Available online at www.sciencedirect.com
SCIENCE DIRECT®
ELSEVIER
Nuclear Instruments and Methods in Physics Research B 231 (2005) 394–399
NIM B Beam Interactions with Materials & Atoms
www.elsevier.com/locate/nimb

Proton beam writing of erbium-doped waveguide amplifiers

T.C. Sum [a,*], A.A. Bettiol [a], K. Liu [b], M.Q. Ren [a], E.Y.B. Pun [b], S. Venugopal Rao [a], J.A. van Kan [a], F. Watt [a]

[a] *Centre for Ion Beam Applications (CIBA), Department of Physics, National University of Singapore, 2 Science Drive 3, Singapore 117542, Singapore*
[b] *Department of Electronic Engineering, City University of Hong Kong, Tat Chee Avenue, Kowloon, Hong Kong*

Available online 17 March 2005

Abstract

Buried channel waveguide amplifiers in Er^{3+}–Yb^{3+} co-doped phosphate glasses were fabricated by proton beam writing using a focused sub-micron beam of 2.0 MeV protons with a fluence ranging from $0.5–6.0 \times 10^{15}$ particles/cm^2. The waveguides were located at a depth of ~38 μm beneath the surface. Above a threshold fluence of 3.0×10^{15} particles/cm^2, a negative refractive index change occurs, preventing any light confinement in the channel. A peak net gain of ~1.57 dB/cm was measured for waveguides fabricated with a fluence of $\sim 0.9 \times 10^{15}$ particles/cm^2. These measurements were performed at 1.534 μm signal wavelength, with 100 mW pump power at 975 nm wavelength.
© 2005 Elsevier B.V. All rights reserved.

PACS: 07.78.+s; 42.65.Yj; 42.70.Jk; 42.82.Cr; 42.82.Et

Keywords: Proton beam writing; Buried channel waveguides; Erbium; Ytterbium; Phosphate glass; IOG-1; Waveguide amplifier

1. Introduction

In the last few years, erbium- and erbium/ytterbium-doped glasses have received much attention due to their applications as amplifiers in optical telecommunications and as compact light sources for eye safe range-finding in the 1.55 μm spectral range [1]. Erbium-doped waveguide amplifier (EDWA) technology is commonly used in telecommunications networks to compensate for signal loss. It is replacing erbium-doped fiber amplifiers as it has a much smaller footprint and hence is more cost-effective. EDWA has applications as receivers, transmitters and in-line amplifiers. Many intensive studies on the fabrication of optical amplifiers using various techniques, such as ion-exchange [2], radio frequency sputtering [3], sol–gel [4], and plasma-enhanced chemical vapor deposi-

* Corresponding author. Tel.: +65 6874 2817; fax: +65 6777 6126.
E-mail address: phystc@nus.edu.sg (T.C. Sum).

0168-583X/$ - see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.nimb.2005.01.089

tion [5], have been motivated by the need of integrated optics to fabricate both active and passive components on a small substrate. A comparison shows that these techniques demonstrate different formations and qualities of the waveguides. van den Hoven et al. [6] reported the optical gain performance of Er-implanted Al_2O_3 waveguide amplifiers at 1.53 μm. Osellane [7] presented gain characteristics in erbium/ytterbium (Er^{3+}/Yb^{3+}) co-doped waveguides fabricated by femtosecond laser pulses.

Ion beam irradiation (using various ion species) have been used extensively to fabricate planar waveguides in many optical materials [8–10], including glasses. Many of the irradiations were simply 'blanket' irradiations of the sample or were performed with the aid of a mask to define the waveguides. Direct-write of waveguides in fused silica using focused ion beam irradiation have also been previously reported [11–13]. The main advantage of direct-writing of waveguides is that complex prototype optical circuits can be quickly realized without the need for a mask. However, in these works, only passive waveguides were fabricated. There are few reported studies on the optical effects of ion implantation in Er^{3+}/Yb^{3+} co-doped phosphate glass [8,9].

