TU/e EINDHOVEN UNIVERSITY OF TECHNOLOGY

Erbium ion implantation doping of opto-electronic materials operating at 1.5 mu m

Citation for published version (APA):
Polman, A., Snoeks, E., Hoven, van den, G. N., Custer, J. S., Hendriksen, B., Diemeer, M. B. J., Uffelen, van, J. W. M., Oei, Y. S., Smit, M. K., Fleuster, M., & Buchal, C. (1993). Erbium ion implantation doping of opto-electronic materials operating at 1.5 mu m. In *19th European Conference on Optical Communication : ECOC '93* (pp. 61-64). Swiss Electrotechical Association.

Document status and date:
Published: 01/01/1993

Document Version:
Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

• A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
• The final author version and the galley proof are versions of the publication after peer review.
• The final published version features the final layout of the paper including the volume, issue and page numbers.

Link to publication

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

• Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
• You may not further distribute the material or use it for any profit-making activity or commercial gain
• You may freely distribute the URL identifying the publication in the public portal.

If the publication is distributed under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license above, please follow below link for the End User Agreement:

www.tue.nl/taverne

Take down policy
If you believe that this document breaches copyright please contact us at:

openaccess@tue.nl

providing details and we will investigate your claim.

Download date: 04. Oct. 2023

Erbium Ion Implantation Doping of Opto-Electronic Materials Operating at 1.5 μm

A. Polman, E. Snoeks, G.N. van den Hoven, J.S. Custer,
FOM-Institute AMOLF, Kruislaan 407, NL-1098 SJ Amsterdam, The Netherlands,
B. Hendriksen, M.B.J. Diemeer,
PTT Research, P.O. Box 421, NL-2260 AK Leidschendam, The Netherlands,
J.W.M. van Uffelen, Y.S. Oei, M.K. Smit,
Delft University of Technology, Mekelweg 4, NL-2628 CD Delft, The Netherlands,
M. Fleuster, Ch. Buchal,
ISI, KFA Jülich, Forschungszentrum Jülich, Postfach 1913, D-5170 Jülich, Germany

Soda-lime silicate and Al_2O_3 waveguide films, $LiNbO_3$ single crystal, as well as crystal Si are doped with erbium by ion implantation. All materials show luminescence at 1.5 μm, characteristic for Er, with lifetimes up to 12 ms.

1. INTRODUCTION

Thin films of opto-electronic materials, doped with erbium, can find important applications in planar integrated optical technology. Erbium, when incorporated in the trivalent state, exhibits intra-4*f* luminescence around 1.5 μm, a standard telecommunication wavelength. If planar optical waveguides could be doped with Er, active devices such as planar waveguide lasers and amplifiers can be fabricated. When an optically active material such as $LiNbO_3$ is doped with Er, Q-switched and tuneable planar waveguide lasers may be developed. In silicon, Er doping may create a solution around the problem of the indirect bandgap which precludes efficient light emission from Si. If high Er concentrations can be incorporated and activated in Si, Si-based optical devices operating at 1.5 μm may be fabricated.

Ion implantation is a technique which is ideally suited for the local doping of thin films with Er. Using MeV energies, the ion range is in the order of microns, the typical dimension of planar waveguides. The Er depth and concentration profile can be easily controlled with the Er ion energy and fluence. Because implantation is a non-equilibrium technique, novel structures and metastable solutions may be fabricated. Moreover, ion implantation, a standard technique in the semiconductor industry, is compatible with other optical device processing steps.

In this paper we discuss the use of ion implantation for the optical doping with Er of soda-lime silicate and Al_2O_3 waveguide films, $LiNbO_3$ and Si. The implantation procedures are described, photoluminescence spectroscopy data are given, and applications in planar integrated optical devices are discussed.

2. EXPERIMENTAL

Four different substrate materials were used:
1) commercially available soda-lime silicate glass (Fisher Premium), 1 mm thick,
2) polycrystalline Al_2O_3 films, 600 nm thick, deposited onto thermally oxidized Si substrates using radio-frequency magnetron sputtering,
3) commercially available $LiNbO_3$ samples (Crystal Technology), *x*-cut, 1 mm thick, and
4) Czochralski-grown *p*-type (100)Si wafers with a resistivity of 1 Ωcm.

