A Novel Wet-Etching Method Using Joint Proton Source in $LiNbO_3$

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***Abstract*—By mixing benzoic and adipic acids as the source of proton exchange, wet etched ridge waveguides are fabricated in Z-cut $LiNbO_3$. Under appropriate adipic–benzoic acid concentration ratios, the joint proton source can suppress the proton diffusion in the lateral directions, and thus, the sidewalls are much more vertical than the usual. The etched depths are also enhanced for more than 30%. And meanwhile, the fabricated ridge waveguides possess smooth surfaces and the scattering losses are lower than that of the conventionally produced samples.**

***Index Terms*—Integrated optics, lithium niobate, optical ridge waveguide, proton exchange (PE).**

I. Introduction

RIDGE waveguides in $LiNbO_3$ are of great interests for many researchers on the realm of integrated optics. Either for better high-frequency operation of optical modulator [1] or for lower driving voltages of Mach–Zehnder modulators [2], [3] and polarization converters [7], a ridge waveguide is able to provide extensive help. In recent years, to increase the density of optical integrated circuits, a lot of effort is focused on various waveguide structures, such as waveguide mirrors [4], T-junction [5], and photonic crystals [6]. All these structures require materials of high-contrast refractive indexes and more importantly mature etching techniques to fabricate waveguides with smooth surfaces and vertical sidewalls. The silicon or III–V-based processes can easily meet the requirements. However, it is much more difficult to produce such devices in lithium niobate. This is owing to the limitation of the conventional etching techniques. To fabricate the ridge waveguide in lithium niobate, dry etching techniques, such as plasma etching [9], sputter etching [10], or reactive ion etching [11] were used. These conventional dry etching methods were facing problems of slow etching rates and rough etched surfaces. In 1992, Laurell *et al.* [12] proposed a wet etching method for lithium niobate. They showed that the proton-exchanged part of the $LiNbO_3$ substrate can be easily etched by a mixture of HF and HNO_3 acids. By utilizing this method, ridge waveguides with a several-micron etched depth and a smooth surface could be obtained. However, due to the lateral diffusion of protons during the proton exchange (PE) process, the etched sidewalls had sloping or curved shapes. It is a fatal drawback for the increase of integration density. Recently, Wang *et al.* [8] have used electric-field-assisted PE to solve the vertical issue. By applying a static electric field during PE, the etched depths and the shapes of sidewalls were proven to be better than those of the conventional wet etching method. However, a relatively complex process is the price paid for the improvement.

The proton-exchange process is not only used to fabricate ridge waveguides. As a matter of fact, it is more well-known for the fabrication of high-index low loss waveguides [13]. A mixture of adipic and benzoic acids has already been used as a proton source to form optical waveguides [14]. However, it has not been used in a wet etching process for lithium niobate. In this letter, it is demonstrated that by utilizing the mixed benzoic and adipic acids as the PE source, the final etched ridge waveguides possess better shapes of sidewalls and larger ridge heights. The etched surfaces are very smooth and the scattering losses are small. And notably, the complexity of the fabrication process is hardly increased in comparison with the conventional method.

II. Experimental Results and Discussion

A. Ridge Waveguide Fabrication

Ridge waveguides are fabricated in optical grade Z-cut $LiNbO_3$ substrates. The fabrication process is as follows. First, by photolithography, the pattern is defined on the $LiNbO_3$ substrate. Then, 200-nm tantalum films are sputtered to the substrate as the mask of PE to stop protons from penetrating the substrate. After lifting Ta off the substrate, the sample is ready for PE. The proton sources are mixed by benzoic acid and different molar percentages of adipic acid. The concentrations of adipic acid are ranged from 0 to 50 mol% with an interval of 10 mol%. At 235 °C, both acids are melted in the beaker, and the sample can be completely immersed. The PE process lasted in air for 8 h. Then the sample was cooled naturally to room temperature and cleaned by ultrasonic bath in methanol to remove the residual acid. Because the protons penetrating the substrate will induce structural defects in $LiNbO_3$, the $1HF:2HNO_3$ solution can easily and uniformly etch the proton-exchanged region while hardly etch the rest of the bulk. By immersing the sample into the etchant for 6.5 h, the ridge structures with a width of 10 μm can be produced. However, only ridge structures are not enough for waveguiding. Through another photolithography and liftoff process, the titanium strips are sputtered onto the ridge surface. The width and the thickness of the Ti-strip are 7 μm and 80 nm, respectively. At 1020 °C, titanium is diffused into substrate for 280 min, and then the ridge waveguides are successfully fabricated.

