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SPECIAL ISSUE

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Fabrication of low-loss symmetrical rib waveguides based on x-cut lithium niobate on insulator for integrated quantum photonics

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Abstract

Lithium niobate on insulator (LNOI) is a promising material platform for applications in integrated quantum photonics. A low optical loss is crucial for preserving fragile quantum states. Therefore, in this study, we have fabricated LNOI rib waveguides with a low optical propagation loss of 0.16 dB/cm by optimizing the etching conditions for various parameters. The symmetry and smoothness of the waveguides on x-cut LNOI are improved by employing a shallow etching process. The proposed method is expected to facilitate the development of on-chip quantum photonic devices based on LNOI.

KEYWORDS

integrated quantum photonics, lithium niobate on insulator, low-loss rib waveguide, microring resonator, shallow etching

1 | INTRODUCTION

Lithium niobate on insulator (LNOI) is an attractive platform for advancing both classical and quantum photonic integrated circuits, owing to its exceptional optical properties. The high index contrast and small cross-section of submicron-thick LNOI enable tight mode confinement, which is a critical factor in fabricating compact integrated photonic devices [1, 2]. Tight mode confinement enhances the efficiency of nonlinear frequency conversion processes, such as second-harmonic generation (SHG), sum-frequency generation (SFG), and differencefrequency generation $[3, 4]$. Moreover, it maximizes the generation of entangled photon pairs in the spontaneous parametric down-conversion (SPDC) process, which is

essential for quantum communications, quantum sensing, and quantum computing applications [5–7]. The efficiency of these processes can be improved by fabricating periodically poled lithium niobate (PPLN) waveguides to establish quasi-phase-matching conditions for the interacting waves [3, 6, 8]. Thus, a waveguide based on LNOI is a remarkable platform for compact integrated quantum photonic devices [2].

Multiple PPLN waveguide devices have been demonstrated based on the x-cut LNOI, which is preferable to the z-cut LNOI due to the electrode placement and mode losses. In contrast to the x-cut LNOI, which uses planar electrodes on the surface, the z-cut LNOI typically requires the fabrication of buried ground electrodes for poling and high-speed electro-optic modulators. This is

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challenging and can cause additional optical mode losses [9, 10].

To ensure the highly efficient performance of the waveguide, strict requirements are imposed, particularly to the surface quality. The surface quality criteria typically focus on achieving low sidewall roughness, which is crucial for minimizing scattering-induced propagation losses and ensuring high-efficiency optical transmission, frequency conversion, and entanglement photon generation in quantum photonics devices [2]. Although the etching of lithium niobate (LN) thin films to fabricate waveguide structures has been extensively studied, and various techniques have been applied, achieving smooth surfaces remains challenging [11, 12].

The propagation axis of the x -cut PPLN waveguide must be aligned along the y-axis and perpendicular to the inverted ferroelectric domains along the z -axis $\left[3, \right]$ 13, 14]. However, the LN crystal structure is strongly anisotropic in the z-direction, which causes asymmetric etching characteristics along the $+z$ - and $-z$ -directions [15]. Therefore, the roughness and angle of the z-sidewalls can vary depending on the sign of z. In such waveguides, careful control and optimization of the sidewall roughness are essential because quantum entanglement is easily affected by loss-induced decoherence [2]. Nevertheless, a comprehensive study addressing

Herein, we report a fabrication method for improving the sidewall smoothness and symmetry of the x-cut LNOI rib waveguide. We obtained a high intrinsic quality factor (Q factor) of 2.58 \times 10⁶ corresponding to the low optical propagation loss of 0.16 dB/cm in our LNOI microring resonator. Various fabrication steps, including rib waveguide dry etching, wet cleaning, and shallow etching, were optimized. A shallow etching process with an $Ar/O₂$ gas mixture was applied as a novel method to improve the asymmetric lateral etching quality and roughness of y-guided waveguides on the x-cut LNOI. This method is currently applied in the fabrication of our SHG, SFG, and SPDC devices on the x-cut LNOI $[16]$. We expect that this study will contribute to the development of on-chip quantum photonic devices using LNOI.

