



Optical and structural properties of single-crystal lithium niobate thin film



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ABSTRACT

High-refractive-index contrast, single-crystal lithium niobate thin films are emerging as a new platform for integrated optics. Such lithium niobate thin films are prepared using ion implantation and direct-wafer bonding to a SiO₂ layer deposited on a LN substrate. However, the ion-implantation process can cause changes in the refractive index and result in lattice damage, and there are few studies on the optical and structural properties of lithium niobate thin film to compensate for this. In this paper, we reported that the refractive index of lithium niobate thin film can reach that of the bulk material by annealing in an oxygen atmosphere at 500 °C for 5 h. The experimental results of high-resolution X-ray diffraction (HRXRD) and Rutherford back-scattering spectrum (RBS) showed a good crystal lattice arrangement in the LN thin film. These experimental results confirmed that the refractive index and crystal-lattice structural properties of the lithium niobate thin film were similar to that of the bulk material. To demonstrate the application on integrated optics, a 1 μm wide photonic wire was fabricated and the near-field intensity profile at 1.55 μm wavelength was obtained and compared with the simulation result.

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1. Introduction

Lithium niobate (LiNbO₃, LN) is a nonlinear optical crystal widely used in integrated optics, due to its excellent electro-optic, acousto-optic, nonlinear optic properties, and wide transparency range [1]. Strong-guiding, high-refractive-index-contrast structure based on LN thin film enables ultra-small waveguides with a cross-section below 1 μm² and bending radii smaller than 10 μm, resulting in the development of ultra-compact photonic-integrated devices and circuits [2]. There are a few methods to form lithium niobate thin films, such as evaporation, sputtering, and epitaxy. However, single-crystal LN thin film is preferred to avoid the light scattering at the crystal-grain boundary so as to reduce the optical transmission loss. A favorable structure is a thin single-crystal LN thin film bonded to a SiO₂ layer deposited on a LN substrate, called lithium niobate on insulator (LNOI), which is similar to SOI (silicon on insulator). LNOI is an ideal platform for various integrated functional optical devices [3]. Free-standing LN films are fabricated by ion slicing and some excellent optical devices have been demonstrated [4]. LN thin films bonded with SiO₂ or BCB (benzocyclobutene, a well known adhesive polymer) cladding have been reported, and a micro-ring resonator is successfully fabricated [5,6]. Compared to the BCB layer, the SiO₂ cladding layer has a lower refractive index and better thermal stability, which allows

thermal annealing at much higher temperatures to recover electro- and nonlinear optical properties. Recently, the fabrication of three-inch LNOI wafers was reported, and high-quality LNOI photonic wires were developed [7], and photonic crystal was successfully demonstrated [6,8]. For device applications, the LN layer is expected to have similar physical properties as bulk crystal. However, there are few studies on the optical and structural properties of LNOI. Specifically, ion implantation can change the refractive index and cause point defects in the LN layer, which reduces the birefringence of LNOI. This paper reports that the refractive index of LNOI can reach that of bulk crystal by annealing in an oxygen atmosphere at 500 °C for 5 h. The FWHM (full-width at half-maximum) of high-resolution X-ray diffraction (HRXRD) from the LN thin film is as small as 0.0392°. We also evaluate the crystalline quality of the films using Rutherford back-scattering (RBS) spectrum. Both HRXRD and RBS show that the LN thin film has a similar crystal lattice structure as the bulk material. To demonstrate application on integrated photonics, a 1 μm wide photonic wire is fabricated using focused ion-beam (FIB) milling, and the near-field intensity profile of the photonic wire at 1.55 μm is obtained and compared with the simulation result.

2. Experimental

The process used to fabricate LNOI using ion implantation and direct bonding is schematically shown in Fig. 1. First, a Z-

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cut LN wafer was implanted by 250 keV He ions with a dose of 4×10^{16} ions/cm², forming an amorphous layer at about 810 nm underneath the surface. The He ions stayed in the dark red area shown in Fig. 1(1). Next, another Z-cut LN handle sample was coated with a plasma-enhanced chemical-vapor deposited (PECVD) SiO₂ layer polished to 1.2 μm thickness using chemical mechanical polishing (CMP). The implanted wafer and the handle wafer were directly bonded at room temperature. The bonded pair was then annealed at 190 °C for 6 h to improve the bonding strength. Using a further annealing procedure at 228 °C for 2 h, the handle wafer split along the He stayed amorphous layer and a thin LN layer 810 nm thick remained on the SiO₂/LN substrate. The sample was then annealed at 400 °C for 8 h to further the bonding strength. Using another CMP process, the roughness of the LN–air interface was reduced to 0.5 nm. The fabrication was performed at the research center of Nanolin.

The lattice damage in the LNOI caused by ion implantation could be reduced by annealing in an oxygen atmosphere. The ordinary refractive index (n_o) and extraordinary refractive index (n_e) of LN thin film were measured by the prism-coupling method using a Model 2010 prism coupler (Metricon Corporation) with a 632.8 nm laser source. During measurement the intensity of the incident light reflected from the prism base-sample interface dropped abruptly at a particular angle called the critical angle. These critical angles (α_m) were closely related to the effective refractive indices of the planar waveguide modes ($2\pi n_p \sin \alpha_m / \lambda_0 = \beta_m$, where λ_0 is the wavelength of the laser in air and β_m is the propagation constant of a specific mode “m”). If the refractive index of the prism (n_p) is known and the effective refractive indices are precisely measured, the refractive index and the thickness of the thin film could be calculated. The resolution of the refractive index measurement was about 0.0002.

