# Etching of Lithium Niobate: From Ridge Waveguides to Photonic Crystal Structures

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Abstract. Recent progress of wet etching of Z-cut LN, of inductively coupled plasma (ICP-) etching of X-cut LN, and of ICP-etching of proton-exchanged X-cut LN is reported to fabricate low loss ridge guides, micromechanical, and photonic crystal structures.

# Introduction

The development of lithium niobate (LiNbO<sub>3</sub>, LN) integrated optical devices requires etching techniques for a reliable fabrication of deep (sub-) micrometer structures. Examples are ridge guides, Bragg gratings, and photonic crystal structures. The existing etching methods can be classified into two categories: wet (chemical) etching and dry (ion) etching. Wet etching is generally performed in a mixture of HF and HNO<sub>3</sub>, which attacks the -Z-face of the crystal, whereas the +Z-face is hardly affected. Therefore, selective chemical etching can either be achieved by depositing a metallic mask of the structure to be fabricated on the -Z-face [1] or by defining the structure first by a corresponding domain inversion [2]. Wet etching can also be applied to proton-exchanged or ion-implanted LN to form (sub-) micrometer structures in the surface of the crystal [3, 4]. In dry etching, plasma etching, ion beam milling and focussed ion beam etching are generally used [5, 6, 7]. In this contribution we report our recent progress of wet etching of Z-cut LN to fabricate low loss ridge guide and micromechanical structures, of inductively coupled plasma (ICP-) etching of X-cut LN to get ridges and other microstructures, and of ICP-etching of proton-exchanged X-cut LN to develop photonic crystal structures.

#### Wet etching of Z-cut LN: ridge waveguides and micromechanics

Wet etching of Z-cut LN with HF/HNO<sub>3</sub> proved to be a simple and reliable method to fabricate low loss Ti-doped ridge waveguides with TE propagation losses of 0.3 dB/cm only [4]. To get optical guiding, the ridges were defined in a planar Ti:LiNbO<sub>3</sub> waveguide. Cr-stripes have been used as etch masks.

Here we report a modified procedure yielding ridge guides of propagation losses nearly one order of magnitude lower (TE). They were fabricated in three steps using undoped (congruent) Z-cut LN as substrate: *1. Ridge fabrication*: Using a Cr-mask on the –Z surface of the LN substrate 4 to 12  $\mu$ m wide ridges were fabricated by wet chemical etching in a mixture of 21 ml HF (40% concentration), 14 ml HNO<sub>3</sub> (100% concentration) and 5 ml ethanol, following the procedure described in [1]. A ridge of 6.5  $\mu$ m (10  $\mu$ m) height (top width) is shown in Fig. 1 (left); it is aligned parallel to the X-axis. 2. *Tistripe definition:* A novel photolithographic process was developed, which allows coating the surface of a ridge selectively. Spin-coating of the sample with photo-resist results in an inhomogeneous thickness distribution with a thinner layer on top of the ridge. This is exploited by the following flood exposure (5 seconds), which leads to full exposure only on top of the ridge. By the subsequent development the photo-resist on the top of the ridge is totally removed, while a thin layer remains besides the ridge and on the ridge walls. After lift-off a Ti-stripe is precisely defined on top of the ridge only (see Fig. 1-middle). 3. *Ti-indiffusion:* The Ti-stripe can now be indiffused using conventional parameters (1060 °C @ 8.5 hrs in Ar (7.5 hrs) and O<sub>2</sub> (1 hr) atmosphere, respectively). The result is a Ti-doped ridge waveguide as shown in Fig. 1 (right); the edges are rounded and the surface roughness is reduced (see Fig. 1-right).



*Fig. 1:* Wet etched ridge on Z-cut LN, (left), Ti-coated ridge before indiffusion (middle), and after indiffusion (right). The height (top width) of the ridge is  $6.5 \,\mu m (10 \,\mu m)$ , aligned parallel X.

Therefore, also the propagation losses of the ridge guides, monomode up to a top width of 9  $\mu$ m, are significantly lower than previous results [1]. They were measured using the Fabry-Perot resonance method at 1.55  $\mu$ m wavelength. Both, the TE and TM losses decrease with increasing width. For TE-polarization the loss drops from 0.22 dB/cm at 5  $\mu$ m width to 0.05 dB/cm at 7  $\mu$ m width. For TM-polarization the losses are 1.3 and 0.36 dB/cm at 5  $\mu$ m and 9  $\mu$ m widths, respectively. They are significantly larger than TE-losses, though the TM mode is smaller than the TE-mode with lower field strength at the ridge walls. An explanation for the strongly polarization-dependent losses might be that due to the growth of Cr<sub>2</sub>O<sub>3</sub> under the mask and due to outdiffusion of Li<sub>2</sub>O the concentration of Li and O might vary near the surface leading to corresponding fluctuations of the extraordinary index of refraction (the ordinary index would remain nearly unaffected). Such index fluctuation would lead to scattering losses of the TM-mode alone.

It was even possible to control the slope of the ridge walls by adjusting the etching temperature. This allows getting even steeper walls and to fabricate micromechanical structures such as the interdigital fingers given as example in Fig. 2. It was etched at a temperature of 8 °C. The lamellas have a width of 2.6  $\mu$ m with a separation of 2.8  $\mu$ m. The etching depth is 7.7  $\mu$ m.



*Fig. 2:* Wet etched interdigital lamellas of 2.6  $\mu$ m width and of 2.8  $\mu$ m separation on Z-cut LN. The etching depth is 7.7  $\mu$ m.

