Ion-exchanged waveguides in periodically poled Rb-doped KTiOPO₄ for efficient second harmonic generation

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Abstract: An ion-exchange process has been developed for periodically poled Rb-doped KTiOPO₄ (RKTP) which warrants high efficiency and low loss channel waveguides. The domain stability was investigated, and it was found that domain gratings with uncharged walls could stand the ion-exchange process without deterioration. 3.1 mW of blue second harmonic light was generated from 74 mW of radiation at 940.2 nm coupled into an 8 μm wide and 7 mm long waveguide, corresponding to a normalized conversion efficiency of 115%/Wcm². Waveguides in PPRKTP open the possibility for stable operation at high optical powers, as well as generating entangled photons at low optical powers, and enable the investigation of novel nonlinear processes such as counter-propagating interactions in a waveguide format.

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1. Introduction

Nonlinear interactions can be tailored with quasi-phase matching (QPM) to generate coherent light at desired wavelengths [1], and these can be efficient in waveguides as a high intensity is maintained over a long interaction length [2]. Waveguides also provide higher robustness, stability and compactness compared to traditional free-space optics. All of this is of utmost importance in quantum optics applications where parametric processes are used to generate entangled photon pairs with low intensity light sources [3–5]. Mostly, periodically poled LiNbO₃ (PPLN) waveguides have been used for these applications so far, as the material is of high quality and the waveguide fabrication process in LiNbO₃ (LN) is well established [6]. However, it is difficult to fabricate QPM structures in LN for visible wavelengths by periodic poling, as it requires dense QPM gratings, and the material is also susceptible to photorefractive effects, as well as green and blue induced infra-red absorption (GRIIRA, BLIIRA), which impose limitations when short-wavelength lasers are used [7–9]. Furthermore, for traditional titanium-diffused and annealed-proton-exchanged waveguides the fundamental modes are elliptical which leads to poor mode-matching with optical fibers. However, it should be pointed out, that MgO doped LN (MgOLN) can be used to reduce GRIIRA [8], and with the novel thin film LN on insulator platform it has been possible to fabricate short and even sub-μm period QPM gratings [10,11]. These have been used for record-high efficiency nonlinear conversion [12]. On the other hand, it is difficult to efficiently butt-couple to such waveguides with optical fibers and it might require grating assisted in- and out-coupling [13].

In this context is KTiOPO₄ (KTP) an interesting alternative platform to LN and MgOLN as it can be periodically poled with short periods for use in first order (m=1) QPM [14] and it does not experience photorefractive effects and is much less sensitive to GRIIRA and BLIIRA [15,16]. Furthermore ion-exchanged waveguides can be made in KTP with circular mode-profiles, which

provides a high overlap between interacting modes and an excellent coupling to optical fibers [17].

Entangled photon-pair sources pumped in the blue-green wavelength range, together with inexpensive and efficient near-IR avalanche and single-photon detectors will likely find practical implementations in real world quantum communication systems [18,19]. For this reason, interest in KTP waveguides has increased considerably. Still several problems must be overcome before they outperform those in LN. Segmented ion-exchanged waveguides, which were first introduced in conventional flux-grown KTP [20–22] have some stability issues, particularly during high-power operation [23]. Waveguides made by laser writing were also developed, but these are comparably lossy [24–26]. Later, by fabrication of diamond-diced ion-exchanged waveguides it was possible to get good modal overlap and obtain low loss [27–29].

An improvement over segmented waveguides could be the continuous ones, where the QPM domain grating and the waveguide are fabricated independently in two separate steps. The remaining problems to address then will be the reproducibility in the waveguide fabrication, which is related to composition variations in the as-grown crystal resulting from K^{+} -vacancies, as well as maintaining a good domain grating after ion-exchange and long term waveguide stability [23].

