The Influence of Annealing Treatment on H$^+$ Ion-implanted LiNbO$_3$ Waveguides

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We have studied the influence of different annealing conditions on the crystal structure and the refractive index profiles of high dose H ion implanted LiNbO$_3$ planar waveguides. After a series of annealing treatments, dark modes are measured by using a prism coupling technique. The results show that the waveguide structure was maintained after annealing at 310 °C, and disappeared completely after annealing at 490 °C. The effective indices of the dark-modes indicate a virgin-like LN structure undergoing a series of annealing treatments till 490 °C. The chemical etching experiment confirms the results checked by the prism coupling technique.

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I. INTRODUCTION

Ion implantation, as a mature method to modify the surface property of materials, is an effective way for fabricating various waveguide structures. It has been adopted for waveguide fabrication in more than 100 kinds of substrates [1–5]. One of the significant advantages of ion implantation for waveguide formation is its wide applicability and versatility for a large number of optical materials. In addition, the waveguide dimensions could be easily managed by accurate control of both the depth and lateral concentrations of the implanted atoms. Up to now, implantations of many different ions with various energies (ranging from hundreds of keV to tens of MeV) and doses have been applied to produce waveguides, in both planar and channel cases, in many optical materials, such as optical crystals, glass, semiconductors, and polymers, especially in LiNbO$_3$ crystals [6–9].

Lithium niobate (LiNbO$_3$, LN) is an important optical crystal, with many characteristics, such as large electro-optical, birefringence, nonlinear optical coefficients, intensive acoustic-optic, piezoelectric, ferroelectric, photovoltaic effect, etc. It is one of the best crystals which have the most excellent optical performance and comprehensive indices [10]. Up to now, LN is the most basic and important functional material which is being used in the optoelectronics field. It is also the most preferred optical crystal for ion implanted waveguides [11–14].

Although ion implantation has been demonstrated as an effective method in forming waveguides in LN, it also has disadvantages: it introduces damage throughout the ion range.

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and degradation in terms of absorption and electro-optic properties in the guiding region. As a result, post-implant annealing is always necessary and helpful in removing the point defects and decreasing absorption loss. It has been demonstrated that the lattice structure and the electro-optic properties can be restored to some degree by a suitable annealing treatment [15]. However, a felicitous annealing condition is crucial for such an H-implanted waveguide, since a balance has to be made between the removal of defects and the resident of the damaged lattice layer, which must ensure a good boundary for the waveguide. In order to know the influence of the annealing treatment on an ion implantation waveguide, H$^+$ ions with high dose are implanted in an LN crystal, then the samples are annealed under a series of different temperatures. The dark modes of the waveguides are measured by using a prism coupling method. The etching rate of the samples after 490 °C annealing is measured by chemical etching and a stylus profiler.

II. EXPERIMENTS

Two identical size 10 × 20 × 1 mm Z-cut LN samples with polished faces were used to deposit a Cr film of 80 nm in thickness and 3 mm in width by using the sputtering technique, which is shown in Fig. 1. The formed planar waveguide is along the y axis after ion implantation. The etching rate of the Cr film in the HNO$_3$ /HF etchant at room temperature is below 4 nm/hour, and the etching rate of the +Z LN surface is the same as the rate of the Cr film, so the Cr film can work as a mask in the −Z surface etching process [16]. Each etching thickness of LN can be measured using a stylus profiler after every etching.

FIG. 1: The scheme of the Cr film.

The Cr deposited LN samples were implanted by 500 keV H$^+$ of 1 × 10$^{17}$ ions/cm$^2$ at the Key Lab of Heavy Ion Physics, Peking University. One of the two samples is implanted on the −Z surface and the other is on the +Z surface. During implantation the LN samples are tilted 7° off normal to avoid the channeling effect.
After implantation, the two samples became a bit dark. The prism coupling instrument is used to investigate the waveguide structure, no dark-mode profile has been observed. Then the samples were annealed in the ambient air at 200 °C, 250 °C, 280 °C, 310 °C, 340 °C, 370 °C, 400 °C, 430 °C, 460 °C, and 490 °C, respectively, each annealing lasts for 30 minutes. The samples were checked for the dark mode of the waveguides with a prism couple instrument after each annealing time.

In order to obtain the structures characteristic of implanted LN crystal after 490 °C annealing, the two samples and a virgin LN crystal which was +Z surface polished are etched in turn in a mixture of 20 ml HF and 20 ml HNO₃ with a concentration of 40% and 100%, respectively. Ethanol is added into this solution with a volume of 1:7 to make sure the etched surface is flat.

III. RESULTS AND DISCUSSIONS

III-1. Profile of damage

In order to get some information of the damage caused by nuclear energy deposition after ion implantation, a SRIM2003 code (Transport of Ions in Matter) was used to simulate the process of ion implantation. According to the SRIM2003 simulation, the LN lattice damage profile caused by implantation is given in Fig. 2.

![Crystal damage of H⁺ ion implantation into LN simulated by SRIM2003.](image)

FIG. 2: Crystal damage of H⁺ ion implantation into LN simulated by SRIM2003.

From Fig. 2, it can be seen that damage caused by implantation is very small. The relative displacement of the crystal atoms in the near-surface region is very small and
negligible. Even at the damage peak, the relative displacement is only 0.0025%. The maximum lattice damage is about 3.6 μm beneath the surface, and the width of the damaged region (full width at half maximum) is about 0.3 μm.