2 | EXPERIMENTAL RESULTS

2.1 | Waveguide fabrication process

We use a 600-nm-thick x-cut LN on top of a 4.7- μ m-thick silicon dioxide (SiO₂) and intrinsic ($ρ$ > 10000 Ω cm) silicon handle wafers from NanoLN as shown in Figure 1A. A hydrogen silsesquioxane (HSQ)-based resist (FOx-16) is

FIGURE 1 Schematic of the fabrication process of an lithium niobate on insulator (LNOI) rib waveguide. (A) Preparation of an LNOI wafer; (B) FOx-16 spin-coating; (C) e-beam lithography; (D) develop; (E) inductively coupled plasma (ICP)-reactive ion etching (RIE) process; (F) ER removal; (G) wet cleaning. An asymmetrical cross-section of the waveguide can be observed after etching due to lithium niobate (LN) crystal anisotropy. See main text for details. (H) Shallow etching process with $Ar/O₂$ gases.

spin-coated onto an LNOI substrate pretreated with oxygen plasma and hexamethyldisilazane to enhance adhesion (Figure 1B), followed by the application of an anticharging agent to prevent charge accumulation. The rib waveguide pattern is defined in the FOx-16 resist using standard electron beam lithography with a beam acceleration voltage of 100 keV (EBPG 5000 plus), which mitigates proximity effects and increases the exposure precision (Figure 1C). After development, the LN thin film is dry-etched with argon plasma (Ar^+) using a commercial inductively coupled plasma (ICP) reactive ion etching (RIE) system (Figures $1D,E$). Following the plasma etching, the remaining HSQ on the rib is removed by buffered oxide etchant (Figure $1F$). Wet cleaning in a mixture of hydrogen peroxide and ammonium hydroxide is performed to remove by-products redeposited during the etching process (Figure 1G). Subsequently, shallow etching with Ar and O_2 gases is implemented using ICP-RIE, aiming to improve the sidewall symmetry and smoothness of the rib waveguide (Figure 1H).

2.1.1 | ICP-RIE process

Dry etching is suitable for fabrication of high-quality LNOI waveguides owing to its high anisotropy and controllability. Among the dry etching techniques, focused ion beam (FIB) milling [17, 18], fluorine-based RIE [19, 20], and argon-based ICP-RIE (argon ion milling) $\left[8, \right]$ $21-23$] have been used to etch LNOI [1, 24]. FIB enables the directional etching of LN thin films with high precision; however, scaling up the process to accommodate large wafer sizes is challenging $[1]$. This issue can be addressed using RIE-based techniques. However, RIE, which employs argon and fluorine-based mixtures, can cause LN surface contamination by lithium fluoride (LiF) redeposition. The LiF redeposition is difficult to eliminate; thus, it increases LN sidewall roughness and scattering losses [1, 24]. By contrast, argon-based ICP-RIE is a purely physical sputtering method that results in a less severe redeposition of chemical by-products. A high Q factor value of 10 million in LNOI was achieved using argonbased ICP-RIE [22]. Therefore, argon-plasma-based ICP-RIE is preferred for etching LNOI for integrated photonics $[21-23, 25, 26]$. Despite that, ICP-RIE is a complex process that requires the careful optimization of process parameters, such as ICP and RIE powers, working pressure, etching duration, design geometry, and temperature, to achieve a low-loss waveguide [11, 12].