Ion implantation was performed using a 1 MV van de Graaff accelerator or a 1.7 MV tandem accelerator, with Er ions produced in a sputter ion source. In the silica samples, channel waveguides were fabricated by using a $Na^+ \leftrightarrow K^+$ ion exchange process, followed by the deposition of a polymer cladding layer. In Al_2O_3, channel waveguides were fabricated by etching ridges using reactive ion etching, followed by deposition of a SiO_2 cladding layer.

Thermal annealing in vacuum was performed on some samples using a standard tube furnace. Erbium concentration profiles as a function of depth were determined using Rutherford Backscattering Spectrometry (RBS) using a 2 MeV He beam. Photoluminescence (PL) spectroscopy was performed using an Ar^+ laser as a pump source. Some measurements were performed in a liquid nitrogen-cooled cryostat. PL decay measurements were also performed.

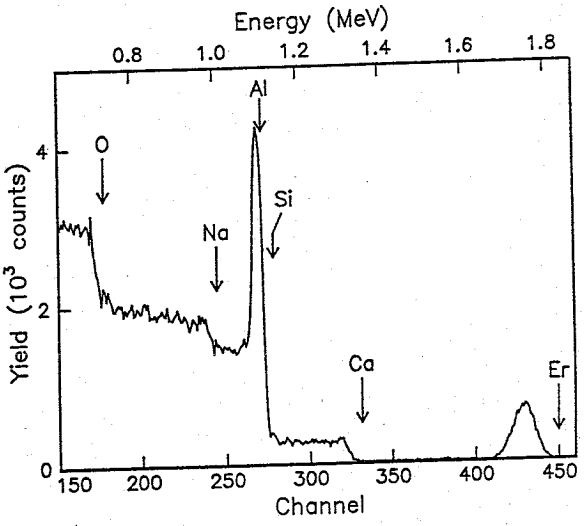

Fig. 1 RBS spectrum of Er-implanted (500 keV, $5.4x10^{15}$ cm^{-2}) soda-lime silicate glass.

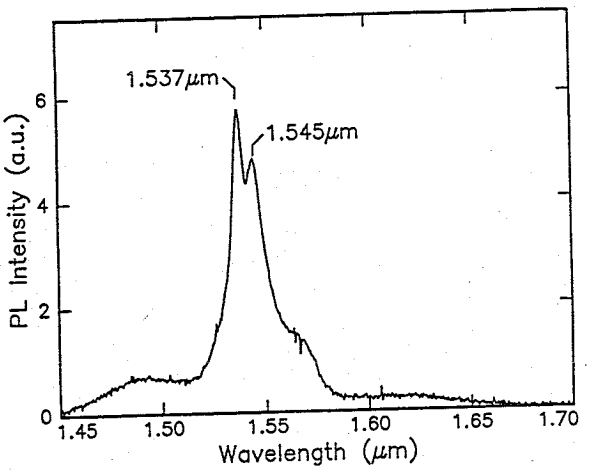

Fig. 2 Room-temperature PL spectrum of Er-implanted soda-lime silicate glass (500 keV, $3.7x10^{15}$ cm^{-2}), annealed at 512 °C.

3. RESULTS

3.1 Soda-lime silicate glass

Figure 1 shows an RBS spectrum of Er-implanted soda-lime silicate glass (500 keV, $5.4x10^{15}$ cm^{-2}). The spectrum shows the different elements in the multi-component glass, as well as the 420 Å thick Al coating used during implantation to avoid charging. In addition, the Er implantation profile is shown. It is nearly Gaussian shaped, peaks at a depth of 100 nm, with a full width at half-maximum (FWHM) of 90 nm. The Er peak concentration is 0.6 at.%.