An alternative substrate material to KTP for waveguide fabrication is flux-grown Rb-doped $KTiOPO_4$ (RKTP) [30]. This commercially available material is obtained by adding a small concentration of Rb ions in the melt during growth, which results in a Rb-concentration of 0.3% in the as-grown crystals. Recently, we reported fabrication of ridge waveguides in periodically-poled RKTP (PPRKTP) using a planar Rb^{+} -ion exchange in PPRKTP followed by diamond-blade dicing [28]. Unfortunately, these waveguides presented a low refractive-index contrast between the waveguide region and the substrate, and relatively high loss. The loss was caused by the rough edges obtained from the dicing process; whereas the low refractive-index change was attributed to the fact that RKTP already contains a small amount of Rb, which slows down the ion-exchange and partly blocks the Rb^{+} -ion diffusion. In order to fabricate well-confined waveguides in PPRKTP, it is therefore important to accelerate the ion-exchange process while still preserving the periodic domain structure.

In this work we present the fabrication of channel waveguides in PPRKTP crystals with a QPM grating with a period of $5.82\ \mu m$. The waveguides had low loss and demonstrate efficient blue second-harmonic generation (SHG) via type 0 QPM. Moreover, the key parameters needed to maintain high conversion efficiency in periodically-poled waveguides were identified. These results could pave the way for counter-propagating nonlinear interactions [31] in waveguide format.

2. Waveguide fabrication and domain structure

Ion-exchange in KTP with monovalent ions, like Rb^{+} , is a diffusion-controlled process, taking place through the c-face of the crystal. It depends on several parameters such as the temperature, ion-exchange time, melt composition and the number of defects/vacancies in the crystal [32]. When the crystal is submerged in the melt with dopants, K^{+} -ions close to the c-surface diffuse out of the crystal and leave a negatively charged potassium vacancy behind. These negatively charged vacancies, in turn, electrostatically attract the dopant ions in the melt, whereby they diffuse into the crystal to counteract the charge unbalance. Moreover, preexisting vacancies, obtained during the crystal growth, attract dopant ions from the melt. In fact, in a purely monovalent melt the diffusion rate of the dopant ion in the KTP crystal is strongly dependent on the pre-existing vacancy concentration. The Rb^{+} -ion has a larger ionic radius and higher activation energy than the K^{+} -ion and is therefore much less mobile in the crystal. The depth profile of the

Rb⁺-concentration follows a complementary error function:

$$c(z) = c_S \times \operatorname{erfc}\left(\frac{z}{d}\right) + c_{KTP}, \quad (1)$$

where c_S is the Rb⁺-ions concentration change at the surface, c_{KTP} is the Rb⁺-ion concentration in the bulk and d is the characteristic depth of the exchange. With a linear relation between Rb⁺-ion concentration and refractive-index change the latter can be written [32]:

$$n(z) = n_S \times \operatorname{erfc}\left(\frac{z}{d}\right) + n_{KTP}, \quad (2)$$

where n_S is the refractive index change at the surface, d is the depth of the exchange and n_{KTP} is the bulk refractive index.

For RKTP a small amount of Rb⁺-ions are already located at the larger K(2) sites in the crystal [30], and when Rb-exchange is performed the intrinsic Rb⁺-ions partially block the K⁺-channels, leading to a slower and shallower ion-exchange. It results in a refractive index change considerably lower than what is obtained for ordinary flux-grown KTP. On the other hand, the fact that the larger Rb⁺-ions are less mobile in the RKTP lattice, means that Rb-exchanged waveguides in RKTP will be much more stable than those made in KTP, as it is known that the Rb-waveguides can diffuse in KTP over time when operated at high optical powers [23].

In order to obtain a larger diffusion rate and deeper exchanged layers, a divalent ion, as, Ba²⁺, can be added to the exchange melt [32,33]. The divalent ion diffuses together with a negative vacancy, V⁻, to maintain charge neutrality of the system, and these vacancies become diffusion sites for Rb, leading to a faster ion-exchange.

However, it is reported that domain inversion can occur when Rb⁺- and Ba²⁺-ions are substituted into the polar surfaces of KTP [34], which can erase or deteriorate the domain grating in periodically poled crystals. To avoid this effect, K⁺-ions can be added to the melt to reduce the maximum Rb-concentration in the crystal. Thus, the ideal ion-exchange process should avoid domain reversal, and at the same time grant as large refractive-index change as possible to get tight mode confinement. Here, we compared ion-exchange with three different nitrate melt compositions on planar c-cut PPRKTP samples, studied waveguiding, and the impact on the domain structure.

