Characterization of proton-exchanged waveguides in MgO:LiNbO₃

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We report the fabrication and characterization of proton-exchanged waveguides in MgO-doped LiNbO₃, a high-optical-damage-threshold material. Results indicate waveguide characteristics similar to those of waveguides fabricated in undoped LiNbO₃ except for slower diffusion rates and freedom from etching of the y face when pure benzoic acid is used as a proton source. An optical-damage threshold of 70 kW/cm² was measured at 0.5145 μ m in a MgO:LiNbO₃ waveguide, corresponding to a factor-of-2 improvement over undoped LiNbO₃.

It was shown by Zhong *et al.*,¹ and more recently by Bryan *et al.*,² that LiNbO₃ doped with approximately 5% or more MgO exhibits a remarkably reduced photorefractive response compared with undoped LiNbO₃. It is believed that the reduced photorefractive response is due to the increased photoconductivity of the MgO:LiNbO₃.² Such a material offers great promise for nonlinear and integrated optics, for which induced photorefractivity (optical damage) has been a serious limitation in the past. It must be noted that the enhanced photoconductivity may cause deleterious effects in devices requiring the application of low-frequency electric fields.

As a first step toward the demonstration of efficient nonlinear guided-wave devices, it was interesting to fabricate proton-exchanged waveguides in MgO:LiNbO₃. Compared with other waveguide fabrication processes in LiNbO₃, proton exchange has been shown to be a rapid low-temperature process and to yield waveguides with interesting applications in polarization filtering and birefringence control.³ In this Letter we report the fabrication of H⁺-exchanged waveguides in MgO:LiNbO₃ and describe the waveguide characteristics and power handling at short wavelengths.

Waveguides were fabricated in x- and y-cut 5% MgO-doped LiNbO₃ crystals grown by Crystal Technology, Palo Alto, California. The now standard technique of proton exchange in a melt of benzoic acid containing varying amounts of lithium benzoate was used.^{4,5} Benzoic acid has been shown to have an appropriate dissociation constant, melting temperature, and stability as a liquid for use in this process.^{3,4} The addition of lithium benzoate to the melt provides a means of controlling the proton concentration in the exchanged region, which has a strong bearing on the crystal structure and the optical properties and quality of the waveguide.⁶

The acid melt, containing between 0 and 2 mol % lithium benzoate, was contained in a glass flask equipped with a condensing column. The column was used to reduce material loss and composition change of the melt, as the waveguide characteristics have been

shown to depend strongly on the concentration of Li⁺ ions in the melt.⁴ The wafer to be exchanged was held in a glass tube provided with lateral cuts to let the liquid penetrate inside the tube. All exchanges were performed at the boiling temperature of the melt, about 249°C in pure acid and 246 and 243°C for melts containing 1 and 2% lithium benzoate, respectively. This arrangement yielded easily reproducible results.

After fabrication the waveguide index profiles were characterized using the prism-coupling launching-angle measurement to determine the effective indices of the guided modes. The guide index profile was then recovered from the mode indices with a standard inverse WKB method. Figure 1 shows a typical set of index profiles measured at different wavelengths for an x-cut MgO:LiNbO₃ waveguide exchanged for 3 h in pure benzoic acid. The profiles exhibit the same step-index shape as is characteristic of proton-exchanged waveguides in undoped LiNbO₃ with an increase of the ex-

Fig. 1. Typical n_e and n_o profiles of a proton-exchanged waveguide in MgO:LiNbO₃. The x-cut wafer was processed for 3 h in pure benzoic acid at 245°C. The profiles were shifted vertically for clarity.

Fig. 2. Waveguide depth versus the square root of the exchange time in doped and undoped LiNbO₃ treated in melts of different compositions.

traordinary index (of the order of 0.135 at the wavelength of $\lambda = 0.6328 \mu\text{m}$) and a decrease of the ordinary index. This result indicates that the presence of MgO in the crystal lattice does not significantly alter the exchange process.