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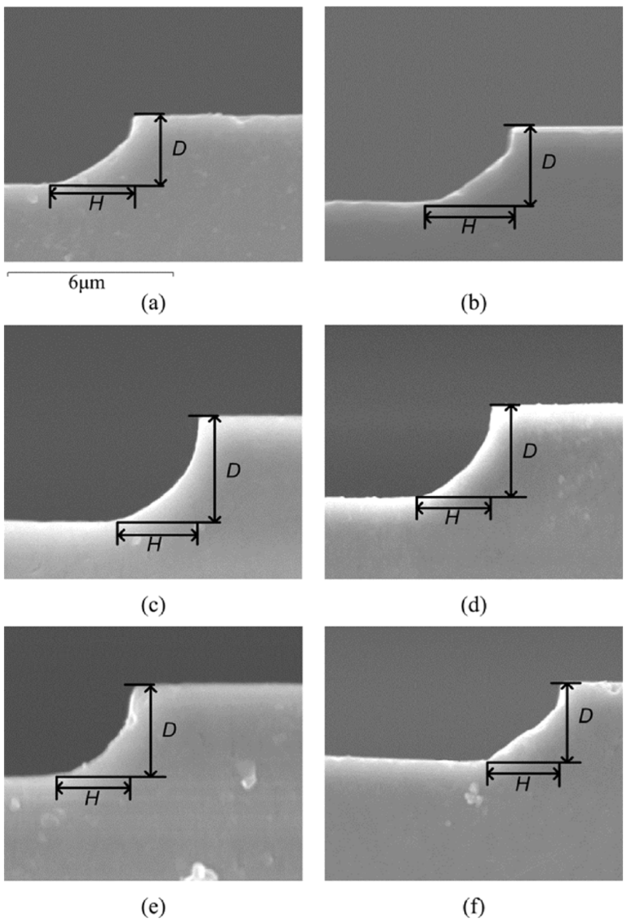

Fig. 1. SEM photographs of the etched profiles correspond to the concentration of adipic acid: (a) 0 mol%; (b) 10 mol%; (c) 20 mol%; (d) 30 mol%; (e) 40 mol%; (f) 50 mol%.

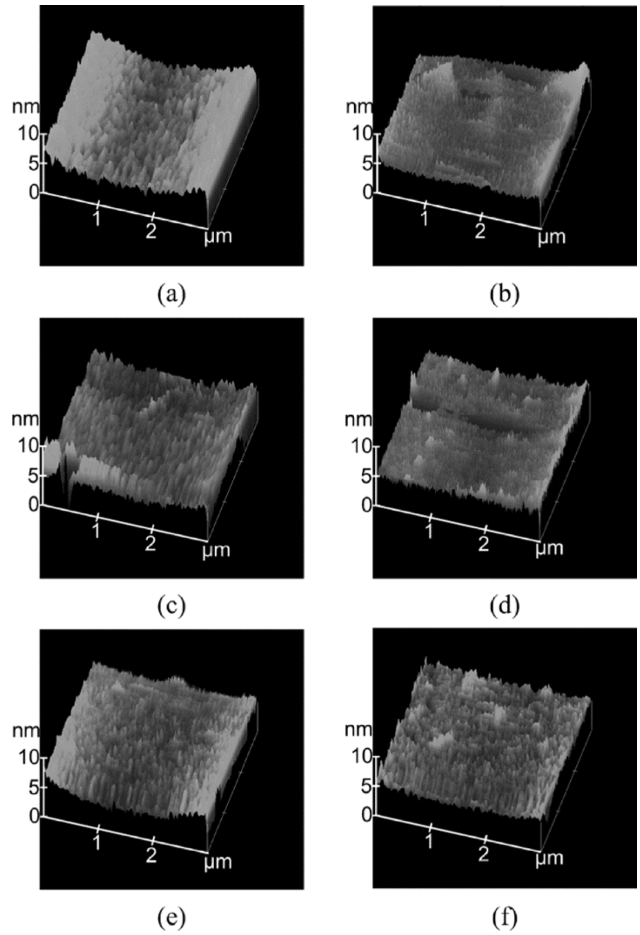

Fig. 2. AFM images of the etched surfaces correspond to the concentration of adipic acid: (a) 0 mol%; (b) 10 mol%; (c) 20 mol%; (d) 30 mol%; (e) 40 mol%; (f) 50 mol%.