In this paper, the experimental procedures of proton beam writing (PBW) of Er^{3+}/Yb^{3+} co-doped phosphate glass (IOG-1) and the waveguide formation mechanism in this material is reported and discussed. In our earlier paper [14], we emphasized the optical characterization of the phosphate glass (IOG-1) waveguide amplifiers fabricated using PBW [15]. PBW uses a focused sub-micron beam of high energy protons to direct-write on a suitable material, such as polymers, phosphate glass, fused silica etc. In this work, a proton beam (2.0 MeV) was used to modify the optical properties of the active phosphate with fluences ranging from $0.5–6.0 \times 10^{15}$ particles/cm^2. The maximum amount of modification is expected to occur near the end-of-range region, where the maximum amount of energy is deposited, resulting in an increase in the refractive index (i.e. positive refractive index change, $+\Delta n$). In this manner, a weakly guiding buried channel waveguide can be formed. It is important to realize that the waveguide formation mechanism in this work differs from that by Zhou et al. and Chen et al. [8,9], where they have used much higher ion fluence (typically $\sim 10^{16}$ particles/cm^2) to create a low index optical 'barrier' at the end-of-range (i.e. $-\Delta n$). The light confinement in optical barrier waveguides takes place in the region between the low index barrier and the surface of the waveguide.

2. Experimental

IOG-1 Phosphate laser glass (*Schott Glass Technologies Inc.*) is a chemically durable, sodium–alumino–phosphate glass developed for use in both active and passive waveguide devices. Typically, these waveguides are fabricated by ion-exchange in a molten KNO_3 or $AgNO_3$ salt bath. The phosphate glass is also co-doped with 2.16 wt% of Er_2O_3 and 4.70 wt% of Yb_2O_3. Though the IOG-1 phosphate glass is commercially available, the exact composition and the atomic concentrations of the constituent elements are not known. It is important to know this information as one of the crucial steps in PBW is the dose normalization procedure [16]. Knowledge of the composition and the atomic concentration is important for the accurate fitting of the Rutherford backscattering spectrometry (RBS) spectrum and hence an accurate determination of the proton fluence used in the waveguide fabrication. One solution to this dilemma is to employ the proton-induced X-ray emission (PIXE) technique to determine the composition and the atomic concentration of the respective elements in the IOG-1 phosphate glass. This atomic concentration was used for fitting the RBS spectrum of the sample.

PBW was carried out using proton beams from the High Voltage Engineering Europa (HVEE) 3.5 MeV Singletron^{TM} accelerator at the Centre for Ion Beam Applications (CIBA), Department of Physics, National University of Singapore. The IOG-1 samples of dimensions approximately 1 cm × 1 cm × 1.6 mm (length × breadth × width) were edge-polished prior to the irradiation. The linear waveguides were direct-written using a 2.0 MeV beam of protons with a beam spot size of approximately 1.0 μm and fluences ranging from $0.5–6.0 \times 10^{15}$ particles/cm^2. The proton beam was magnetically-scanned over a distance of 5 μm in one direction while, simultaneously the stage was traversed perpendicular to this magnetic scan direction at a speed of ~5 μm/s. Multiple stage scanning loops were performed to ensure an 'even' irradiation along the whole length of the waveguide. A description of the dose normalization process and the fabrication process can be found in [14,16] respectively.

After the PBW, the waveguides were thermally annealed in a furnace at a temperature of 220 °C for 30 min to reduce the waveguide propagation losses. Annealing helps to remove the defects arising from the electronic (inelastic) and nuclear (elastic) interactions of the incident ions and substrate atoms [17]. The average propagation loss of the waveguides was reduced from ~3.2 to ~0.8 dB/cm [14]. After the annealing process, waveguide characterization experiments to study the near field mode profiles and to measure the net optical gain were performed. The signal gain at 1.534 μm was measured by pumping the waveguide with a 975 nm laser diode. The input signal source was a HP8161A tunable laser, and a 980 nm/1550 nm wavelength division multiplexing coupler was used to combine both signal and pump light. An optical spectrum analyzer was used to record the optical gain and the amplified spontaneous emission spectrum. A more detailed description of these measurements can be found in [14].