The negative ordinary index profile was measured by launching a wave at an angle θ to the usual propagation direction (along y) in an x-cut waveguide. The resulting index step Δn seen by the wave is then a known combination of Δn_e and Δn_o , from which the latter can easily be extracted. Alternatively, one can measure the critical angle θ_c for which the wafer no longer supports a guided mode. This method yielded the value of $\Delta n_o = -0.06 \pm 0.006$, in good agreement with previously published values for the undoped material.⁴

We show in Fig. 2 the evolution of the waveguide depth with the square root of the exchange time for waveguides fabricated in MgO-doped and -undoped LiNbO₃ in melts of different compositions. The dependence is linear, which suggests a diffusionlike behavior for both materials. One can thus characterize the exchange rate by the diffusion coefficient D , defined by $d = (4Dt)^{1/2}$, where d is the depth (at half-maximum) of the waveguide and t the exchange time. As is shown in Fig. 3, the exchange rate decreases as the Li⁺ melt concentration is increased, following an exponential law previously established for undoped LiNbO₃.⁷ The exchange rate is lower in z-cut than in x-cut wafers for the undoped material, and is even lower in the x-cut orientation for the doped material. This result is similar to the situation in Ti-diffused LiNbO₃, where the presence of Ti slows down the proton-exchange rate.⁸ Note that the exchange rate is still very high with melts containing up to 1% of lithium benzoate; a single-mode waveguide can be fabricated in less than a few minutes.

Waveguides were also fabricated in y-cut MgO:LiNbO₃ in pure benzoic acid with no evidence of surface etching up to the maximum exchange time that was tested, about 4 h. Again, this observation parallels that of other authors concerning proton exchange in waveguides previously doped with titanium.⁸ The diffusion

rates were found to be essentially the same for x-cut and y-cut MgO:LiNbO₃.

Figure 4 illustrates the index changes Δn_e measured in two MgO:LiNbO₃ waveguides at different wavelengths and the corresponding single-pole Sellmeier curve to which they were fitted. The index change was found to vary substantially across the visible and near-infrared ranges, from about 0.18 at $0.4579 \mu\text{m}$ to 0.12 at $0.820 \mu\text{m}$ for waveguides made in pure acid. This fairly strong Δn_e dispersion indicates a higher index dispersion for Li_{1-x}H_xNbO₃ (whether or not doped with MgO) than for unexchanged LiNbO₃ for the wide range of proton concentration x that was tested. For a given Li concentration, we found that Δn_e was larger in the doped than in the undoped material by about $2-3 \times 10^{-3}$ for x-cut samples. Δn_e decreases linearly with increasing Li concentration in the melt, with essentially the same slope for the MgO-doped (x-cut) and undoped (x and z cuts) material. We measured a slope $d(\Delta n_e)/d[L_1^+]$ of $-0.019/\text{mol } \%$ at $\lambda = 0.6328 \mu\text{m}$.

Several authors reported aging of proton-exchanged LiNbO₃ waveguides, apparent as a reduction of the surface index and of the waveguide depth over a period of a few days, especially in waveguides fabricated in pure acid.⁷ The same phenomena were observed in H⁺:MgO:LiNbO₃ waveguides. Stronger mode coupling was also noticed in waveguides exchanged in pure acid. Postannealing, a process that was shown to eliminate these difficulties,⁷ is clearly also needed for doped LiNbO₃ to control the crystal phase of the exchanged layer and stabilize its surface index. Since we did not anneal our waveguides after exchange, we performed all the measurements described here approximately one day after the wafer was removed from the melt to keep our results consistent.

Zero-field photorefractive sensitivity (optical damage) in both types of waveguides was observed to have a fairly high threshold at short wavelengths. Up to about 1 mW of light was coupled into our waveguides for routine characterization without apparent long-term effects on the waveguide index, even at $0.4579 \mu\text{m}$.

Fig. 3. Comparison of proton-diffusion rate in doped and undoped LiNbO₃ as a function of the Li⁺ concentration in the melt.

