The lattice expansion, damage effect and propagation loss of KTiOPO₄ waveguides formed by ion implantation

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ABSTRACT

We investigated the damage effect on the refractive index as well as the lattice expansion induced by C³⁺ ion implantation into KTiOPO₄ crystals. A KTiOPO₄ channel waveguide was formed by area-selective 6.0 MeV C³⁺ ion implantation at a fluence of 5×10^{13} ions/cm² using photoresist masking, and planar waveguides were formed by ion implantation with 6.0 MeV C³⁺ ions at fluences from 8×10^{12} ions/cm² to 6×10^{14} ions/cm². The propagation loss of the KTiOPO₄ planar waveguides at 633 nm fabricated at fluences of 2×10^{13} ions/cm² and 5×10^{13} ions/cm² after annealing is as low as 0.38 and 0.54 dB/cm, respectively. Prism coupling and refractive index profile reconstruction using the reflectivity calculation method were used to investigate the anisotropy of the refractive indices in the KTiOPO₄ waveguides region before and after annealing. The lattice expansion induced by ion implantation was investigated by combining X-ray rocking curves with atomic force microscopy. Rutherford backscattering spectrometry and channeling was applied for damage analysis, and the relationship between effective refractive index and relative defect concentration was studied.

Keywords: Optical waveguide; Ion implantation; Damage effect; KTiOPO₄ crystal; Lattice expansion.

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1. Introduction

Potassium Titanyl Phosphate (KTiOPO₄, KTP) with its large optical nonlinearity, high optical damage threshold and outstanding thermal stability, is an important material for integrated optics [1]. Optical waveguides, as basic component of modern integrated photonic circuits, can confine light propagation in small volumes and reach much higher optical density with respect to bulk materials [2-4]. Optical waveguides have been fabricated in various materials by ion exchange [5], ion implantation [6-9], swift heavy-ion irradiation [10] and femtosecond laser inscription [11, 12]. For the implantation of KTP with medium mass ions, such as C, an increase of the refractive indices n_x and n_y occurs in the near surface region, which is called an “enhanced well + barrier” index distribution [13]. The “enhanced well + barrier” waveguide has three advantages compared to typical “barrier” waveguide: lower ion fluences are needed for fabrication, mono-mode waveguide can be obtained and tunneling effect can be prevented [14, 15]. It was reported that in KTP waveguides formed by 6.0 MeV C³⁺ ion implantation with a fixed fluence of 1×10^{14} ions/cm², the values of effective refractive index $n_{x,eff}$ (1.7576) and $n_{y,eff}$ (1.7667) are lower but near the ones of virgin KTP crystal ($n_{xsub} = 1.7621$, $n_{ysub} = 1.7716$) before annealing. Both $n_{x,eff}$ (1.7635) and $n_{y,eff}$ (1.7727) refractive indices are higher than the ones of substrate after annealing [13].

A major effect of ion irradiation in optical materials is the modification of the refractive index, which depends on the radiation damage produced by ion implantation [16-19]. Ion implantation induced radiation damage is caused by the displacement of lattice atoms and excitation and ionization of target atoms [20, 21]. Lattice strain and stress induced by H and He coimplanted into KTP crystal was reported by Ma *et al.* [22]. However, there has little research on lattice expansion of KTP induced by ion implantation and the lattice damage effect on the properties of the KTP waveguides been done.

In this work, we investigate the effect of implantation damage on the properties of the KTP waveguides. The main purposes of this paper are first, to show the effective refractive indices of the KTP waveguides as a function of the ion fluence before and after annealing; second, to investigate the implantation damage and lattice expansion induced by ion implantation; third, to give the relationship between effective refractive index and relative defect concentration.

