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Research of selective etching in LiNbO₃ using proton-exchanged wet etching technique

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Abstract
Lithium niobate material (LN) has shown great application potentials in the fabrication of integrated optical devices due to its excellent physical properties, especially with the occurrence of lithium niobate-on-insulator (LNOI) substrate. However, the greatest challenge of micro/nano optical devices based on LN material lies in the precise etching process and thus limits its applications. In this paper, we comprehensively analyze the etching results treated by the proposed proton-exchanged wet-etching method (PEWE) combining with theoretical simulations and experiments. It is found that the proton-exchanged layer in the LN material can be easily etched after using a mixture acid of HF/HNO₃, leading to the improvement of etching rate and surface morphology. The lowest roughness of the optical waveguide is measured to be 0.81 nm, which is beneficial for the performance improvement of LN-based optical devices. Ultimately, a quasi-vertical sidewall of the upper part of optical waveguide with improved surface morphology is successfully realized by utilizing the PEWE. Moreover, this method could also be extended to improve the performance of LNOI-based optical devices and pave the way for ultra-compact photonic integrated circuits based on LNOI.

1. Introduction
Lithium niobate (LN), known as the optical silicon in the current optoelectronics age, is one of the most widely-used materials for the fabrication of integrated optical devices because of its commercial availability and multifunctional properties [1], such as the excellent transparency from visible to infrared light, high nonlinearity and large electro-optical coefficient [2]. During the past decades, LN material has drawn great attentions in realizing high-density integrated photonic/optical circuits due to its high performance [3], especially with the occurrence of a newly-arisen optical material, namely, lithium niobate-on-insulator (LNOI). However, the LN material is chemically inert, as a result, it can hardly be etched without any pre-treatments. In addition, mechanical processing for the LN material is also difficult to proceed due to its high hardness and wear resistance. With continuous improvements of fabrication techniques, specifically for the LN-based micro- or nanostructure fabrication, a variety of fabrication processes to achieve optical waveguides with strong optical confinement have been investigated. Two most widely-used diffusion-based approaches to manufacture waveguides have been well-established in bulk LN material, i.e. the titanium thermal diffusion and the proton exchange (PE) method. However, the main disadvantage of the waveguides based on these two methods is that the refractive index contrast in the in-plane dimension is highly limited, so it is difficult to guarantee the small volume and low optical loss simultaneously. What’s more, it is incompatible with the common CMOS process. For LNOI platform, high difference of refractive index contrast of the fabricated channel waveguides is obtained due to its unique structure, leading to a better optical confinement in a much smaller volume. To realize the ultra-compact integrated photonic circuits, several kinds of etching methods have been reported to fabricate high refractive index difference waveguides based on the LN material, such as reactive ion beam etching (RIE),
inductively coupled plasma etching (ICP-RIE), focused ion milling (FIB), and wet etching and so on [3–5]. Nevertheless, some inevitable problems exist in these etching methods. For instance, RIE process is obviously anisotropic, ICP-RIE produces LiF deterioration on the surface resulting in high scattering loss, FIB and wet etching are both time-costing for practical application. And as far as we are concerned, LN material after treated by these etching methods faces the problems of high loss [6–8], shallow sidewall angle [6, 9–11], limited etch rate [12–15], or a combination thereof. In 1922, Laurell et al [16] proposed a new wet-etching method by using PE and a mixture of HF/HNO₃ acids for LiNbO₃ to overcome the drawbacks caused by a dry-etching process, and finally obtained a ridge waveguide with smooth etching surface. During the past 20 years, LN-based ridge waveguides and related optical devices fabricated by this method have been successfully developed [17–22]. Wang et al [23] used electric field-assisted proton exchange in the lateral directions and thus not only created a vertical sidewall near the substrate surface with smooth surface, but also effectively increased the vertical sidewall depth. Ting et al [24] utilized an improved wet-etching method by using mixed benzoic and adipic acids as the source of PE to improve the aspect ratio. Lai et al [25] presented a novel wet-etching method to fabricate a LN-based ridge structure with the assistance of gamma-ray irradiation. However, the main point of these studies only focuses on the improvements of the fabrication method itself to obtain better etched profiles and etched morphologies, the detailed etching mechanisms including proton exchanged process, phase transition and the resulted morphology changes are still remained to be investigated intensively.

In this paper, we comprehensively analyzed the efficient proton-exchanged wet-etching (PEWE) method combining with the theoretical simulations and experiments based on LN material. The variation of concentration of H⁻ ion, the crystal micro structure and surface roughness for the waveguide are studied. The relationship between the phase structures and surface chemical-physical properties was also investigated. In contrast to the LN material without proton-exchange process, PEWE method could effectively increase the etching rates by nearly 100 times, and a much smoother surface with the roughness as low as 0.81 nm is achieved. Meanwhile, a quasi-vertical sidewall of ~90 degree of the upper part of optical waveguide is also observed. To the best of our knowledge, this is also the minimum roughness ever reported at the corresponding etching depth with a sharp upper side of etching sidewall. Additionally, this proposed PEWE method could be applied to fabricate the LNOI-based optical devices and enhance the foundations for ultra-compact photonic integrated circuits based on LNOI.