The sidewall roughness of a waveguide can be improved by increasing the ICP power, which enhances the plasma density. The RIE generator controls the argon-ion bombardment (kinetic) energy, which should

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be higher than the binding energy of the LN crystal. Similarly, a higher RIE power can reduce the sidewall roughness [11]. Moreover, if the RIE bias voltage is sufficiently high, the redeposition rate decreases because of the increased reetch rate of the by-products accumulated on the sidewall. At the same time, high-energy ions can be reflected from the sidewalls and etch the slab adjacent to the rib, forming deep trenches [12]. When the ICP and RIE powers are excessively high, the etching reproducibility and rate become unstable. Thus, the power increase is limited. We succeeded in determining the stable conditions at an ICP/RIE power of 150 W/250 W (an etch rate of \sim 40 nm/min and a mask selectivity of 1:1), providing reproducible fabrication of low redeposition and low roughness waveguides. The remaining redeposition can be effectively removed and smoothed through the following wet cleaning and shallow etching processes, as discussed later. To enhance mask selectivity, we cooled the substrate temperature to 5 $°C$ [11].

Regarding the working pressure, the collision rate of argon ions decreases at lower pressures, resulting in a longer mean free path and higher kinetic energy, which improves the etching efficiency. By contrast, a higher working pressure can increase the redeposition rate due to the higher rate of atom recollisions [11]. We conducted a series of etching processes at working pressures ranging from 1 to 5 mTorr. At higher pressures of 5 and 4 mTorr, the redeposition rate was too high for the redeposited material to be completely removed from the sidewalls using the standard wet cleaning process. In contrast, at lower pressures of 3 and 2 mTorr, the redeposited material was efficiently removed via posttreatment. However, consistent results were obtained only at 2 mTorr. Meanwhile, 1 mTorr pressure was insufficient for our ICP-RIE system to maintain a stable plasma. Therefore, a working pressure of 2 mTorr was employed, whereas the base pressure was approximately 7–8 \times 10^{–7} Torr.

2.1.2 | Wet cleaning process

In the ICP-RIE process, the redeposition of LN material can significantly affect the sidewall roughness. When argon ions with kinetic energy exceeding the binding energy of the LN constituents collide with the LN surface, the constituents are ejected and scattered in all directions. Some of these scattered constituents (by-products) can adhere back to the surface of the rib, forming redeposition, which increases the sidewall roughness [11, 12]. The high roughness of the rib sidewalls strongly scatters the guided light, thereby deteriorating the optical performance of the waveguide. These redeposition-

FOx-16 Redeposition 1 μm LN 1 μm LN Redeposition waveguide (B) (C) 2 μm SiO₂ +*Z* +*X* (A) \perp_{WILEY} \vdash \blacksquare the contract of t

FIGURE 2 (A) Scanning electron microscopy (SEM) image of the lithium niobate on insulator (LNOI) rib waveguides after etching. (B) Enlarged cross-sectional view of the straight waveguide in (A). (C) Top-view SEM image of the waveguide after FOx-16 removal. The redeposition on the sidewall was observed.

induced by-products were observed in scanning electron microscopy (SEM) images taken before (Figure 2A,B) and after (Figure 2C) removal of the FOx-16 layer.

We use a solvent consisting of ammonium hydroxide (NH4OH) and hydrogen peroxide (H2O2) in a ratio of 1:1 to remove the LN redeposition induced by the ICP-RIE process. This solvent mixture has also been employed for the wet etching of LN [15]. Therefore, a wet cleaning process based on this solvent mixture is an effective way to remove the LN redeposition [11, 25]. Wet cleaning is performed by using the solvent mixture at 85 °C for 1 h. Figure 3A,B shows the bright- and dark-field optical microscopy (OM) images of the rib waveguide before wet cleaning, respectively. By-products on the rib sidewall due to the LN redeposition were observed in both OM images. After applying the wet treatment, the sidewalls roughness was reduced due to the removal of by-products (Figure 3C,D).