#### **ICP-etching of X-cut LN: ridges and microstructures**

Plasma etching is a very controllable process and has the advantage of being highly anisotropic. Plasmas based on fluorine gases are generally used for plasma etching of LN due to the good volatility of fully fluorinated niobium species at temperatures around 200 °C. However, a problem is the formation and re-deposition of LiF, which has a melting temperature of more than 800 °C. It will be deposited on all surfaces and will lower in this way the etching rate. Therefore, if re-deposition dominates the process, vertical side walls, which are necessary for the definition of small structures, can hardly be obtained. Our solution for this problem is as follows: at first, the sample with a Cr layer defining the structure to be fabricated is ICP-etched for several minutes in a  $C_4F_8/He$  (1:1) plasma. Then the etching process is stopped and the sample is cleaned in SC-1 solution (70% H2O, 20% H2O2, 10% NH4OH) for 1 minute to remove the deposition, before ICP-etching is continued. These two steps are repeated several times until the desired etching depth is reached. Fig. 3 shows as an example etched ridges in X-cut LN, aligned along the Y-direction; the ridges have a height (width) of 5.8 µm (8 µm). On the right of Fig. 3 more complicated microstructures of the same height are shown demonstrating that even small connections between the squares can be fabricated in a reproducible way with nearly vertical walls.



*Fig. 3:* ICP-etched ridges on X-cut LN of 5.8 µm height and 8 µm width (left and middle). Further microstructures of the same height with nearly vertical side walls (right).

# ICP-etching of H<sup>+</sup>-exchanged X-cut LN: photonic crystal structures

Another way to reduce the problem of LiF re-deposition is to lower the Li concentration in LN by a proton exchange (PE) process [8]. Thus, the rate of LiF re-deposition will be significantly reduced in comparison to etching of pure LN. As a consequence, the etching rate will be increased and the etch profiles will be improved. We demonstrated the realization of this concept be performing first a PE of congruent X-cut LN to a depth of 1.4  $\mu$ m. Then the surface of the PE-LN was covered by a 110 nm thick Cr film deposited by sputtering and coated with a 120 nm thick photo-resist layer. Using conventional optical contact lithography, photonic crystal structures were defined in the



*Fig. 4:* Holes of a photonic crystal structure defined in photo resist (left), transferred by wet etching in the Cr film (middle) and by ICP-etching into the LN substrate.

photo-resist (Fig. 4, left). The smallest lines have a width of 170 nm. By wet etching these structures were transferred into the Cr film (Fig. 4, middle). Then the sample was ICP-etched for several minutes, and cleaned afterwards in SC-1 solution to remove all depositions. This process was repeated several times until the desired etching depth in the PE-LN was reached (Fig. 4, right). Fig. 5 shows the photonic crystal structure as a whole together with some details in higher magnification.



*Fig. 5:* Photonic crystal structure in PE-LN as a whole (left) and with more details in higher magnification (middle and right).

Proton exchange is also used for the fabrication of single polarization waveguides, as it increases only the extraordinary index of refraction. A subsequent reverse proton exchange (RPE) can even increase the Li concentration again to form buried waveguide profiles.

### Conclusions

In conclusion, significant progress has been achieved to improve wet etching of Zcut LN, ICP-etching of X-cut LN, and ICP-etching of proton-exchanged X-cut LN allowing a reliable fabrication of integrated optical devices with deep (sub-) micrometer structures. As examples ridge guides of very low propagation losses, micromechanical, and photonic crystal structures have been demonstrated.

# References

- [1] H. Hu, R. Ricken, W. Sohler, and R. B. Wehrspohn, "Lithium Niobate Ridge Waveguides Fabricated by Wet Etching", IEEE Photon. Technol. Lett., vol. 19, pp. 417–419, Mar. 2007.
- [2] I. E. Barry, G. W. Ross, P. G. R. Smith, and R. W. Eason, "Ridge waveguides in lithium niobate fabricated by differential etching following spatially selective domain inversion," Appl. Phys. Lett., vol. 74, pp. 1487–1488, Mar. 1999.
- [3] T.-L. Ting, L. -Y. Chen, and W.-S. Wang, "A novel wet-etching method using joint proton source in LiNbO<sub>3</sub>", IEEE Photon. Technol. Lett., vol. 18, pp. 568–570, Feb. 2006.
- [4] D. M. Gill, D. Jacobson, C. S. White, Y. Shi, W. J. Minford, and A. Harris, "Ridged LiNbO<sub>3</sub> modulators fabricated by a novel oxygen-ion implant/wet-etch technique", J. Lightwave Technol. vol. 22, pp. 887–894, Mar. 2004.
- [5] Masashi Tamura, Shinzo Yoshikado, "Etching characteristics of LiNbO<sub>3</sub> crystal by fluorine gas plasma reactive ion etching", Surface and Coatings Technology, Vol. 169–170, pp. 203–207, 2003.
- [6] P. Rabiei and W. H. Steier, "Lithium niobate ridge waveguides and modulators fabricated using smart guide," Appl. Phys. Lett., vol. 86, Art. No. 161115, 2005.
- [7] F. Lacour, N. Courjal, M.-P. Bernal, A. Sabac, C. Bainier, M. Spajer, "Nanostructuring lithium niobate substrates by focused ion beam milling", Optical Materials, Vol. 27, pp. 1421–1425, 2005.
- [8] H. Hu, A. P. Milenin, R. B. Wehrspohn, H. Herrmann, and W. Sohler, "Plasma etching of protonexchanged lithium niobate", J. Vac. Sci. Technol. A, vol. 24, pp. 1012-1015, Jul./Aug., 2006