Photorefractive-damage-resistant
Zn-diffused waveguides in MgO:LiNbO₃

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Planar waveguides were fabricated by diffusing Zn into MgO:LiNbO₃ and LiNbO₃. Zn-diffused waveguides in MgO:LiNbO₃ guide both the ordinary and extraordinary polarizations and have propagation losses at 633 nm in the range of 0.4–1.2 dB/cm. Single-beam-induced in-plane scattering due to photorefractive damage was not observed at 515 nm up to intensities of as much as 90 kW/cm².

Ti diffusion and proton exchange are the two widely used techniques for fabricating optical waveguides in LiNbO₃. Ti diffusion is a high-temperature process (near 1000°C) that provides waveguides for both the ordinary and extraordinary polarizations in MgO:LiNbO₃ and LiNbO₃. Annealed proton exchange is a relatively low-temperature process (250–350°C) that produces waveguides that guide only the extraordinary polarization. Both types of waveguide exhibit low propagation losses at 633 nm, typically less than 0.5 dB/cm. Proton-exchanged waveguides in LiNbO₃ exhibit a fairly high resistance to photorefractive damage, but Ti-diffused waveguides in both LiNbO₃ and MgO:LiNbO₃ suffer from severe photorefractive damage even at wavelengths as long as 0.85 μm. The objective of this research was to develop a new fabrication process based on diffusion to produce low-loss optical waveguides that guide both polarizations and exhibit high resistance to photorefractive damage.

We targeted Zn as a likely dopant for several reasons. First, we observed that dopants such as Mg⁺⁺ and H⁺ reduce photorefractive damage, whereas Fe⁺⁺, Fe⁺⁺⁺, Nd⁺⁺, and Ti⁺⁺ increase photorefractive damage. We therefore speculated that a cation dopant with a single valence state less than or equal to two, such as Zn⁺⁺, may reduce photorefractive damage. Second, Zn has an ionic size (0.074 nm) comparable with that of the cation sites (Nb⁵⁺, 0.068 nm; Li⁺, 0.069 nm) and should not cause undue stress in the crystal. Before this research, ZnO films had been diffused into LiNbO₃ and were shown to increase the index of refraction (although whether it increased the ordinary or extraordinary index, or both, was not specified) without introducing absorption bands in the visible. It was also reported that the diffusion of metallic Zn in LiNbO₃ produced surface degradation that precluded the observation of guided modes. In this Letter we report the fabrication and characterization of low-loss, high-damage resistance Zn-diffused planar waveguides in MgO:LiNbO₃ that guide both the ordinary and extraordinary polarizations. Waveguides in LiNbO₃ substrates are also reported.

The Zn-diffused waveguides were fabricated by sputtering ZnO films with thicknesses between 30 and 100 nm onto LiNbO₃ substrates, followed by diffusion in a furnace with an alumina process tube. During diffusion, the samples were placed on platinum foil stands in a covered alumina boat filled with congruent LiNbO₃ powder, the purpose of which is to suppress the net transport of Li ions into or out of the LiNbO₃ crystal. The alumina boat was placed in a tube furnace for diffusion at elevated temperatures in dry air. The thermal cycle, carried out in dry air, typically involved heating at approximately 7°C/min up to the diffusion temperature of 1000–1100°C, diffusion for typically less than 1 h, and cooling at an initial rate of 10°C/min. The dimensions of the waveguide substrates were 20 mm × 6 mm × 1 mm for LiNbO₃ and 20 mm × 6 mm × 2 mm for MgO:LiNbO₃, and the samples used for diffusion studies were 6 mm × 4 mm × 1 mm.

The dopant source was found to have a strong influence on the surface quality of the waveguides. ZnO was chosen instead of metallic Zn to avoid any need for oxidation of the metal film, during which metastable phases in the Li–Zn–N–O system can form and degrade the surface quality. We found that surface roughening occurred if the thickness of ZnO exceeded some maximum value. The formation of intermediate compounds such as ZnLiNbO₄, which precipitated and adversely affected the surface, was observed by transmission electron microscopy on surfaces in which thick films of ZnO were diffused. This undesirable second-phase precipitation and the associated surface degradation were avoided by keeping the ZnO thickness below approximately 160 nm for MgO:LiNbO₃ and 100 nm for LiNbO₃.