2. Experiment

Optical waveguides were fabricated in commercial optical grade 500-μm-thick Z-cut LN substrate (4-inch, NANOLN). The fabrication process of PEWE method is depicted in figure 1:

(1) Cr mask fabrication: After LN substrate was cleaned by DI water, a Cr film with 100-nm thickness was deposited on the surface of the positive z facet. Subsequently, standard photolithography was performed on the Cr mask using spin-coated AZ5214 photo-resist to form 6-μm-wide waveguide patterns. The Cr mask was then wetting-etched using a mixture solution of ammonium ceric nitrate (NH₄)₂Ce(NO₃)₆ and nitric acid (HNO₃), the remained photoresist structure was removed by acetone along with ultrasonic process. Finally, the 4-inch LN substrate was divided into four identical samples.

(2) Proton exchange: 6-μm-wide Cr stripes were used as the mask to prevent proton from penetrating into the substrate. Then three samples (#1~#3) were immersed in the melt of benzoic acid in a sealed furnace at

![Figure 1. Fabrication process for PEWE method: Cr deposition (a); photoresist spin-coating (b); photolithography (c); Cr corrosion (d); photoresist removal (e); proton etching (f); wet etching (g); Cr removal (h).](image-url)
240 °C for different PE time (benzoic acid, a solid at room temperature, is melted in a quartz glass tube at normal pressure. The samples were placed in the melt for the desired time, then removed and allowed to cool.). Detailed experiment parameters are listed in table 1. During the exchange reaction, H+ ions could diffuse into the LN material for a certain depth to replace the Li+, leading to the formation of HxLi1-xNbO3, then new crystal phases were produced.

(3) Wet etching: After the PE process, samples #1∼#4 were cleaned again and then immersed in the mixture solution of nitric acid (a concentration of 68% HNO3) and hydrofluoric acid (a concentration of 40% HF) at room temperature to complete the etching process. To be noted, the Cr stripe mask is inert to the mixture acid.

(4) Polishing and removal of Cr mask: The four samples after etching process were cut along the vertical direction of the optical waveguide propagation, and the formed waveguide end faces were then carefully polished using SiO2 suspension to facilitate the following SEM measurement. Subsequently, the Cr films were removed using a mixture solution of (NH4)2 Ce(NO3)6 and HNO3 again. Sample #4 without PE process was used as the reference.

The etching depth is measured by a surface profiler (Veeco, Dektak 150). And COMSOL Multiphysics software is employed for the numerical simulation of the transient dynamic analysis of PE process. The phase structure of proton-exchanged LiNbO3 is characterized by high-resolution x-ray diffraction (HRXRD) (Bruker, D8 Advance). The etched profiles are measured using a scanning electron microscope (SEM, ZEISS, Gemini 300). And the etched surface morphology was obtained by atomic force microscope (AFM, WITec).

3. Results and discussion

3.1. Direct proton exchange

First and for most, it is quite necessary to figure out the precise PE process in order to understand the proposed PEWE method. It mainly involves in-diffusion process of protons ions (H+) from a proton source and out-diffusion process of interstitial ions (Li+) from the LN crystal lattice [26], the whole process can be summarized by the following chemical reaction [27, 28]:

\[
\text{LiNbO}_3 + iH^+ \leftrightarrow H_x\text{Li}_{1-x}\text{NbO}_3 + i\text{Li}^+
\]

Due to the electro-neutrality, the diffusion velocity would meet a dynamic equilibrium finally. Since the PE process is time-dependent, the continuity equation for two-dimensional diffusion of H+ is shown as follow [26]:

\[
\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} \left( D(C) \cdot \frac{\partial C}{\partial x} \right) + \frac{\partial}{\partial y} \left( D(C) \cdot \frac{\partial C}{\partial y} \right)
\]  

\[\text{(1)}\]