2.1.3 | Asymmetrically etched waveguide

We fabricated two perpendicular rib waveguides spanning the y - and z -axes of the LN thin film, as shown in the top-view SEM image in Figure 4A. Notable asymmetric etching can be observed on the sidewalls of the y-oriented waveguide (vertical), with higher roughness on the –z-sidewall (denoted by yellow arrows) compared with the $+z$ -sidewall. This could be due to the strong anisotropic properties of LN along the z-axis [15]. Magnified side views of the $-z$ -sidewall of the y-oriented rib waveguide and cross-sectional image of the same rib are shown in Figure 4B,C, respectively. The asymmetry of the rib and its roughness, which have a detrimental effect on the light propagation efficiency, were observed. Residual by-products remained on the $-z$ -sidewall even after wet cleaning. Since the anisotropy of the LN along the y-axis is less pronounced, both the $-y$ - and $+y$ -sidewalls of the z-oriented waveguide (horizontal) exhibit similar roughness, with only slight differences observed in their

FIGURE 3 Bright- and dark-field optical microscopy (OM) images of the lithium niobate on insulator (LNOI) rib waveguide before (A,B) and after (C,D) wet cleaning.

FIGURE 4 Scanning electron microscopy (SEM) images of (A) top, (B) tilted, and (C) cross-sectional views of cross-shape LN rib structure. (D–F) SEM images of the same waveguide after the shallow etching process corresponding to (A), (B), and (C), respectively.

angles. As our primary focus is on the y-oriented waveguide, which can satisfy quasi-phase-matching conditions after periodic poling along the z-axis, we will further consider only this geometry [13].

2.1.4 | Shallow etching process

A shallow etching process was further performed on the same waveguide using ICP-RIE to mitigate the asymmetric etching effect. The etching was done with an ICP/RIE power of 300 W/400 W at a gas flow rate of Ar/O2 $= 5$ sccm/15 sccm. The etch rates for the depth and top width of the rib were 6.7 and 50 nm/min, respectively. The depth of the shallow-etched layer was 20 nm. Figure 4D,E,F shows the corresponding SEM images of the top, $-z$ -sidewall, and cross-section of the rib, respectively, obtained after the shallow etching. As shown, the redepositions on the $-z$ side were completely removed by the shallow etching, improving the waveguide symmetry.

As niobium (Nb) atoms are heavy and difficult to be carried away from the LN surface during etching, they are considered to be the main constituents of the redeposited material. By introducing O2 gas during the shallow etching process, it bonds with Nb atoms and results in their effective elimination from the sidewall. Therefore, by applying the shallow etching with a low Ar/O2 gas ratio, the symmetric and smooth rib waveguides can be fabricated. Furthermore, this approach can improve the asymmetry of the y-oriented rib, as shown in Figure 4D. The overall parameters of the optimized fabrication process are listed in Table 1.

2.2 | Optical characterization

Figure 5A shows a cross-sectional schematic view of the LNOI microring resonator. After the shallow etching, the total thickness of LNOI (h_2) and the height of the rib (h_1) were 580 nm and 330 nm, respectively. The angle of the sidewall was defined as 55 [∘] . We fabricated a waveguide-

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coupled microring with a radius of 200 μm, a coupling gap of \sim 500 nm (between the top surfaces), and a top width (w_t) of 2.1 µm to measure the Q factor that directly reflects the waveguide propagation losses (Figure 5B). Figure 5C,D displays the SEM images of the right and left sidewalls of the microring, respectively, which indicate the smooth surface morphology on both z sides.

A schematic diagram of the optical setup is shown in Figure 6A. The optical transmission spectrum of the resonator was measured in the wavelength range of 1540 nm–1560 nm by scanning with a tunable external-cavity diode laser (ECDL, TLB-6728). Simultaneously, we measured the transmission of a Mach–Zehnder interferometer (MZI) with a narrow bandwidth of 204 MHz to obtain a precise resonant mode linewidth [25, 27]. The rib waveguide facet was butt-coupled using a lensed fiber (OZ Optics). The transmitted signals were detected by an avalanche photodiode (APD).

The measured optical transmission spectrum of the microring resonator and MZI are shown in the top and bottom panels of Figure $6B$, respectively. The loaded Q factor was obtained as $Q_l = 1.31 \times 10^6$ near the wavelength of

FIGURE 5 (A) Cross-sectional schematic of the lithium niobate on insulator (LNOI) microring resonator. (B) Optical microscopy (OM) image of the microring. Inset: Scanning electron microscopy (SEM) image of coupling point with bus waveguide. (C,D) SEM images of both sides of the resonator.