Both dopant and refractive-index profiles in Zn-diffused waveguides were characterized. For sam-
Fig. 1. Extraordinary refractive-index profile and the Zn dopant profile of an x-cut MgO:LiNbO₃ substrate after diffusion of a 200-nm ZnO film at 1000°C for 10 h.

ples with diffusion depths larger than 10 μm, dopant profiles were measured by electron microprobe analysis (EMPA), and refractive-index profiles were measured by interference microscopy. For waveguide samples with a depth less than 10 μm, the refractive-index profiles were measured by prism coupling and IWKB analysis. In general, the dopant and refractive index exhibited similar profiles. This is illustrated in Fig. 1, which shows the extraordinary refractive-index and dopant profiles of a waveguide in x-cut MgO:LiNbO₃ fabricated by diffusing a 200-nm ZnO film for 10 h at 1000°C.

Diffusion coefficients were inferred from the dopant profiles in both x- and z-cut MgO:LiNbO₃ substrates. For x-cut material, we characterized two samples fabricated by diffusing a 200-nm ZnO film at 1000°C (10 h) and 1100°C (1 h). For z-cut material, three samples were characterized, with the following ZnO film thickness, diffusion temperatures and times: 1 μm, 950°C, 10 h; 160 nm, 1000°C, 10 h; and 1 μm, 1100°C, 1 h. Dopant profiles were measured by EMPA at three different locations on each of these five samples. The diffusion coefficients, inferred from fitting to a complement of error function, are 110 ± 20 μm²/h at 1100°C and 27 ± 4 μm²/h at 1000°C. Within the precision of these measurements, diffusion was found to be isotropic. Similar measurements in congruent LiNbO₃ showed that the diffusion coefficient is larger, 36.5 ± 4.5 μm²/h for z-cut material at 1000°C. For comparison, the diffusion coefficient of Ti in LiNbO₃ at 1050°C is 0.5 μm²/h.

The surface refractive-index change measured at 633 nm for a film thickness of 30 nm diffused at 1000°C for 30 min into an x-cut MgO:LiNbO₃ substrate was 0.0036 ± 0.0006 for the extraordinary index (Δnₑ). The ordinary index change (Δnₒ) was too small to be reliably measured by prism-coupling measurements but was estimated by interference microscopy to be approximately one fifth of Δnₑ, or approximately 0.0007. In z-cut congruent LiNbO₃, for a 100-nm film diffused at 1000°C for 30 min, Δnₑ is approximately 0.0034 ± 0.0006. Extraordinary modes in Zn-diffused LiNbO₃ waveguides were successfully excited by end firing but not by prism coupling. Interferometry showed that Δnₑ exhibits a complex, apparently nonmonotonic profile that depends on processing conditions, which we have not yet fully characterized.

The dependence of the index change on the Zn concentration, [Zn], was found by comparison of the dopant concentration and refractive index at different depths in a given sample (see Fig. 1). The extraordinary refractive-index change versus Zn concentration is shown in Fig. 2, which contains data from Fig. 1 and from three Zn-diffused x-cut MgO:LiNbO₃ waveguides fabricated at the same temperature, 1000°C. Within the resolution of the measurements the dependence is linear, with a slope of 1.4 × 10⁻³ per Zn mol. %.

Waveguide propagation losses were measured by comparing the transmission of the waveguide with the transmission of the substrate. 10× microscope objectives were used to couple the light from a 633-nm He–Ne laser into the planar waveguide and to image the output through a slit onto a diode detector. The output power was measured first with the laser beam coupled into the waveguide, then with the waveguide translated so that the focused laser beam passed through the substrate. The ratio of these two measurements provided an upper bound on the value of the propagation loss. These measurements were performed at 633 nm with the two waveguides whose indices were characterized above. In the Zn-diffused MgO:LiNbO₃ waveguide losses were 0.3 ± 0.1 dB/cm for the extraordinary modes and 0.8 ± 0.4 dB/cm for the ordinary modes. In the Zn-diffused LiNbO₃ waveguide these figures were 0.9 ± 0.1 and 0.5 ± 0.1 dB/cm, respectively. Further measurements are necessary to understand the dependence of the loss on the material and processing conditions. Nevertheless these initial results are encouraging, as the losses of Zn-diffused planar waveguides appear to be sufficiently low for many practical applications.