In the model of inter-diffusion of H+ and Li+, C represents the concentration, D(C) is the diffusion coefficient, i.e.:

\[
D(C) = \frac{D_{H^+}}{1 - \alpha C}
\]

\[
C = C_{H^+}/C_0
\]  

\[\text{(2)}\]

where,

\[
\alpha = 1 - \frac{D_{H^+}}{D_{Li^+}}
\]  

\[\text{(3)}\]

\[D_{H^+}\text{ and } D_{Li^+}\text{ are the diffusion coefficients of } H^+\text{ and } Li^+, \text{ respectively. At the beginning of the PE process, } D_{Li^+}\text{ is a thousand times higher than } D_{H^+}. \text{ And } D_{H^+}\text{ is defined as below:} \]

| Samples | PE source | PE temperature/°C | PE time/h (T) |
|---------|-----------|------------------|---------------|
| #1      | benzoic acid | 240 °C | 1              |
| #2      | benzoic acid | 240 °C | 3              |
| #3      | benzoic acid | 240 °C | 4              |
| #4      | benzoic acid | 240 °C | 0              |
where, $R$ is the gas constant, $T$ is the absolute temperature, $D_0$ is the diffusion constant of the ion, $Q$ is activation energy. The depth of the diffusion profiles, as a function of the exchange time and the temperature, obey the standard square root law:

$$d = 2\sqrt{D_H^+ t}$$

Meanwhile, the relationship between refractive index and concentration can be expressed as below [26, 29, 30]:

$$n(C) = n + \Delta n(\lambda)(1 - e^{-kC})$$

Then, the transient dynamic analysis of PE process is conducted using COMSOL Multiphysics. The exchange temperature is set to be 240 °C. And the simulated results are shown in figure 2. It is found that during the PE process protons move not only perpendicularly into the LN material, but also laterally to both sides. And as the exchange depth increases, the proton concentration decreases. The proton concentration at the boundary is calculated to be 20% lower than that in the middle region.

### 3.2. Evaluation of the etching rate

As shown in figure 3(a), a quasi-linear relationship between the etching depth and the etching time is achieved in sample (#1∼#3). However, the pure LN (sample #4) is almost not dissolved in the acid mixture at room temperature. The etching rates are also calculated and plotted in figure 3(b). On one hand, as can be seen, the etching rates for sample #1∼#3 contain two periods. In the beginning 50 min, etching rates are practically independent on etching time. And then the etching rates for the three samples decrease with increasing etching time. On the other hand, with the increasing of PE time, the etching rate is also slightly increase. As is known, the etching rate is closely related to the proton concentration and phase character in LN material. According to simulated proton concentration shown in figure 2(c), proton concentration is approximately the same at the very beginning, and then decreases rapidly. Therefore, the proton concentrations near the surface of sample #1∼#3 are considered to be nearly the same, thus, leading to the same etching depth and same etching rate as demonstrated in figures 3(a) and (b), respectively. And longer PE duration could also result in higher proton concentration at the same position in LN material. That is the main reason that the highest etching rate is obtained in sample #3. And the etching rate ranges from 7.66 to 8.89 nm min$^{-1}$ as PE duration varying from 1 h.

![Figure 2. Proton concentration distribution simulated by COMSOL (a); Normalized proton concentration along the horizontal line (i-v) (b); Normalized proton concentration of vertical line (I) as a function of PE times (c); Normalized proton concentration of vertical line (II) as a function of PE times (d).](image-url)
to 4 h. Compared with reference sample #4, of which the average etching rate is only 0.07 nm min$^{-1}$, the etching rate after PEWE process increases nearly by 100 times, which facilitates the etching process significantly.

For comparison, the standard deviation and the relative standard deviation of the etching rate for all samples are calculated and listed in table 2. The maximum relative standard deviation of etching rate is just 10.8% when using the PEWE method, indicating relative stability of the etching rate after the PE process, while sample #4 without PE process shows an obviously unstable etching rate.

### 3.3. Structural properties of the proton-exchanged LiNbO$_3$

HRXRD measurement is conducted to evaluate the phase transition in the LN material after the PE process. According to the XRD data shown in figure 4, clear satellite peaks for sample #1~#3 are detected on the left of the main peak of the LN substrate, meaning the interspace of LN (00.12) crystal face of the proton exchanged layer is different from that of the substrate, that is phase transition. It is mainly caused by the increase of the lattice constant and lattice distortion. And as the PE duration increases, the intensity of the satellite peak increases as well, which is result from the increased thickness of proton-exchanged layer in LN material. According to the previous reports [31, 32], the newly-occurred phase are $\beta_1$ phase, $\beta_1 \& \beta_2$ phase, $\beta_1 \& \beta_3$ phase in figures 4(b)–(d), respectively. From the figure 4(b), only the $\beta_1$ phase is presented, which means the exchanged layer is homogeneous [31]. With the increase of PE duration, the proton concentration gets higher, phase situation of the proton-exchanged layer is getting more and more complicated. In figures 4(c) and (d), the $\beta_2$ phase and $\beta_3$ phase occur accompanied by the $\beta_1$ phase, implying the existence of transition layers between the LN substrate and the top homogenous layer with $\beta_1$ phase. And with the proton-exchanged layer stratified in two single-phase layers, severe strain situation would be produced, leading to the introduction of more structural defects. As a result, the proton-exchanged layer could be etched much easier than the pure LN material. Therefore, this is another reason for the increase of etching rate.