Fig. 4. Δn_e dispersion of x-cut MgO-doped LiNbO₃ waveguides for two Li⁺ concentrations in the melt.

Quantitative measurements also indicated a good short-term tolerance to relatively high optical intensities. Short-term optical-damage thresholds were measured by prism coupling a 0.5145- μ m beam focused in the waveguide plane to a 40- μ m-diameter spot size with a cylindrical lens. The TE₀ mode output was apertured with a narrow slit and recorded as a function of time for different power levels. Damage appeared as a broadening of the output mode in the plane of the waveguide and was recorded as a drop of the measured power. The short-term-damage threshold was arbitrarily defined as the coupled power at which the recorded output dropped by 10% in 1 min. Damage thresholds were slightly higher in annealed samples (annealing was performed by bringing the wafers up to about 250°C and letting them cool slowly over several hours). Initial measurements indicate a threshold of 13 mW in a nine-mode undoped LiNbO₃ waveguide and 10.5 mW in a three-mode MgO:LiNbO₃ waveguide. This corresponds to an intensity of 35 and 70 kW/cm², respectively, one of the highest intensities achieved to date in a LiNbO₃ waveguide at this wavelength and about 4 orders of magnitude higher than we observed in Ti-diffused LiNbO₃ guides.

It should be noted that the photorefractive sensitivity of a LiNbO₃ waveguide is a complicated function of the optical and thermochemical history of the device. The simple test described here is useful for the comparison of short-term damage effects in various waveguides, but it must be recognized that more-sensitive measurement techniques⁹ would undoubtedly find damage effects at intensities much lower than those reported here.

It was recently reported that proton-exchanged waveguides in undoped LiNbO₃ have significantly increased dark conductivity. This enhanced dark con-

ductivity probably accounts for the relatively small advantage of highly photoconductive MgO:LiNbO₃ over the undoped material for proton-exchanged waveguides.¹⁰ Further tests are now under way to improve the power tolerance of the waveguides.

In conclusion, we have characterized the parameters of proton-exchanged waveguides in high-damage-threshold MgO:LiNbO₃ and have compared these with waveguides made in undoped LiNbO₃. The major differences include a slower proton-diffusion rate and no etching of the y face in MgO:LiNbO₃ waveguides, as has also been observed in Ti-diffused, proton-exchanged waveguides. A factor-of-2 improvement in optical damage threshold (about 70 kW/cm²) at 0.5145 μ m was achieved in a proton-exchanged MgO-doped LiNbO₃ waveguide compared with a waveguide made of undoped LiNbO₃.

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References

1. G.-G. Zhong, in *Proceedings of 11th International Quantum Electronics Conference*, IEEE catalog no. 80, CH1561-0 (Optical Society of America, Washington, D.C., 1980), p. 631.
2. D. A. Bryan, R. Gerson, and H. E. Tomaschke, *Appl. Phys. Lett.* **44**, 847 (1984).
3. J. L. Jackel, C. E. Rice, and J. J. Veselka, *Appl. Phys. Lett.* **41**, 607 (1982).
4. M. DeMicheli, J. Botineau, S. Neveu, P. Sibillot, D. B. Ostrowsky, and M. Papuchon, *Opt. Lett.* **8**, 114 (1983).
5. J. L. Jackel, C. E. Rice, and J. J. Veselka, *Electron. Lett.* **19**, 387 (1983).
6. C. E. Rice and J. L. Jackel, *Mat. Res. Bull.* **19**, 591 (1984).
7. J. L. Jackel and C. E. Rice, *Proc. Soc. Photo-Opt. Instrum. Eng.* **460**, 43 (1984).
8. M. DeMicheli, J. Botineau, P. Sibillot, D. B. Ostrowski, and M. Papuchon, *Opt. Commun.* **42**, 101 (1982).
9. R. A. Becker, *Soc. Photo-Opt. Instrum. Eng.* **460**, 95 (1984).
10. R. A. Becker, *Proc. Soc. Photo-Opt. Instrum. Eng.* **517**, 194 (1984).