Fig. 2. Extraordinary refractive-index change versus Zn concentration. Data are obtained from the sample shown in Fig. 1 and from three other Zn-diffused x-cut MgO:LiNbO₃ waveguides fabricated at 1000°C. The solid line is the linear fit of the data.
Single-beam in-plane scattering was used to characterize the waveguide photorefractive damage. These measurements were conducted in x-cut samples, since damage-induced spreading of the signal occurs in the z direction. Light from a laser was launched into the waveguide by end firing, and the laser power gradually increased until spreading of the guided signal in the plane of the waveguide was observed. A useful semiquantitative measure of the photorefractive damage sensitivity can be obtained by projecting the prism-outcoupled beam onto a slit oriented perpendicular to the plane of the waveguide, adjusted to transmit 50% of the signal at low power, and recording the time dependence of the slit throughput as the power is incrementally increased. If damage occurs, spreading of the beam results in a decrease in the power transmitted through the slit.\(^{13}\)

These measurements were carried out in a Zn-diffused waveguide in MgO:LiNbO\(_3\) (30 nm, 1000°C, 30 min) at 515 nm using a cw Ar\(^+\) ion laser and the extraordinary-polarized mode. The laser was coupled into the waveguide with a cylindrical doublet (\(f = 127\) mm) followed by a 10\(\times\) objective, which produced an elliptical beam in the input plane of the waveguide, with \(1/e^2\) radii of 3 and 87 \(\mu\)m. The purpose of this elliptical beam was to reduce beam divergence in the plane of the waveguide so as to maintain a nearly constant intensity along the 19-mm length of the guide while still coupling efficiently (35%, corrected for Fresnel loss). Using this arrangement, the waveguide did not suffer from photorefractive damage for as much as 3 min at the maximum power available from the laser, 675 mW coupled into the waveguide. This corresponds to an approximate intensity of 90 kW/cm\(^2\). To increase this intensity, we also coupled the laser beam by end firing with a single 5\(\times\) microscope objective. The in-plane \(1/e^2\) radius of the beam was 7 \(\mu\)m at the input and 40 \(\mu\)m at the output of the waveguide. We still did not observe damage at a maximum intensity of 180 kW/cm\(^2\) at the input end of the waveguide. For comparison, in proton-exchanged waveguides under similar conditions the slit throughput decreased by 10% in 1 min at intensities of 35 kW/cm\(^2\) in congruent LiNbO\(_3\) and 70 kW/cm\(^2\) in MgO:LiNbO\(_3\), while Ti-diffused LiNbO\(_3\) waveguides exhibited damage at intensities of approximately 4 orders of magnitude lower.\(^{13}\) Zn-diffused waveguides in MgO:LiNbO\(_3\) therefore appear to be far more resistant to photorefractive damage than other waveguides reported to date in LiNbO\(_3\). Recent reports of damage-resistant Zn-doped LiNbO\(_3\) bulk crystals lend support to this observation.\(^{14}\)

In conclusion, planar waveguides were fabricated in both congruent and MgO-doped LiNbO\(_3\) by using Zn diffusion from a ZnO source. Proper selection of the thickness of ZnO eliminated second-phase precipitation and produced waveguides with good surface quality and losses in the range of 0.2–1.2 dB/cm at 633 nm. Diffusion of Zn in MgO:LiNbO\(_3\) is essentially isotropic with a diffusion coefficient of 140 \(\mu\)m\(^2\)/h at 1100°C, 2 orders of magnitude larger than that observed for Ti diffusion. Zn-diffused waveguides in MgO:LiNbO\(_3\) exhibited high resistance to photorefractive damage at 515 nm, with no measurable beam spreading in planar waveguides at the highest intensity available, 90 kW/cm\(^2\), which is the highest reported value for LiNbO\(_3\) waveguides at visible wavelengths. Planar waveguides were also fabricated successfully in congruent LiNbO\(_3\), and guiding was observed for both polarizations under the fabrication conditions tested.

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