III-2. The influence of annealing on the dark mode property

There are two kinds of modes in a planar waveguide, which are the TE mode and the TM mode. As for the TE mode, its electric field component is vertical to the incident plane when the electromagnetic wave travels along the y axis, so it is senkrecht polarization light (s light), with corresponding refractive index being the ordinary index $n_o$. As for the TM mode, it is parallel polarization light (p light), with corresponding refractive index being the extraordinary index $n_e$. The implanted samples checked by the prism coupling instrument show that there is neither a TE dark mode nor a TM one in both samples, probably because of the large absorption and scattering loss. Then the samples are annealed in the ambient air at a series of temperatures. The dark modes profiles are firstly detected after annealing at 310 °C for 30 mins. The dark-mode profiles for both the TE and TM modes after a series of annealing processes with a step of 30 °C obtained with prism coupling measurement are shown in Fig. 3 and Fig. 4.

The refractive indices of each dark-mode and the indices of the substrate are shown in Table I.

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>TE mode</th>
<th>TM mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>310</td>
<td>2.2783</td>
<td>2.2725</td>
</tr>
<tr>
<td>340</td>
<td>2.2799</td>
<td>2.2748</td>
</tr>
<tr>
<td>370</td>
<td>2.2817</td>
<td>2.2764</td>
</tr>
<tr>
<td>400</td>
<td>2.2820</td>
<td>2.2768</td>
</tr>
<tr>
<td>430</td>
<td>2.2834</td>
<td>2.2779</td>
</tr>
<tr>
<td>460</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

It can be seen from Fig. 3 for the TE mode profile that a good confined guiding mode exists after annealing at 310 °C, where the high reflectance seems unchanged on the both sides of the dip indicting a propagation mode in the waveguide. With increasing annealing temperature, the reflectance on the right side of the dip decreases, showing a lot of light through the index barrier. After 430 °C annealing, all the dark-modes have disappeared, and the reflected spectrum distribution is close to that of the substrate material. After 490 °C annealing, the measured reflective index of 2.2865 is almost the same as that of the substrate pure LN.

Slightly different TM mode profiles are found in Fig. 4. A sharp dip corresponding to a propagating mode existed in the sample after annealing at 310 °C, 340 °C, and 370 °C, respectively, and became poorly confined after annealing at 400 °C. It represents a typical
FIG. 3: Mode plots of TE polarization light after different temperature annealing.
FIG. 4: Mode plots of TM polarization light after different temperature annealing.
barrier-confined waveguide mode. Meanwhile, a mode with index larger than that of the substrate evolved from weak to strong during the annealing treatment, and became obvious after annealing at 430 °C indicating an index enhanced waveguide near the sample surface. After 460 °C annealing, no barrier-confined mode can be detected, and the effective index of the left mode approaches to the value of the substrate. After 490 °C annealing, the measurement result is fully consistent with that of the substrate material, and the refractive index is equal to the extraordinary index of the LN crystal.

It is known that an ion-implanted waveguide is formed due to the existence of a significant decrease of the ordinary refractive index in the lattice damage layer. The experimental results show that the change ∆n_o of the ordinary index in the barriers is reduced with increasing the annealing temperature. In other words, the damaged lattice caused by implantations is restored after annealing. For the extraordinary index, a raised index is observed after implantation and annealing from 310 °C to 340 °C, where the waveguide consists of a raised index region and a reduced index barrier. After 340 °C annealing, the raised index in the waveguide region and the reduced index in the barrier are weakened with increasing annealing temperature, because the damaged lattice is restored gradually. After annealing at 460 °C, there only exists a poor confinement with a small index difference between the guiding region and barrier. After 490 °C annealing, the lattice is restored fully.
FIG. 6: Etching curve of the LN +z surface: (a) virgin crystal, (b) H ion implanted.

III-3. The etching rule of H⁺ ion-implanted LN after annealing

The chemical etching experiment is as follows: Three samples were etched. One is the H⁺ ion-implanted LN sample with the −Z-face polished shown in Fig. 5. The total etching thickness is about 4 μm, covering the whole implantation range.
The result shows nearly a uniform etching rate throughout the whole implantation range. It indicates no obvious damage-enhanced region in the sample, because the implant-induced lattice damage has already been removed by annealing at 490 °C. The vibration of the etching curve is mostly caused by the change of the environment temperature while the sample is etched, because temperature can affect the etching speed to some degree.

Another is the H\textsuperscript{+} ion-implanted LN sample with the +Z-face polished, and the third one is a virgin LN sample with +Z-face polished. Their etching curves are shown in Fig. 6.

Fig. 6 shows the relations of the etching time versus depth measured by the stylus profiler. It can be found that the etched depth of the virgin LN crystal fluctuates around 0 from Fig. 6(a), meaning no detectable etching occurring at the +z surface of virgin LN. In Fig. 6(b), the average etching depth becomes negative (about −12.5 nm), which is due to the depth being measured at the boundary of the Cr coating and some Cr-film has been etched and the etching speed of LN is even slower than that of Cr-film. In as long as 20 hours, the measured depth of the LN surface keeps also unchanged, which means no etching occurring at the LN surface. The fluctuation of the step height mostly comes from the poor flatness of the original crystal. Usually the surface fluctuation of the optical polishing technique is tens of nanometers.

IV. CONCLUSIONS

The influence of annealing on a waveguide formed by 500 KeV H\textsuperscript{+} at the dose of 1 × 10\textsuperscript{17} ions/cm\textsuperscript{2} implanted LN crystal has been studied. The index-barrier confined TE mode can sustain till the annealing temperature reaches 370 °C. Upon further increasing the temperature, an obvious leakage can be found due to the light tunneling through the barrier. For the TM modes, a higher annealing temperature can be employed, since an index-enhanced waveguide can be resident till annealing at 460 °C. After annealing at 490 °C, no waveguide modes can be found, and the measured results of prism-coupling are fully consistent with that of the substrate material, indicating a restored lattice structure of LN. This conclusion is supported by the following etching experiments.

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References

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