by a 50-nm layer of SiO_2. The propagation loss measured by edge coupling method was obtained to be less then 0.2 dB/cm. The 28-mm-long Y-splitters in $LiNbO_3$ were also fabricated by HTPE. By comparing input and total output power the insertion loss was estimated to be less then 5 dB, that points to good confinement of the α-phase channel waveguides.

IV. CONCLUSION

High temperature proton exchange in LiNbO$_3$ has been shown to be quite effective for producing high-quality α-phase waveguides. The new proton exchange source, the stearic acid diluted by lithium stearate, has been proposed for HTPE. The parameters of HTPE process and properties of waveguides fabricated by this way are studied. The α-phase waveguides can be fabricated by direct HTPE in a relatively short period time. There is not any phase transition when the α-phase waveguides are fabricated by direct exchange. This phase presents the same crystalline structure as that of LiNbO$_3$ and, as expected, maintains the excellent nonlinear properties of the bulk material similar to SPE waveguides [8]–[12].

Finally, this possibility of producing high-quality waveguides with relatively short processing times opens the way for mass production of a large variety of integrated optical components.

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