Abbreviations: ICP, inductively coupled plasma; RIE, reactive ion etching.

FIGURE 6 (A) Schematic of the optical measurement setup. APD, avalanche photodiode; ECDL, external-cavity diode laser; MZI, Mach–Zehnder interferometer; PC, polarization controller; VOA, variable optical attenuator. (B) Top: normalized transmission spectrum of the microring resonator corrected for background slope. The black dots and the red solid line represent the experimental data points and the Lorentzian fitting curve, respectively. Bottom: MZI transmission signal. (C) Wide range transmission spectrum of the microring resonator. The peak denoted by the red star symbol is depicted in (B).

1550 nm by fitting with a Lorentzian function. The intrinsic Q factor (Q_i) is related to the measured Q_i , as follows:

$$
Q_i = \frac{2Q_l}{1 \pm \sqrt{T_0}},\tag{1}
$$

where T_0 is the normalized on-resonance transmission, and $+$ and $-$ indicate the under- and over-coupled regimes, respectively. In the critical coupling regime, T_0 can be approximated as zero, resulting in $Q_i \approx 2Q_l$. In Figure $6B$, T_0 reaches approximately zero, indicating that the microring resonator is in the critical coupling regime. Thus, the intrinsic Q factor was obtained as $Q_i = 2.58 \times$ 106.

The mode index can be expressed by the following relationship [21, 28]:

$$
n_g = \frac{c}{\text{FSR}_f \cdot 2\pi R},\tag{2}
$$

where R is the radius of the microring, c is the speed of light, and λ is the resonant wavelength. The free-spectral range in the frequency span (FSR_f = c/λ^2 FSR_i, where FSR_{λ} is the free-spectral range in the wavelength span) was defined as 105 GHz for the red-colored peaks in Figure 6C. Based on this relationship, we obtained a value of $n_g = 2.3$. The mode index for the fundamental TE mode $(TE₀)$ of the microring resonator was calculated to be 2.31, which agrees well with the experimental data. The FSR_f of the blue peaks was \sim 99 GHz, which corresponds to the TE₁ mode with $Q_l = 5.52 \times 10^5$. The optical propagation loss can be obtained using the following relationship:

$$
L_p = 10 \cdot \frac{2\pi n_g}{Q_i \lambda} \cdot \log e. \tag{3}
$$

Consequently, the propagation loss of our rib waveguide for the TE_0 mode was estimated to be 0.16 dB/cm, which is comparable to the previously reported values on the LNOI in the telecom band $[29-32]$. If we apply the annealing process at temperatures above 500 [∘] C, the propagation optical loss can be further reduced because of the improved crystallinity of the LNOI [32, 33]. However, this approach is challenging for the PPLN waveguide devices because the periodic poled-domain structure can easily degrade or backswitch at high temperatures [34].

3 | CONCLUSION

We described a fabrication method for a low-loss symmetric rib waveguide based on the x-cut LNOI, which is a facile approach for the on-chip integration of high-performance passive and active devices with planar electrodes. A wet-etching process using a mixture of ammonium hydroxide and hydrogen peroxide effectively removed the redeposition-induced by-products on the sidewalls of the rib waveguide. However, in the case of the rib waveguide spanning along the y-axis, an asymmetric etching effect on the $-z$ -sidewall was observed owing to the LN crystal anisotropy. The sidewall roughness of the y-oriented waveguide is an important factor in the fabrication of SHG, SFG, and SPDC devices using x-cut PPLN waveguides for quantum applications. We showed that applying the newly suggested shallow

etching with $Ar/O₂$ can improve the rib waveguide sidewall symmetry and smoothness. Consequently, a high intrinsic Q factor of 2.58×10^6 was achieved, which corresponds to a low propagation loss of 0.16 dB/cm. We expect that our results will contribute to the development of highly efficient on-chip quantum devices based on LNOI.

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CONFLICT OF INTEREST STATEMENT

The authors declare that there are no conflicts of interest.

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