TABLE I
OVERALL ETCHED DEPTHS AND HORIZONTAL DISTANCE OF SLANT FOR DIFFERENT MOLAR PERCENTAGE OF ADIPIC ACID

Adipic acid (mol%)	D (μm)	H (μm)	D/H
0	2.81	3.12	0.90
10	3.3	3.3	1.00
20	3.66	2.79	1.31
30	3.5	2.7	1.29
40	3.2	2.88	1.11
50	3.02	2.82	1.07

B. Etched Depth and Profile

To fabricate waveguide mirrors or T-junction, the etched ridge has to be deep enough, and its sidewall, as mentioned before, must be sufficiently vertical. A series of samples are examined by scanning electron microscope (SEM). Fig. 1 is the SEM photographs of the etched profiles. The distinctions are pretty obvious. Either the depths or the shapes are of great differences. To evaluate the degree of verticality, the overall etched depths D and the horizontal distance of the slant H are measured. The more vertical the ridge is, the larger the ratio of D/H will be. This gives an intuitive sense of verticality. The measured results are summarized in Table I. When no adipic acid is added, as shown in Fig. 1(a), the etched profile of this standard sample is very slant-shaped. As long as adipic acid is affiliated, an immediate change of profile has accompanied with different concentrations. The enhancement in D peaks at 30.3% with 20 mol%. Meanwhile, as a key figure-of-merit, D/H is 1.31, which is larger than others. Though the overall etched depth is enhanced, the horizontal distance of the slant is even reduced. In other words, the vertical diffusion of protons is strengthened, and the lateral diffusion is lessened. Notably, the facilitation, however, does not soar with more additive adipic acid. As more adipic acid is joining the PE process, the improvement degrades. For 50 mol%, the enhancement in D plunges to 7.5%, and the shape is again of great slant.

C. Etched Surface and Scattering Loss

The roughness of the etched substrate is analyzed by atomic force microscope (AFM). The contact-mode scans in an area of 3×3 μm are illustrated in Fig. 2. The root-mean-square (RMS) roughness of the standard sample is 1.027 nm. For other samples, the RMS roughness is no higher than 0.684 nm, which corresponds to Fig. 2(e). The lowest RMS roughness, 0.211 nm, belongs to the sample of 30 mol%. Clearly, the samples fabricated with joint acids possess smoother surfaces. Even for blue-light optical waveguide, the wavelength is longer than 400 nm, which is far larger than the measured RMS roughness. It implies that

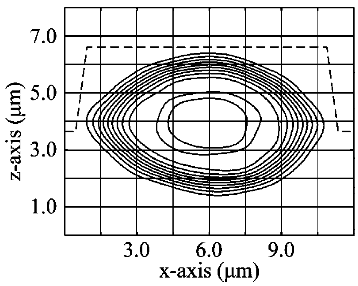

Fig. 3. Optical intensity field contour of Ti-indiffused ridge waveguide. Each contour line represents 9% of intensity drop.

TABLE II
MEASURED PROPAGATION LOSSES

Samples	Adipic acid (mol%)	PE time (hr)	Ridge height (μm)	Propagation loss (dB/cm)
A	0	8	2.8	0.7
B	20	5	2.85	0.5
C	20	8	3.6	0.6

the ridge waveguides made by the proposed method may suffer little scattering losses. To verify this, propagation losses are measured at a wavelength of 1550 nm for three different Ti-indiffused ridge waveguides. The optical intensity fields of these three waveguides are almost the same. One of the intensity field contours is shown in Fig. 3. Each contour line represents 9% of intensity drop. The visualized ridge waveguide section is illustrated as dashed lines. The measured losses are shown in Table II. The PE temperature is set at 235 °C. Samples A and C are of equal PE time but different proton sources. Sample B is set to have identical source to Sample C but roughly equivalent ridge height to Sample A. Samples produced by the proposed method have slightly lower losses than that of Sample A, which is still acceptable in practical use. To go a step further, the etched surfaces are smooth enough to produce waveguide mirrors or low-loss electrooptic components.

III. Conclusion

A novel wet etching method in $LiNbO_3$ using joint acids as the source for PE has been demonstrated in this letter. The PE with the source mixed by benzoic and adipic acid has less lateral diffusion but stronger vertical diffusion. The SEM photographs reveal that the sample fabricated by 20 mol% of additive adipic acid has the overall etched depth of 30.3% larger than that of the standard sample. The ratio of D/H is enhanced from 0.9 to 1.31. In addition, the etched surfaces are examined by AFM and propagation loss measurement. The experimental results show that the surfaces are very smooth and, hence, the scattering losses caused by etched surface are very small. It is notable that the whole process is hardly complicated compared to the conventional wet etching method. By utilizing the proposed wet etching method, high-quality ridge waveguides are successfully fabricated.

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