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Lattice damage and waveguide properties of a proton-exchanged LiNbO₃ crystal after oxygen-ion implantation

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ABSTRACT

A z-cut LiNbO₃ crystal was immersed in a molten benzoic acid for 10 min and then was implanted with 6-MeV oxygen ions at a fluence of 6×10^{14} ions/cm². Lattice damage in this crystal was measured by a Rutherford backscattering and channeling technique and was compared with lattice damage in a proton-exchanged LiNbO₃ crystal and an oxygen-ion-implanted LiNbO₃ crystal. A totally amorphous layer was formed at the crystal's surface after both proton exchange and oxygen-ion implantation processes were performed, even though either process alone never led to a relative disorder of the lattice up to 0.2. It indicates that the crystal lattice in the proton-exchanged layer is unstable and can be easily damaged by ion implantation subsequently. The waveguide structure formed by proton exchange was destroyed by oxygen-ion implantation. Oxygen-ion implantation induced an increase in extraordinary refractive index and formed another waveguide structure underneath the amorphous surface layer.

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BEAM INTERACTIONS WITH MATERIALS AND ATOMS

1. Introduction

Lithium niobate (LiNbO₃ or LN) crystal has been one of the most attractive materials due to its outstanding acousto-optic, electrooptic, and nonlinear properties. A combination of the waveguide structures with its optical properties makes LiNbO₃ the most widely used material in integrated optics. The devices such as electro-optical modulators, switches, and power dividers have been used in optical information processing and optical communication. Optical waveguide structures have been fabricated on LiNbO₃ by several methods, such as proton exchange, titanium diffusion, ion implantation, femtosecond laser writing, and swift heavy ion irradiation [1–6]. Among these methods, proton exchange is the most popular one. The replacement of lithium ions with protons takes place when the LiNbO₃ wafer is immersed in a molten benzoic acid. It induces an increase in the extraordinary refractive index in the proton-exchanged surface layer and forms a waveguide between air and the substrate.

In late 1970s ion implantation was demonstrated to be an alternative method for forming waveguide structures in optical materials [7]. Since the first proton-implanted waveguide in fused silica was reported, waveguides have been so far fabricated in more than 100 optical materials (including crystals, glasses, and even polymers) by implantation of various ions at the energies of several kilo-electron-volts (keV) up to several mega-electron-volts (MeV)

* Corresponding author. Fax: +86 53188363350. E-mail address: xuelinwang@sdu.edu.cn (X.-L. Wang). [8]. There is one model to explain the waveguide structure in ion-implanted crystals. The incoming ions cause cascade collisions mostly at the end of the ion track. Large disordering of the crystal lattice is formed there, which results in an expansion of the volume and a decrease of the density. Therefore the refractive index decreases in this buried layer. This layer acts as an optical barrier which confines the light in a narrow layer between the barrier and the crystal surface with relatively higher refractive index, forming an optical waveguide. Moreover, ion implantation with appropriate fluence caused refractive index increase in some crystals, such as LiNbO₃ [3], YVO₄ [9], KTiOPO₄ [10], YAG [11], β -BaB₂O₄ [12]. The light is confined in an index-enhanced well and do not leak into the substrate.

Each method for waveguide formation in crystals distorts the crystal's lattice. Based on damage-induced volume expansion and a decrease in spontaneous polarization, the refractive index change in ion-implanted LiNbO3 crystals was related to the lattice damage ratio [13]. On the other hand, lattice distortion would changes the crystal's optical properties, mostly decreases its electro-optic coefficient or nonlinear coefficient. Ion implantation shows its advantage among these techniques. Generally, ion implantation, especially with light ions (such as H and He), causes very small deformation of the lattice in the layer between the surface and the optical barrier, where the electronic excitation is dominant. However, when the target material is pre-damaged, electronic energy deposition may act in a different way. It has been proven that huge electronic energy deposition induced by swift heavy-ion irradiation leads to recrystallization of pre-damaged semiconductors [14,15].

In this paper, the lattice disorder profiles of three LiNbO₃ crystals with proton exchange, ion implantation, and both of these processes were investigated by a Rutherford backscattering and channeling technique. Compared with the pristine LiNbO₃ crystals, ion implantation showed a different effect in the proton-exchanged LiNbO₃ crystals. The optical modes of the waveguides in the three samples were measured. And it showed that ion implantation eliminated the waveguide structure previously formed by proton exchange.