The three melt compositions, together with the resulting surface refractive index change and the number of modes are displayed in Table 1. The first one was a pure RbNO₃ melt. For the remaining two we used a 20 mol% K⁺-concentration and varied the ratio of Ba/Rb. All exchanges were carried out at 340°C for 2 hours. Afterwards, the TM mode indices were measured in the planar waveguides via prism coupling with a He-Ne laser at 633 nm and a blue laser at 457nm, and the corresponding index profiles were calculated using the inverse-WKB method [35]. As expected, the melt with the largest Ba-concentration gave the deepest waveguide and the largest refractive index increase. In a complementary measurement the Rb-profile was measured with energy-dispersive x-ray spectroscopy (EDX) on the sample exchanged with 5mol% Ba in the melt. The characteristic depth of the exchange was determined to be 8.5 μm, in good agreement with the depth calculated from the mode data from the prism coupler. The Rb-concentration profile fits well to a complementary error function, see Fig. 1. It should be noted that the Rb-exchange is much slower for RKTP than for KTP, as expected [36].

In order to investigate whether the ion-exchange process has a negative impact on the domain structure, the following experiment was done. Four in-house poled PPRKTP samples with 3.414 μm periodicity were selectively etched in a KOH-KNO₃ solution at 100°C in order to visualize the domain structure. Three of the samples underwent ion-exchange, using the melt

Fig. 1. Rb concentration profile measured below the surface with EDX.

Table 1. Melt compositions used for ion exchange in RKTP, surface refractive index change and number of modes

Melt composition	Surface refractive index change n_s	Number of modes at 633 nm
Pure RbNO ₃	0	0
77mol% RbNO ₃ / 20mol% KNO ₃ / 3mol% Ba(NO ₃) ₂	0.0021	2
75mol% RbNO ₃ / 20mol% KNO ₃ / 5mol% Ba(NO ₃) ₂	0.0045	4

compositions displayed in Table 1. The fourth one was solely submitted to the same temperature treatment (340°C for 2h). Afterwards, the samples were etched a second time to reveal any changes in the domain pattern. First, it is worth noting that pre-etching of the crystal surface did not influence the ion-exchange process, as the refractive-index change measured for these samples was consistent with the results presented in Table 1. Changes in the domain structure of similar nature were seen for all the three ion-exchanged samples, regardless the melt composition, as well as in the sample that was solely exposed to the temperature treatment. Figure 2, shows an optical micrograph of the etched relief on the c-faces of a representative sample, where domain merging and back-switching can be seen. The domain structure prior to ion-exchange is seen slightly dimmer while it appears with larger contrast after the exchange and the second etching step. Note that the areas that remained unchanged after ion-exchange (marked with arrows) were the well-poled ones with straight domain walls throughout the crystal thickness. These results are consistent with our previous studies on thermal stability of domains [37], although they were done for longer time and at much higher temperature (12 h, $T=730^\circ\text{C}$): the domains can merge or back-switch during heat treatment if the domain walls are charged, which happens when they are tilted (non-parallel to the polar axis). This tilting normally happens, either as the domains have not propagated fully through the bulk, or because the domains have broadened during periodic poling.

From the present results it becomes obvious that straight, uncharged domain walls throughout the crystal thickness are required to avoid deterioration of the domain grating, and that the ion-exchange process has no major impact on the QPM grating. Moreover, cutting the sample sides, in order to leave the domain ends to air uncharged [38], might be needed for fabrication of waveguides with short periodicities.

Fig. 2. Optical micrograph of the etched crystal with corresponding domain structures on the former c^- face. The domains that back-switched or merged appear as dimmer in the picture. The arrows show the domain regions with straight domain-walls that extend throughout the crystal thickness. These regions were not affected by the temperature or ion-exchange treatments.