Figure 2 shows a PL spectrum of an Er-implanted (500 keV, $3.7x10^{15}$ cm^{-2}) sample after thermal annealing at 512 °C for 1 hour. Annealing is necessary to annihilate the implantation-induced silica network defects which can partly quench the luminescence.[1] The spectrum shows peaks at 1.537 and 1.545 μm and broad shoulders extending from roughly 1.45 to 1.67 μm. This emission is characteristic for the intra-4*f* transitions between the $^4I_{13/2}$ and $^4I_{15/2}$ manifolds of Er^{3+}.[2] The peak structure is attributed to Stark splitting of the degenerate 4*f* levels. This, as well as inhomogeneous and homogeneous broadening leads to a FWHM of 19 nm.

In Fig. 3, the PL peak intensity as well as the PL lifetime are plotted against the Er fluence and peak concentration. The PL intensity first increases linearly with Er fluence, but levels off for higher concentrations. At the same time, the PL lifetime decreases with fluence. The saturation in PL intensity is explained by an increase in the contribution of non-radiative decay at high Er fluences.[1] This is caused by concentration quenching or additional defects present in the glass after high-fluence implantation. These data put an upper limit to the concentration of Er which can be usefully incorporated in soda-lime silicate glass by ion implantation: 0.4 at.%. Er-implanted channel waveguides (Er peak concentration 0.4 at.%), showed the same optical losses (measured at 1.3 μm) as unimplanted guides: ≈ 1 dB/cm. A simple gain estimate shows that, by using the proper combination of Er implantation energies and fluences, a net optical gain in the order of 0.7 dB/cm may be achieved.

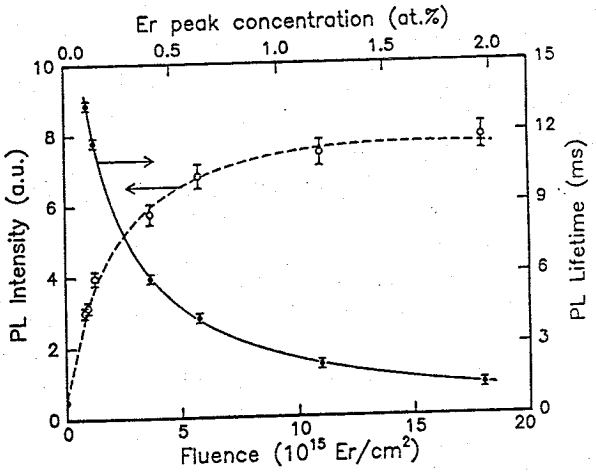

Fig. 3 PL peak intensity and lifetime as a function of Er fluence for Er-implanted (500 keV) soda-lime silicate glass, measured at room-temperature.

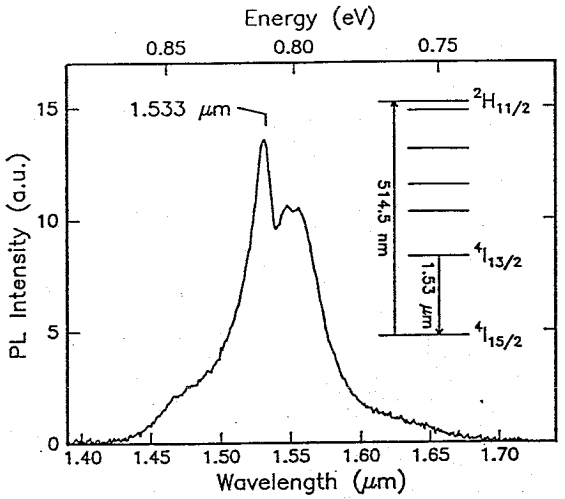

Fig. 4 Room-temperature PL spectrum of an Er-implanted (800 keV, 2.3x10^{15} cm^{-2}) Al_2O_3 film, annealed at 950 °C.

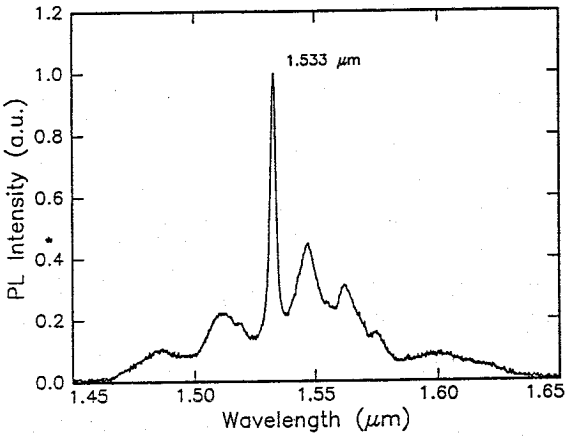

Fig. 5 Room-temperature PL spectrum of Er-implanted $LiNbO_3$ (3.5 MeV, 7.5x10^{15} cm^{-2}), annealed at 1060 °C.