2. Experiments

Benzoic acid powder was heated to 230 °C in a sealed furnace and maintained at this temperature. A z-cut, optically polished LiNbO₃ crystal was immersed in the molten benzoic acid, was pulled out 10 min later, and cooled down slowly to the room temperature within the furnace. Then, it was cut into two pieces. One was kept at room temperature for comparison. The other, along with another pristine z-cut LiNbO₃ sample, was implanted with 6-MeV oxygen ions at a fluence of 6×10^{14} ions/cm². Ion implantation was carried out with a 1.7-MV tandem accelerator at Peking University. In order to minimize the channeling effect during the implantation, both samples were tilted 7° off the incident beam.

All three samples with proton exchange, ion implantation, and both processes were characterized by a Rutherford backscattering and channeling (RBS/C) technique using 2.1-MeV He⁺ ions as the probing beam delivered by a 1.7-MV tandem accelerator at Shandong University.

The optical modes of the waveguide structures in the three samples were detected with a Metricon 2010 prism coupler. During the measurement, a polarized light from a 633-nm He–Ne laser struck the base of a rutile prism. The intensity of the reflected light from the prism bottom was measured by a silicon photodetector. The prism, sample, and photodetector were mounted on a small rotary table so that the incident angle could be changed continuously. The intensity of the reflected light was plotted as a function of the incident angle, where a sharp drop in the intensity profile corresponds to a propagation mode.

Two end facets of the LiNbO₃ crystal with both proton exchange and ion implantation processes were polished for end-face coupling experiment. Light from a 633-nm He–Ne laser was injected into and out of the sample through two $25 \times$ microscope objective lenses. The two objective lenses were located on two three-dimensional optical stages. The sample was placed on a six-dimensional optical stage. The sample's output facet was imaged onto a charge coupled device (CCD) camera recording the field coupled out of the waveguide.

Proton exchange process only increases the extraordinary refractive index n_e in LiNbO₃ crystal. TM-polarized light propagating in z-cut LiNbO₃ crystal has a refractive index of n_e . The measurements mentioned above were collected using TM light.

3. Results and discussion

The channeling spectra of the proton-exchanged LiNbO₃ (PELN), oxygen-ion-implanted LiNbO₃ (O–LN), and oxygen-ion-implanted PELN (O–PELN) crystals are depicted in Fig. 1 by crosses, solid squares, and solid circles, respectively. The channeling and random spectrum of a pristine LiNbO₃ crystal are also depicted in Fig. 1 for damage calculation. The backscattering yields of a surface layer in the PELN sample are higher than the yields of the pristine LiNbO₃ crystal. This layer is the proton-exchanged layer where many lithium ions were replaced by protons and the crystal lattice was deformed. The channeling spectrum of the O–LN sample shows that oxygen-ion implantation alone caused a small lattice distortion in the pristine LiNbO₃ sample's surface where the electronic energy



Fig. 1. RBS/C spectra of proton-exchanged LiNbO₃ (PELN), oxygen-ion-implanted LiNbO₃ (O–LN), and oxygen-ion-implanted PELN (O–PELN) crystals measured using a 2.1-MeV He⁺ ions.

deposition is dominant. The same oxygen-ion implantation showed a different effect in the PELN sample. The backscattering yields of the proton-exchanged layer in the O–PELN sample are at the same height with the random spectrum, which means the lattice is totally disordered in the proton-exchanged layer.

The depth profiles of the displaced niobium atoms were extracted from the RBS/C spectra. This was achieved by a two-beam approximation which is based on Feldman's method [16]. The dechanneling was estimated by a multiple scattering function. The calculations were implemented by an iterative procedure starting at the sample's surface. The relative disorder at any depth was defined as the ratio of the density of displaced niobium atoms to that of the total niobium atoms in the sample. The obtained disorder profiles of the three samples are shown in Fig. 2. The proton-exchanged layer has a thickness of 0.5 µm. The maximum relative disorder in this layer of the PELN sample is less than 0.2. The relative disorder at the surface in the O-LN sample is much less than that in the PELN sample, which shows that oxygen-ion implantation at a fluence of 6×10^{14} ions/cm² caused very small distortion of the lattice in a zcut LiNbO₃ crystal. After both processes, proton exchange and oxygen-ion implantation, were performed successively, the relative



Fig. 2. Damage profiles of PELN, O–LN, and O–PELN samples calculated from RBS/C spectra by a two-beam approximation method.

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