The crystal lattice of the LNOI was evaluated using HRXRD. The contribution to the spectrum was expected from the crystalline LNOI film and LN substrate that possessed a periodic lattice structure, while the SiO₂ amorphous layer, which was embedded between the LN thin film and the LN substrate, had no contribution. The spectra were recorded with a single Cu K α wavelength of $\lambda = 1.9373$ Å. An AXS HRXRD D5005 system from Bruker Inc. with a resolution of 0.0002° was used. HRXRD was conducted for the CuK α 1 line. Fast θ – 2θ scans were conducted in the angle 2θ range of 30–45° with 0.01 steps at a scan speed of 1°/min to determine the position of the diffraction peak. Then higher resolution θ – 2θ scans were conducted on (006) peak of lithium niobate around 39° using the same equipment.

To further investigate the crystal structure of the LNOI, the RBS/channeling technique was used. A channeling effect measurement was used to detect the amount and depth distribution of the lattice disorder. For the “crystal” of stacked planes, the back-scattering yield decreases markedly as the crystal is tilted so that the beam is aligned with the planes. The particles in the beam can make close impact collisions only with the outermost atoms in the planes. Where there are two sets of planes at right angles to each other, the two troughs in the back-scattering yield are at right angles to each other. When the beam is aligned with the crystal at the intersection of the two troughs, the yield decreases further as the crystal appears as rows of atoms with only the uppermost atoms in the row “visible” to the beam [9]. During the experiment, a collimated 2.02-MeV He-ion beam was incident normally upon the film, and the scattered particles were collected by an Au–Si surface barrier detector at an angle of 165° from the incident direction.

The optical waveguide in LNOI was fabricated by FIB milling. Before the milling, a 100 nm thick gold film was deposited on the sample surface to avoid the charging effect during etching and scanning electron microscope (SEM) photographing. Before optical characterization, the gold film was removed. Etching was performed by focusing gallium ions onto the sample with an acceleration voltage of 30 kV. The ridge waveguide was etched by a beam current of 120 pA. Finally, the two end faces of the waveguide were smoothed by FIB to facilitate the coupling between the waveguide and the lensed fiber. The output light at 1550 nm wavelength was collected by a $60 \times /0.85$ N.A. objective with a resolution limit of about 1.1 μm.

3. Results and discussion

Fig. 2 shows the measured refractive index of n_e and n_o at the wavelength of 632.8 nm before and after annealing in an oxygen atmosphere at 400 °C/5 h, 475 °C/5 h, 500 °C/5 h, 525 °C/5 h, and 550 °C/5 h, respectively. LN is a material with birefringence. n_o and n_e of the congruent LN are 2.2860 and 2.2024, respectively, at 633 nm. Lattices damage caused by ion implantation results in a decreasing of birefringence (i.e., n_o decreasing and n_e increasing). Furthermore, the intrinsic absorption and scattering loss induced by implantation would degrade the performance of the integrated optical devices [10]. In this work, n_o and n_e of the LN film before annealing were 2.2786 and 2.2139, respectively. As shown in Fig. 3, n_o ascends while n_e descends with increasing annealing temperature. After annealing in an oxygen atmosphere at 475 °C/5 h and 500 °C/5 h, n_o and n_e , which are 2.2858 and 2.2024, respectively,

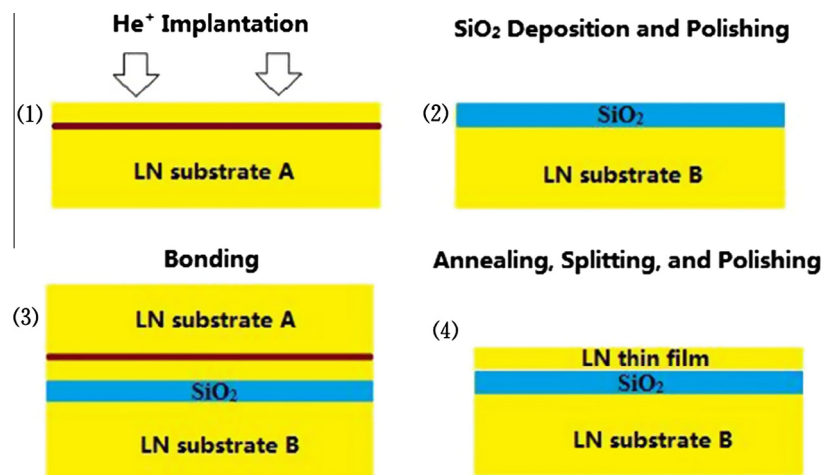


Fig. 1. Fabrication steps of LNOI wafers with three-inch diameter: (1) He⁺ implantation of a Z-cut LN wafer, (2) SiO₂ deposition on another Z-cut LN handle wafer and CMP, (3) bonding of the implanted wafer and the handle wafer, and (4) the splitting of the bonded pair and another CMP process.

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