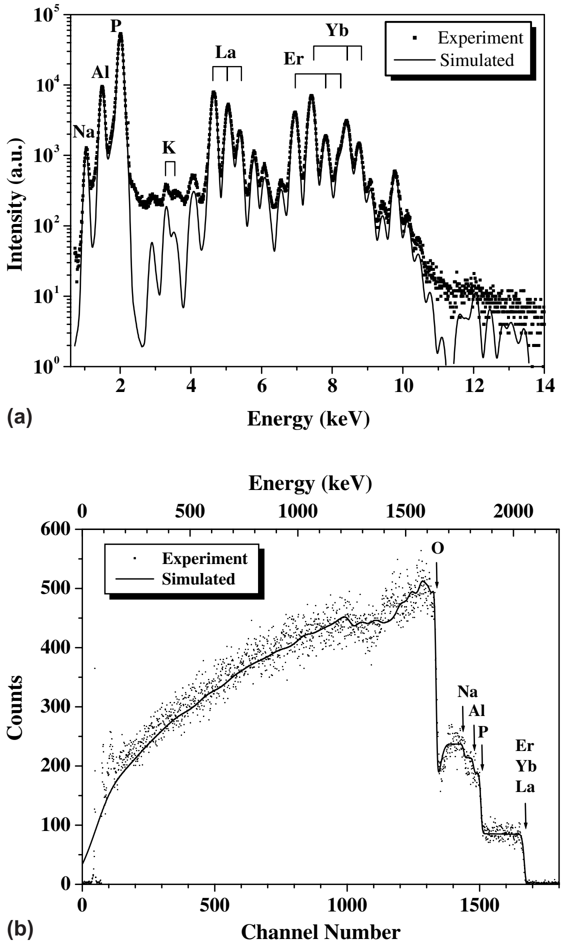

Fig. 1. (a) PIXE spectrum of the IOG-1 glass sample (co-doped with 2.16 wt% of Er_2O_3 and 4.70wt% of Yb_2O_3), (b) RBS spectrum of the sample.

3. Results and discussion

Fig. 1(a) shows the PIXE spectrum of the IOG-1 phosphate glass fitted using Oxford Microbeams DAN32 and Fig. 1(b) shows the RBS spectra of the IOG-1 phosphate glass fitted using SIMNRA. The atomic concentration of the glass sample determined from the PIXE measurements is given in Table 1. Fig. 2 shows a top view of a pair of linear waveguides and a cross-sectional view of a waveguide end-face is given in the inset.

Irradiation with protons leads to the breaking of P–O bonds due to nuclear damage in the phosphate glasses. From TRIM simulations (see Fig. 3), the bulk of the nuclear damage to the material is concentrated near the end of the proton range (i.e. ~38 μm deep), in the form of phosphorus and oxygen vacancies. In addition to the damage caused by nuclear stopping, ionization (electronic stopping – see Fig. 3) also causes damage in a material. Matsunami et al. [18] reported on the O–H bond breaking (on the basis of electronic energy deposition by ions at low fluence – i.e. $<10^{17}$ particles/cm^2) and at higher fluence (i.e. $>10^{17}$ particles/cm^2) using 120 keV protons, the reduction of PO_4 tetrahedron to P colloid overcomes O–H bond breaking due to the interaction

Table 1
Atomic concentration of IOG-1 phosphate glass deduced from PIXE measurements

Element	Atomic concentration
Phosphorous	0.20930
Oxygen	0.62183
Aluminium	0.07094
Sodium	0.08880
Erbium	0.00321
Ytterbium	0.00315
Lanthanum	0.00277

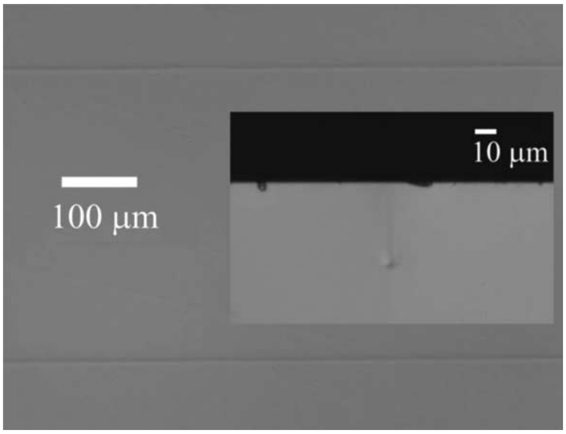

Fig. 2. Differential interference contrast (DIC) image of the top-view of a pair of proton beam written waveguides. Inset shows a DIC image of the end-face of one of the waveguides.

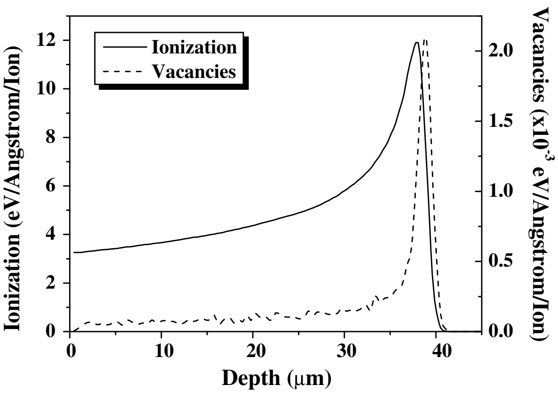

Fig. 3. TRIM simulation results for 2.0 MeV protons in IOG-1 phosphate glass.