3.2. Al_2O_3

A PL spectrum for an Er-implanted Al_2O_3 film (800 keV, 2.3x10^{15} Er/cm^2, annealed at 950 °C for 1 hr.) is shown in Fig. 4. The Er peak concentration in this sample was 0.23 at.%. The spectrum peaks at 1.533 μm and is quite broad (55 nm FWHM). Thermal annealing is necessary to optimize the PL intensity: a very low intensity is observed from the as-implanted sample, it increases on annealing for temperatures up to at least 950 °C.[3] The increase is interpreted in terms of defect annealing, leading to a decrease in the number of non-radiative decay paths as well as an increase in Er active fraction. Channel waveguides fabricated in unimplanted films show a minimum loss (measured at 1.5 μm) as low as 0.35 dB/cm after annealing at 825 °C.[4]

Measurements of the PL intensity as a function of Er fluence show a near-linear increase for concentrations up to 1 at.% Er.[3] The PL lifetime decreases from 8 to 4 ms on increasing the concentration from 0.03 to 1 at.%. An increase in PL lifetime is observed for even higher Er concentrations (3 at.%) and may be associated to structural changes in the Al_2O_3 film after high-fluence implantation.

3.3 $LiNbO_3$

A PL spectrum for Er-implanted $LiNbO_3$ (3.5 MeV, 7x10^{15} cm^{-2}, annealed at 1060 °C for 15 min.) is shown in Fig. 5. A well-defined structure is observed with clear peaks in the wavelength range 1.45-1.65 μm. Measurements at 77 K show that all peaks can be clearly separated, indicating that the width of the lines in Fig. 5 is to a large extent determined by homogeneous broadening. A PL spectrum was also taken from a $LiNbO_3$ sample doped with Er during growth from the melt. It was essentially the same as that for the Er-implanted sample in Fig. 5. The PL lifetime at 1.533 μm is 2.9 ms. Thermal annealing is necessary to obtain luminescence from Er-implanted $LiNbO_3$: no signal was observed for the as-implanted sample. An interesting observation is that 15 min. annealing at 1060 °C is sufficient to optimize the PL intensity and lifetime, while a much longer time (up to 8 hours) is necessary to remove the structural damage in the $LiNbO_3$ crystal as detected by RBS/channeling.[5]

3.4 Si

Crystal Si was implanted at 77 K with 9x10^{14} Er/cm^2 at 250 keV. Implantation causes amorphization of a 160 nm thick surface layer. Solid-phase epitaxial recrystallization was performed using thermal annealing at 600 °C for 15 min. During recrystallization, Er segregation and trapping occurred at the moving amorphous/crystal interface with Er concentrations up to 9x10^{19} cm^{-3} being trapped in single-crystal Si.[6]

Figure 6 shows luminescence spectra of the Er-doped crystal in two pump geometries: from the front and from the back. The 1.5 μm luminescence characteristic of Er is observed when the sample is pumped from the front. This may originate from direct absorption by the Er or by a photocarrier-mediated process. The first process is excluded in the second

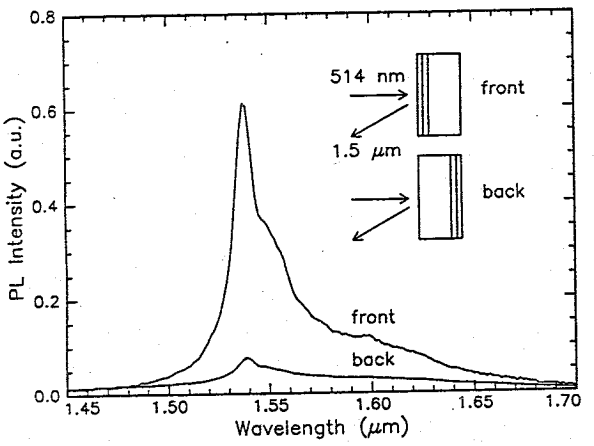

Fig. 6 PL spectra at 77 K of Er-doped crystal Si, measured using two different pump geometries (see text).