2. Experimental details

Z-cut KTP crystals with size of $7 \times 7 \times 1.5$ mm³ and $5 \times 20 \times 1.5$ mm³ were provided by the School of Chemistry and Chemical Engineering, Shandong University. The channel waveguide structures were created in KTP by depositing a specially designed photoresist

mask. The masked sample was implanted with 6.0 MeV C^{3+} ions at an ion fluence of 5×10^{13} ions/cm² using a 1.7 MV tandem accelerator at Peking University. Unmasked KTP samples were implanted with 6.0 MeV C^{3+} ions in the fluence range from 8×10^{12} ions/cm² to 6×10^{14} ions/cm². All implantations were carried out under an angle of 7° and the beam current density was 200 nA/cm². After ion implantation the photoresist mask was removed with acetone. The end faces along y direction of the sample were precisely polished to optical quality to allow for direct end-face coupling of light. All samples were annealed at 260°C for 30 min.

The effective refractive indices of the planar KTP waveguides were measured by prism coupling methods at 633 nm with a resolution better than 0.0002 [23]. Both the transverse electric (TE) and transverse magnetic (TM) modes can be measured through the insertion of actuator driven waveplates. The TE modes are polarized parallel to the surface of the sample, while TM modes are polarized normal to it. An end-coupling-in and end-coupling-out system was set up to measure the near-field intensity distribution of the waveguide, which is a direct way to determine whether the waveguide has the capability of confining the light in a guided mode. The propagation loss of the KTP planar waveguides was measured by the fiber probe technique using the instrument Metricon Model 2010. Laser light with a wavelength of 633 nm is coupled into the waveguide. A fiber optical probe with detector is scanned parallel to the sample surface along the direction of propagating laser light. Thus the intensity of light scattered out of the surface of the waveguide is measured as a function of distance. In order to prevent light scattering through the remaining five surfaces of the sample, these surface were blackened before analysis. The lattice expansion induced by ion implantation is measured by high resolution X-ray diffraction (HR-XRD) rocking curves and atomic force microscopy (AFM). The rocking curves from the (400) plane were obtained on a HR-XRD (AXS HRXRD D5005 system; Bruker Inc.) in the Bragg geometry with Cu $K_{\alpha 1}$ radiation. The samples were analysed by means of Rutherford backscattering channeling (RBS/C) spectrometry using 1.4 MeV He^+ ions at a backscattering angle of 170°. The RBS/C was performed in a special target chamber at the Institute für Festkörperphysik in Jena.

3. Results and discussion

3.1 Optical properties of KTP waveguides

Figure 1(a) and 1(b) show the TE and TM dark mode spectra respectively of a KTP planar waveguide formed by 6.0 MeV C^{3+} ion implantation at a fluence of 5×10^{13} ions/cm² after annealing. The refractive indices of substrate are marked for comparison. The sharp dip at an effective refractive index of 1.7724 and 1.8461 for TE and TM modes, respectively, indicates

that a waveguide structure has been formed in this sample. From Figs. 1 (a) and (b) it can be seen that n_y is higher and n_z is lower than the corresponding refractive indices of the substrate. The same effect is observed for most of the ion fluences implanted after annealing. This will be discussed in more detail below (see Figs. 3, 4 and 9). The near-field light intensity distribution measured from the output facet of the channel waveguide at 633 nm is shown in Fig. 1(c) and 1(d) for TE and TM mode, respectively, which proves that the light is well confined in the waveguide region.

Fig. 1. Measured relative intensity of light in (a) TE and (b) TM polarization reflection from the prism as a function of the effective refractive index for the KTP planar waveguide, (c) TE and (d) TM field intensity distribution of the KTP channel waveguide formed by 6.0 MeV C^{3+} ion implantation with a fluence of 5×10^{13} ions/cm² after annealing, respectively.

The propagation loss of the KTP planar waveguides was measured at 633 nm by the fiber probe technique (see Sect. 2), which is assumed to guarantee that the intensity of the light scattered out of the waveguide is proportional to the transmitted light power in the guide layer. The calculation of propagation loss requires the measurement of the value of the coupled power, the power exiting the output face of the waveguide, and the propagation length (for details see [24]). The calculated propagation loss (Fig. 2) of the KTP planar waveguides at 633 nm fabricated by implanting a fluence of 2×10^{13} ions/cm² and 5×10^{13} ions/cm² and subsequent annealing is 0.38 and 0.54 dB/cm, respectively.

Fig. 2. Intensity of scattered light versus propagation distance along the KTP waveguides formed by ion implantation with fluences of 2×10^{13} ions/cm² and 5×10^{13} ions/cm² after annealing, respectively.