3. Optical experiments

In order to fabricate a QPM waveguide, first a flux grown z-cut RKTP sample of dimensions $10 \times 6 \times 1 \text{ mm}^3$ (x,y,z) was periodically poled with a period of $5.82 \mu\text{m}$ using 5 ms long electric-field pulses [39]. The resulting QPM grating had a length of 7 mm. The poling quality was assessed by first order QPM SHG using a continuous wave (cw) Ti-Sapphire laser. The infrared beam was polarized along the z-direction to exploit the large d_{33} coefficient of RKTP and focused into the crystal to a radius of $20.1 \mu\text{m}$. We translated the sample in the focused beam and revealed high quality QPM structure throughout the whole crystal aperture with a normalized conversion efficiency of 2.3%/Wcm at the phase-matching peak at 933.8 nm. This indicated that the QPM structure had straight and uncharged domain walls.

The sample was patterned on the polar face to form channel waveguides oriented along the x-direction with widths varying from 2.4 to $12 \mu\text{m}$ using standard photolithography. Subsequently, ion-exchange was done with the 5mol% $\text{Ba}(\text{NO}_3)_2$ / 20mol% KNO_3 / 75mol% RbNO_3 composition at 340°C for 2 h. The end facets of the device were then polished to facilitate coupling into the waveguides. Figure 3 shows the top view of an $8 \mu\text{m}$ wide waveguide, where the surface has been

etched to visualize the waveguide. Note that the high-fidelity domain structure was unaltered during the ion exchange process.

Fig. 3. Top view of an 8 μm wide channel waveguide in PPRKTP.

The waveguides were then tested for SHG with the cw Ti-sapphire laser at room temperature. The laser light was launched into the waveguides using an objective lens with 20X magnification focusing the beam down to a beam radius of 3.9 μm. The highest blue SH power, 3.1 mW, was obtained in the fundamental TM₀₀ mode in an 8 μm wide waveguide, from 74 mW of IR radiation at 940.2 nm coupled into the waveguide. Considering the length of the poled structure of $L = 7$ mm, we calculate the normalized internal conversion efficiency as:

$$\eta_{\text{experimental}} = \frac{P_{2\omega}}{P_{\omega}^2 L^2} = 115 \frac{\%}{\text{Wcm}^2}, \quad (4)$$

where P_{ω} and $P_{2\omega}$ denote the power of the fundamental and the second harmonic waves, respectively. This number is close to the best reported values for KTP waveguides [40–42], but still below the highest number for blue light SHG in PPLN waveguides, 600%/Wcm² [43].

Simulations of the mode profiles were done using COMSOL with refractive index data from the prism coupling measurements and the Sellmeier equations of KTP [44]. The overlap between the focused Gaussian input beam and the fundamental mode, TM₀₀, could then be estimated to 92%. By measuring the input and output power in the IR and taking the Fresnel losses of the end facets of the waveguide (8.7%) into account, we calculated the waveguide transmission to be 89%. The full length of the waveguide was 9.4 mm resulting in a waveguide loss of 0.5 dB/cm at 940 nm.

The phase-matching curve of the 8 μm waveguide is shown in Fig. 4, together with CCD images of the two strongest blue SH modes, TM₀₀ and TM₀₁. The lower peaks correspond to

higher order modes (TM_{10} and TM_{11}). The acceptance bandwidth (FWHM=0.24 nm) of the TM_{00} mode is in close agreement with the theoretical expected bandwidth (0.21 nm), confirming a high uniformity of the waveguide. The corresponding temperature bandwidth was calculated to 3.4 °C.

Fig. 4. Measured SH output vs. pump wavelength. The insets are the CCD images of the two strongest SH modes TM_{00} and TM_{10} , at 470.1 nm and 470.7 nm, respectively.

4. Conclusion

In conclusion, we have developed a method to fabricate low-loss channel waveguides in PPRKTP using ion-exchange in a melt containing Rb^+ , Ba^{2+} and K^+ . To minimize a risk of obtaining a deteriorated domain grating during the ion-exchange a study of domain stability was performed, and it was found that well-poled domains with uncharged, vertical walls could stand the processing without either back-switching or merging. Straight channel waveguides with low loss (0.5 dB/cm) were then fabricated and a SH conversion efficiency of 115%/Wcm² was obtained in an 8 μm wide waveguide with 940 nm fundamental light. These results open the possibility for fabrication of waveguides in PPRKTP that are stable in operation at high optical powers and enable investigation of nonlinear processes such as counter-propagating interactions in a waveguide format.

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Disclosures

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