Figures 5(a)–(c) show the end face SEM morphologies of the obtained optical waveguides of sample #1~#3. Since sample #4 has a rather slow etching rate, no obvious changes of the end face are observed. Since the main character of the wet etching is its isotropy, obviously slope outline of the optical waveguide is obtained, except the region near surface as circled in red dotted box, which is quasi-vertical. It is considered to be caused by the domain inversion and the structural defects induced by the accumulation of Li$^+$ on the boundary of the proton-exchanged region. The total etched depth D, the vertical sidewall depth $D_0$ and the horizontal distance of the slant H are measured and listed in table 3. As expected, with the increase of PE duration, D is slightly increased from 3.25 $\mu$m to 3.75 $\mu$m, while H is increased drastically from 2.66 $\mu$m to 3.42 $\mu$m. To evaluate the degree of

| Samples | Average value (nm min$^{-1}$) | Standard deviation | Relative standard deviation |
|---------|-------------------------------|--------------------|----------------------------|
| #1      | 7.66                          | 0.83               | 10.8%                      |
| #2      | 8.53                          | 0.58               | 6.8%                       |
| #3      | 8.89                          | 0.44               | 4.9%                       |
| #4      | 0.07                          | 0.02               | 28%                        |
verticality quantitatively, the aspect ratio of the optical waveguide is defined as $D$ divided by $H$. The better verticality the waveguide has, the larger the aspect ratio will be $[24, 25, 33]$. As listed in table 3, the aspect ratio decreases from 1.22 to 1.09 due to the slightly increase of lateral diffusion of $H^+$ ions, therefore the etched

Figure 4. HRXRD $(00.12)\omega/2\theta$ scanning results for: sample #4 w/o PE (a), sample #1 after PE 1 h (b), sample #2 after PE 3 h (c), sample #3 after PE 4 h (d).

Figure 5. SEM results of etched profiles for sample #1 (a), sample #2 (b), sample #3 (c) and the corresponding AFM images of the etched surfaces for sample #1 (d), sample #2 (e), sample #3 (f). Inside the red dotted box is the vertical part of the optical waveguide.

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profiles in figure 5 are quite similar. The vertical part of etching sidewall is much sharp and $D_0$ slowly increases from 0.85 $\mu$m to 1.13 $\mu$m.

The roughness of the etched substrate is measured by AFM in the area of $3 \times 3 \mu$m as illustrated in figures 5(d)–(f). After using PEWE method, the RMS roughnesses of the etched surface for three samples are measured to be 0.81, 1.07, and 3.85 nm, respectively. It is found that the lowest RMS roughness, 0.81 nm, belongs to the sample #1, which is properly caused by the homogeneity of the proton-exchanged layer. And with longer PE duration, the roughnesses of sample #2 and #3 are increased. However, their roughnesses are still far less than one-tenth of the light wavelength (for example, even for blue-light optical waveguide, the wavelength is longer than 400 nm). Therefore, it is considered that the surfaces are optically smooth [23, 24]. Finally, it is highly suggested that in order to obtain the target etching depth while maintaining the smooth morphology, the PE duration needs to be optimized to achieve an optical waveguide with smaller scattering loss.

4. Conclusions

In conclusion, an efficient method combining the PE process with wet etching to fabricate high-quality optical waveguides in LN substrate has been comprehensively demonstrated. Due to the proton exchange reaction, $H^+$ ions diffuse into the LN substrate to substitute the $Li^+$ ions, leading to the occurrence of phase transition and structural defects. And proton concentration and the newly-occurred defects could both influence the etching rate. According to the SEM and AFM results, it is demonstrated that the etched surface is optically smooth and the sidewall near the surface of the waveguide is quasi-vertical. However, there is a trade-off between the etching rate and the roughness of etched surface. Moreover, the proposed PEWE method can be used to fabricate ultra-compact integrated waveguide devices with small loss on LNOI platform in the future.

Disclosures

The authors declare no conflicts of interest.

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Author Contributions

Y L, T L, C L and D Y conceived and designed the experiments; Y L, M X and J D performed the experiments; Y L, T L and Z W analyzed the data; Z W and D Y provided financial support; Z W and C L provided experimental sites and computing resources; Y L completed COMSOL calculation and wrote the paper.

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| Sample | D ($\mu$m) | H ($\mu$m) | $D_0$ ($\mu$m) | Aspect ratio (D/H) | RMS (nm) |
|--------|-----------|-----------|---------------|-------------------|----------|
| #1     | 3.25      | 2.66      | 0.85          | 1.22              | 0.81     |
| #2     | 3.5       | 2.91      | 0.96          | 1.20              | 1.07     |
| #3     | 3.75      | 3.42      | 1.13          | 1.09              | 3.85     |
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