Fig. 3. Evolution of effective refractive indices (a) $n_{x,eff}$ and $n_{y,eff}$ and (b) $n_{z,eff}$ versus ion fluence for first mode of planar KTP waveguides formed by 6.0 MeV C³⁺ ion implantation before and after annealing.

The effective refractive indices $n_{x,eff}$ and $n_{y,eff}$ of the first mode of the planar KTP waveguides versus fluence before and after annealing are shown in Fig. 3(a). When the fluence is below 4×10^{13} ions/cm², no waveguiding was observed in x direction for the as-implanted samples and hence, $n_{x,eff}$ cannot be determined. The waveguides are always barrier waveguides before annealing (purple and green curves), since the effective refractive indices of the modes are lower compared to the substrate. After annealing (red and blue curves), the samples implanted with fluences from 2×10^{13} ions/cm² to 1×10^{14} ions/cm², formed “enhanced well + barrier” waveguides. That is, the effective refractive indices of $n_{x,eff}$ and $n_{y,eff}$ are higher

than the substrate for the KTP waveguides fabricated at a fluence range from 2×10^{13} ions/cm² to 1×10^{14} ions/cm². Starting at a fluence of 1.1×10^{14} ions/cm², both $n_{x,eff}$ and $n_{y,eff}$ of the KTP waveguides are always lower compared to the substrate before and after annealing. The effective refractive indices $n_{z,eff}$ of the first mode of KTP waveguides versus fluence before and after annealing are shown in Fig. 3(b). In contrast to the behavior of $n_{x,eff}$ and $n_{y,eff}$, the effective refractive index $n_{z,eff}$ decreases with increasing ion fluence and is thus always well below the refractive index of the substrate. The electronic energy deposition causes a small positive change of the lower refractive index n_x , n_y and a negative change of the larger refractive index n_z [25]. All the effective refractive indices $n_{x,eff}$, $n_{y,eff}$ and $n_{z,eff}$ always increase slightly after annealing, which can be mainly attributed to lattice damage recovery. In general the well enhanced guiding region is only very slightly affected by the passage of the fast light ions, excepting that some color centers can be produced by electron ionization during the irradiation processes. Thermal annealing can remove the color center and recover the lattice damage [26].

Fig. 4. Reconstructed (a) n_y and (b) n_z refractive index profiles of the KTP waveguides formed by 6.0 MeV C^{3+} ion implantation with fluencies from 2×10^{13} ions/cm² to 1×10^{14} ions/cm² after annealing.

The reflectivity calculation method [27, 28], which has been proven to be successful for ion implanted waveguides, was used to reconstruct the refractive index distribution of the planar KTP waveguides. The anisotropic index properties of the KTP waveguides are very important for the optimization of waveguide devices such as waveguide modulators and doublers [29]. Figure 4 shows the n_y and n_z depth profiles of the KTP waveguides formed by implantation of different ion fluences and subsequent annealing. The barrier with very low refractive index is formed in a depth around 4.6 μm which the region of nuclear energy deposition of the implanted C ions. From Fig. 4 it can be seen that this barrier remains even after annealing because annealing was performed at a rather low temperature of 260°C. Post-implantation annealing at such a moderate temperature can reduce point defects within the

waveguide region while keeping the barrier and thus fabricate a low loss waveguide. The profiles for n_y shown in Fig. 4 (a) are all typical “enhanced well + barrier” distributions, which contain enhanced wells with positive index change in the waveguiding region and optical barriers with negative index change at the boundaries of the waveguides and the substrates. After annealing and for fluences from 2×10^{13} ions/cm² to 1×10^{14} ions/cm², the n_y in the waveguide region are all enhanced with respect to the substrate. n_y increases for small fluences up to 3×10^{13} ions/cm², shows a maximum at 3×10^{13} ions/cm² and decreases thereafter. Figure 4(b) shows the n_z profiles of the KTP waveguides, where n_z continuously decreases in the waveguide region for increasing fluences. Only barrier waveguides are formed for n_z .