of the irradiated H^+ with the substrate, resulting in the formation of P–OH and H_2O. These ion-induced rearrangements of substrate bonds and the chemical interactions of ions with the substrate will bring about the modification of the optical properties of phosphate glass. Near field mode profiles of a channel waveguide fabricated with a fluence of $\sim 1 \times 10^{15}$ particles/cm^2 are given in Fig. 4. The mode is located at a depth of ~38 μm, near the end-of-range. This suggests that a region of increased refractive index (i.e. $+\Delta n$) has been formed near the end-of-range, thus permitting

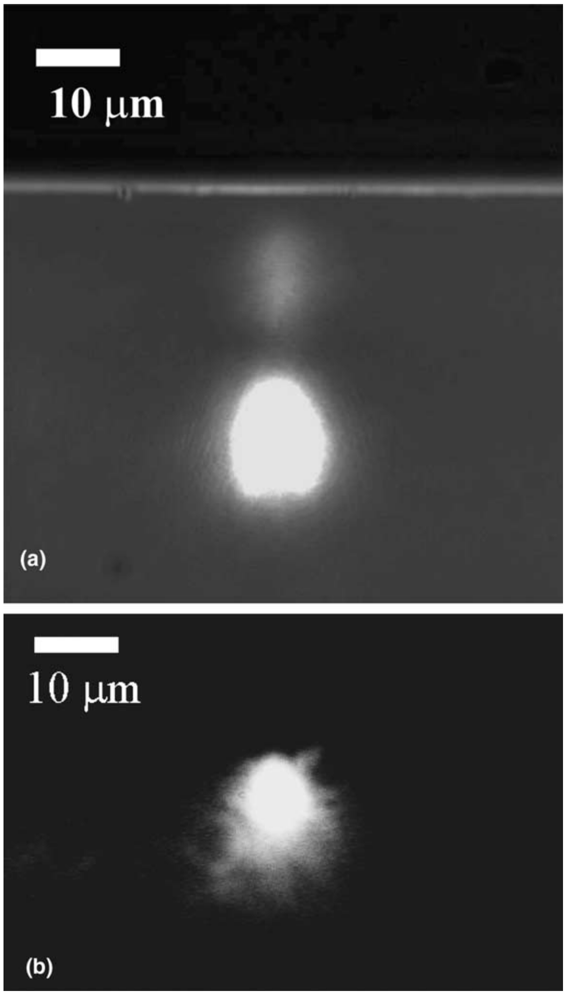

Fig. 4. Near-field mode profiles of a waveguide fabricated with a fluence of 1×10^{15} particles/cm^2 at a wavelength of (a) 632.8 nm and (b) 1550 nm.

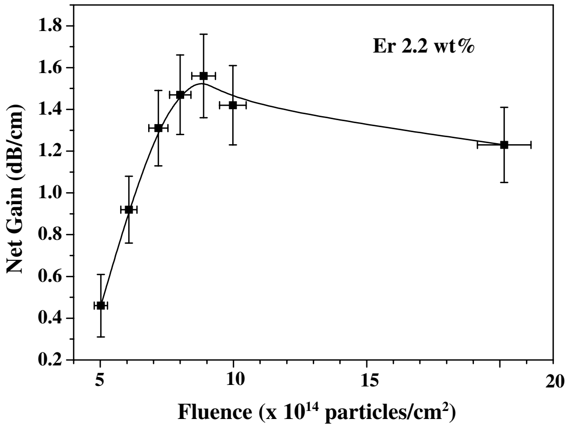

Fig. 5. Graph of net optical gain against the proton fluence used in the fabrication.

light confinement laterally and transversely. Such observation also indicates that the waveguide formation mechanism is different from that reported by Zhou et al. and Chen et al. [8,9] where ion implantation was used to form an optical 'barrier' (i.e. a region of decreased refractive index, $-\Delta n$) for light confinement. For waveguides fabricated with a fluence higher than 3×10^{15} particles/cm^2, no confinement of light within the channel was observed at 632.8 nm or 1550 nm wavelengths. It is believed that the threshold fluence has been exceeded, resulting in a negative refractive index change, thus forming an optical 'barrier' of lower refractive index as discussed earlier.

For waveguide amplifiers fabricated using phosphate glass co-doped with 2.16 wt% of Er_2O_3 and 4.70 wt% of Yb_2O_3, a maximum net gain of ~1.57 dB/cm was measured at 1.534 μm signal wavelength, with 100 mW pump power at 975 nm wavelength. Fig. 5 shows a plot of the net gain as a function of the proton fluence used in the fabrication. As the fluence increases, the net gain increases until a peak net gain of ~1.57 dB/cm was measured for waveguides fabricated with a fluence of $\sim 0.9 \times 10^{15}$ particles/cm^2.