geometry, in which the green pump is fully absorbed in the top few μm of the backside. In that geometry the luminescence at 1.5 μm is also observed, indicating that some of the carriers generated at the backside diffuse through the wafer and cause excitation of the Er implanted at the front. This is a clear indication for a photocarrier-induced process. The PL lifetime at 1.535 μm is roughly 1 ms.

4. DISCUSSION

The previous data show that ion implantation is a useful technique for the incorporation of erbium in a wide class of materials. The main advantage of implantation over other techniques is that higher Er concentrations can be incorporated, and the Gaussian Er depth profile can be adjusted to the optical mode profile in a planar waveguide. The latter is not possible with a conventional technique such as drive-in diffusion. Moreover, Er shows extremely small diffusivity in many materials (Si, SiO_2, Al_2O_3). Alternative techniques for the fabrication of Er-doped thin films have only recently become available and include flame hydrolysis,[7] spray pyrolysis,[8] and sputter deposition.[9]

Concentration quenching and implantation-related defects limit the Er concentration which can be usefully incorporated in soda-lime silicate glass to 0.4 at.%. In Al_2O_3, Er concentrations as high as 3 at.% can be reached and the PL spectrum is relatively broad with only limited structure. This reflects an inhomogeneous site distribution of the Er ions. This may be related to the fact that relatively low annealing temperatures were used in order to maintain the excellent optical quality of the films, or to the polycrystalline structure of the films. The concentration or defect quenching effects are less pronounced in Al_2O_3 than in silica. Possible reasons for this include: different susceptibilities to ion damage, different defect annealing characteristics, or weaker Er-Er interactions because of the larger Stark splitting in Al_2O_3.

The PL line structure, and therefore Er site, is much more well-defined in Er-implanted $LiNbO_3$, than in Al_2O_3. Note that the $LiNbO_3$ was annealed to a much higher temperature (relative to the melting point) than the Al_2O_3 films.

Finally, Er doping of Si seems an interesting way around the problem of the indirect bandgap, which precludes efficient light emission from Si. High concentrations of Er can be incorporated in single-crystal Si and the challenge now lies in full activation of the Er, and the attainment of room-temperature (electro-) luminescence.

5. CONCLUSION

The incorporation of erbium into a wide class of optical materials is investigated using ion implantation. Soda-lime silicate and Al_2O_3 waveguide films, $LiNbO_3$ single crystal, as well as crystal Si are successfully doped with optically active Er. Each materials shows a characteristic PL spectrum, and luminescence lifetimes range from 1 to 12 ms. The implanted films may find important applications in integrated optical technology.

Acknowledgements

This work is part of the research programme of FOM and made possible by financial support from NWO, STW and IOP Electro-Optics

References

1 E. Snoeks, G.N. van den Hoven and A. Polman, J. Appl. Phys, in press.
2 S. Hüfner, *Optical Spectra of Transparent Rare-Earth Compounds* (Academic, New York, 1978).
3 G.N. van den Hoven, E. Snoeks, A. Polman, J.W.M. van Uffelen, Y.S. Oei, and M.K. Smit, Appl. Phys. Lett., in press.
4 M.K. Smit, G.A. Acket, and C.J. van der Laan, Thin Solid Films, Electron. and Optics **138**, 171 (1986).
5 M. Fleuster, Ch. Buchal, E. Snoeks, and A. Polman, to be published.
6 A. Polman, J.S. Custer, E. Snoeks, and G.N. van den Hoven, Appl. Phys. Lett. **62**, 507 (1993).
7 T. Kitagawa et al., Electron. Lett. **28**, 1819 (1992).
8 T.H. Hoekstra et al., Optics. Lett. **17**, 1506 (1992).
9 J. Shmulovich et al., Electr. Lett. **28**, 1181 (1992).