Fig. 5. (a) Measured relative intensity of light from the prism as a function of the effective refractive index and (b) reconstructed n_x , n_y and n_z refractive index profile of the planar KTP waveguide formed by 6.0 MeV C^{3+} ion implantation with a fluence of 6×10^{14} ions/cm² before and after annealing.

The dark mode spectra of n_x (TE polarized), n_y (TE polarized, rotating 90°) and n_z (TM polarized) of the planar KTP waveguide formed by 6.0 MeV C^{3+} ion implantation with a fluence of 6×10^{14} ions/cm² before and after annealing are shown in Fig. 5(a). When the fluence is increased to high fluences at least 6×10^{14} ions/cm², the refractive indices n_x , n_y and n_z of the surface and barrier region are almost the same (as shown in Fig. 5(b)). That is, the anisotropy for highly damaged KTP crystals disappears. It should be noticed that all the effective refractive indices increase after annealing.

3.2 Lattice expansion and damage induced by ion implantation

The volume swelling was investigated by combining X-ray rocking curves with AFM. Figure 6 shows the X-ray rocking curves for KTP samples implanted with fluences of 2×10^{13} ions/cm² and 5×10^{13} ions/cm², respectively, after annealing. A spectrum of an unimplanted KTP sample is included for comparison. The rocking curves of both implanted samples consist of two components. The main component at 16.83° originates from the undamaged layer and its position corresponds to the theoretical Bragg angle of the (004) reflection of pristine KTP [30]. The intensity of this component decreases after ion implantation due to reduced crystallinity in the near surface region. The second component is a broad shoulder located at lower angles and is due to damage remaining after ion irradiation and annealing [31]. The damage-peak shift towards smaller angles corresponds to a lattice expansion of the implanted region.

Fig. 6. X-ray rocking curves for pristine KTP and KTP samples after ion implantation with fluences of 2×10^{13} ions/cm² and 5×10^{13} ions/cm² after annealing, respectively.

Bragg's Law expresses the general relationship between the wavelength of the incident X-rays λ , angle of incidence θ and spacing between the crystal lattice planes of atoms d :

$$N\lambda = 2d \sin \theta. \quad (1)$$

where N (an integer) is the “order” of reflection.

Differentiating Eq. (1), we get

$$\frac{\Delta d}{d} = -\frac{\Delta \theta}{\tan \theta}. \quad (2)$$

The differentiating form of Bragg’s law expresses a relation between the lattice constant change Δd and diffraction peak shift $\Delta \theta$. The broad shoulder induced by the **damage in the ion implanted and annealed layer, which is continuously distributed versus depth**, makes it complicated to calculate the lattice expansion. Here θ is the main peak 16.83° , $\tan \theta$ is about 0.3. The angle of the broad shoulder of the sample implanted with 5×10^{13} ions/cm² is from 16.714° to 16.814° , which is divided into ten sublayers, we get

$$\Delta d = \sum_i \Delta \theta_i \times \frac{\pi}{180^\circ} \times \frac{1}{\tan \theta} \times \frac{I_i}{I_0} \times d = 0.00323 \text{ nm} \quad (3)$$

where $I_0 = \sum_i I_i$. The damage was generated in a depth of about $5 \mu\text{m}$ below the surface of KTP according to SRIM’2008 [32]. We get the lattice expansion height Δh

$$\Delta h = \Delta d \times \frac{D}{d} = 15.2 \text{ nm} \quad (4)$$

The relative lattice expansion is about 0.3%.

Fig. 7. (a) AFM image and (b) cross section profile of the KTP channel waveguide formed by area-selective 6.0 MeV C^{3+} ion implantation with a fluence of 5×10^{13} ions/cm² after annealing.

The surface morphology of the $10 \mu\text{m}$ width channel waveguide fabricated by area-selective 6.0 MeV C^{3+} ion implantation with a fluence of 5×10^{13} ions/cm² was investigated by atomic force microscopy after annealing (see Fig. 7). The lattice expansion along the z -axis is

about 17 nm, which is consistent with the calculated results of HR-XRD. The lattice expansion is most likely caused by the incorporation of the C^{3+} ions within the KTP crystal, nuclear damage formation and ionization-induced lattice relaxation. Small volume expansion occurs due to point-defect accumulation before amorphization [33]. The trapezoidal shape of the waveguide region (Fig. 7(b)) is caused by C^{3+} ions that penetrate through the sloped edges of the photoresist mask [34].