4. Conclusions

PBW is an extremely versatile technique for the rapid prototyping of optical circuits. Buried channel waveguide amplifiers were fabricated using 2.0 MeV protons, with a fluence ranging i.e., (from $0.5 - 6.0 \times 10^{15}$ particles/cm^2). The waveguides were formed at a depth of ~38 μm. For a fluence $>3.0 \times 10^{15}$ particles/cm^2, no confinement of the light at 632.8 nm or 1550 nm wavelengths within the channel is possible, indicating $-\Delta n$ has occurred. For IOG-1 phosphate glass (co-doped with 2.16 wt% of Er_2O_3 and 4.70 wt% of Yb_2O_3), a maximum net gain of ~1.57 dB/cm was measured for the waveguide amplifiers at 1.534 μm signal wavelength, with 100 mW pump power at 975 nm wavelength. Further work is currently in progress on the investigation of the waveguide end-faces using an atomic force microscope and the recovery of the refractive index profile of the waveguides using the propagation-mode near-field method, which will give one a better understanding of the waveguide formation mechanism.

Acknowledgements

The work was supported by a Defence Innovative Research Program grant from the Defence Science and Technology Agency, Singapore, a grant from the Agency for Science Technology and Research, Singapore, as well as the Research Grants Council of the Hong Kong Special Administration Region, People's Republic of China, under grant project no. CityU 1293/03E.

References

[1] B.C. Hwang, S. Jiang, T. Luo, J. Watson, S. Honkanen, Y. Hu, F. Smektala, J. Lucas, N. Peyghambarian, Electron. Lett. 35 (1999) 1007.
[2] S.F. Wong, E.Y.B. Pun, P.S. Chung, IEEE Photonics Technol. Lett. 14 (2002) 80.
[3] R. Schermer, W. Berglund, C. Ford, R. Ramberg, A. Gopinath, IEEE J. Quantum Electron. 39 (2003) 154.
[4] W. Huang, R.R.A. Syms, E.M. Yeatman, M.M. Ahmad, T.V. Clapp, S.M. Ojha, IEEE Photonics Technol. Lett. 14 (2002) 959.
[5] S. Guldberg-Kjaer, J. Hübner, M. Kristensen, C. Laurent-Lund, M. Rysholt Poulsen, M.W. Sckerl, Electron. Lett. 35 (1999) 302.
[6] G.N. van den Hoven, R.J.I.M. Koper, A. Polman, C. van Dam, J.W.M. van Uffelen, M.K. Smit, Appl. Phys. Lett. 68 (1996) 1886.

[7] R. Oscellame, S. Taccheo, G. Cerullo, M. Marangoni, D. Polli, R. Ramponi, P. Laporta, S. De Silvestri, Electron. Lett. 38 (2002) 964.
[8] Z. Zhou, E.Y.B. Pun, S.F. Wong, Y. Zhou, J. Mater. Res. 16 (2001) 1135.
[9] F. Chen, X.L. Wang, X.S. Li, L.L. Hu, Q.M. Lu, K.M. Wang, B.R. Shi, D.Y. Shen, Appl. Surf. Sci. 193 (2002) 92.
[10] D.M. Ruck, S. Brunner, K. Tinschert, W.F.X. Frank, Nucl. Instr. and Meth. B 106 (1995) 447.
[11] A. Roberts, M.L. von Bibra, J. Lightwave Technol. 14 (1996) 2554.
[12] M.L. von Bibra, A. Roberts, S.D. Dods, Nucl. Instr. and Meth. B 168 (2000) 47.
[13] M. Hattori, Y. Ohki, M. Fujimaki, T. Souno, H. Nishikawa, E. Watanabe, M. Oikawa, T. Kamiya, K. Arakawa, Nucl. Instr. and Meth. B 210 (2003) 272.
[14] K. Liu, E.Y.B. Pun, T.C. Sum, A.A. Bettiol, J.A. van Kan, F. Watt, Appl. Phys. Lett. 84 (2004) 684.
[15] F. Watt, J.A. van Kan, T. Osipowicz, MRS Bull. 25 (2000) 33.
[16] A.A. Bettiol, J.A. van Kan, T.C. Sum, F. Watt, Nucl. Instr. and Meth. B 181 (2001) 49.
[17] P.D. Townsend, P.J. Chandler, L. Zhang, Optical Effects of Ion Implantation, vol. 13, Cambridge University Press, 1994.
[18] N. Matsunami, K. Kawamura, H. Hosono, Radiat. Eff. Defects Solids 136 (1995) 311.