Fig. 8. (a) electronic energy loss and (b) energy spectra of 1.4 MeV He^+ ions backscattered on KTP implanted with 6.0 MeV C^{3+} ions to different ion fluences after annealing.

As shown in Fig. 8(a), the electronic energy loss of 6.0 MeV C^{3+} ion implanted KTP in the near surface region is almost the same. Figure 8(b) depicts the RBS/C spectra of 6.0 MeV C^{3+} ion implanted KTP for different ion fluencies and after annealing. Virgin and random spectra are also given for comparison. Only the damage in the near surface region of waveguides, which is dominated by electronic energy deposition, can be unambiguously analyzed from the RBS/C spectra. The backscattering yield in channeling direction increases with increasing ion fluence due to the increasing lattice damage. The initial energy of 1400 keV He^+ ions is reduced to about 1002 keV, 928 keV, 833 keV and 508 keV after backscattering on Ti, K, P and O, respectively. The relative concentration of displaced lattice atoms n_{da} at the near surface is 0.1, 0.15, 0.18, and 0.4 for the samples with different fluencies, which is obtained by analyzing the signal of the Ti sublattice.

Fig. 9 shows the effective refractive indices of $n_{x,eff}$ (blue), $n_{y,eff}$ (red) and $n_{z,eff}$ (purple) as a function of relative defect concentration n_{da} in the KTP samples. It should be noted that the n_{da} is dominated by electronic energy loss in the waveguide region, not by nuclear energy loss at the barrier region. As one can see, when n_{da} is small, $n_{x,eff}$ and $n_{y,eff}$ increased, $n_{z,eff}$ decreased, decreasing the overall refractive index anisotropy. $n_{x,eff}$, $n_{y,eff}$ and $n_{z,eff}$ are all decreased with n_{da} larger than 0.4, and $\Delta n_z > \Delta n_y > \Delta n_x$. At high fluences with large n_{da} , continuous bombardment destroys the longer range lattice structure, which can decrease the

crystalline symmetry and molar polarizability. The changes of $n_{x,eff}$, $n_{y,eff}$ and $n_{z,eff}$ in the waveguiding region are attributed to the decrease of anisotropy of KTP samples, which is due to the changes of molar polarizability and molar volume induced by ion implantation damage [35].

Fig. 9. Evolution of effective refractive indices (a) $n_{x,eff}$ and $n_{y,eff}$ and (b) $n_{z,eff}$ versus relative defect concentration n_{da} of planar KTP waveguides formed by 6.0 MeV C^{3+} ion implantation after annealing.

4. Summary

A KTP channel waveguide was successfully formed by combining 6.0 MeV C^{3+} ion implantation at a fluence of 5×10^{13} ions/cm² with photoresist masking. Planar waveguides were formed by ion implantation with 6.0 MeV C^{3+} ions at fluences from 8×10^{12} ions/cm² to 6×10^{14} ions/cm². The propagation loss of the KTP planar waveguides at 633 nm fabricated with ion fluences of 2×10^{13} ions/cm² and 5×10^{13} ions/cm² is as low as 0.38 and 0.54 dB/cm, respectively. The samples implanted with fluences from 2×10^{13} ions/cm² to 1×10^{14} ions/cm² formed “enhanced well + barrier” waveguides after annealing. After annealing the n_y in the waveguide region increases with increasing fluence, reaching a maximum at 3×10^{13} ions/cm² and decrease thereafter. The refractive indices n_z decreases with increasing ion fluence and is thus always well below the refractive index of substrate. When the ion fluence is increased to 6×10^{14} ions/cm², the anisotropy of the refractive indices vanishes completely in the surface and barrier region of the waveguide. The lattice expansion along the z -axis is about 17 nm for the sample implanted with fluence of 5×10^{13} ions/cm². RBS/C was applied for damage analysis and the effect of relative concentration of displaced lattice atoms n_{da} on effective refractive index was studied.

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