CONF-960994--14

Note: This is a preprint of a paper being submitted for publication. Contents of this paper should not be quoted nor referred to without permission of the author(s).

To be submitted to *Nucl. Inst. & Methods B*

**Loss Measurements and Stoichiometric Dependence
of Ti and O Implanted LiNbO₃ Waveguides**

E. K. Williams, D. Ila, S. Sarkisov, and P. Venkateswarlu
Alabama A&M University
Normal, AL

D. B. Poker and D. K. Hensley
Oak Ridge National Laboratory
Oak Ridge, TN

RECEIVED
NOV 06 1996
OSTI

"The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-96OR22464. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes."

MASTER

Prepared by the
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-96OR22464

October 1996

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

LM

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Loss Measurements and Stoichiometric Dependence of Ti and O Implanted LiNbO₃ Waveguides

E. K. Williams^a, D. Ila^{a*}, S. Sarkisov^b, P. Venkateswarlu^b, D. B. Poker^c and D. K. Hensley^c

^aCenter for Irradiation of Materials, P.O. Box 1447, Alabama A&M University, Normal, AL 35762-1447, U.S.A.

^bCenter for Nonlinear Optics, Alabama A&M University, Normal, AL 35762, U.S.A.

^cSolid State Division of Oak Ridge National Laboratory, P.O. Box 2008, MS 6048, Oak Ridge, TN 37831, U.S.A.

ABSTRACT

Planar waveguides created by the implantation at 500°C of 2.5×10^{17} Ti ions/cm² and 2.5, 5.0 and 7.5×10^{17} O ions/cm² have been characterized for loss by the scattered light and cutback techniques. Results indicate losses of less than 2.5 dB/cm to 3 dB/cm for waveguides with a Ti:O ratio of 1:3 and losses of over 7 dB/cm² for waveguides with Ti:O ratios of 1:1 and 1:2.

"The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-84OR21400. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of the contribution, or allow others to do so."

*Corresponding author: Tel +1 205 851 5866, FAX +1 205 851 5868, e-mail ila@cim.aamu.edu

INTRODUCTION

Lithium niobate is a commercially important electro-optical material, part of the advanced technology effort in optical communication and computing. Electro-optical devices are now used to substitute traditional electronic devices in communications and in information processing. Integrated optical LiNbO_3 devices include couplers, switches, modulators, multiplexers and others [1,2]. Three different techniques have been employed to create waveguides in lithium niobate: proton exchange, Ti diffusion and ion implantation [3,4,5]. Ion implantation allows tighter control over device geometry, dopant concentration and dopant profile than the diffusion and proton exchange methods [6]. Implantation of O following Ti implantation has been found to reduce post-implantation segregation of Ti [7]. In this work the effect of varying the O implant fluence on waveguides loss is investigated.

EXPERIMENTAL

The optically polished x-cut crystals were purchased from Crystal Technology, Inc. The implantation was carried out using the Surface Modification and Characterization Facility at Oak Ridge National Laboratory.

Following the process established by Poker and Xia [8], Ti and O implants of 2.5, 5.0 and $7.5 \times 10^{17}/\text{cm}^2$ were performed at 500°C . The case in which the Ti:O ratio is 1:3 is the stoichiometric case, referring to the Nb:O ratio in LiNbO_3 . Titanium was implanted at 320 keV followed by an oxygen implantation at 120 keV. The ratio of Ti to O energy was computed using SRIM96 [9] to give equal ranges of 180 nm. The width of the waveguide is determined by the thickness of the implanted Ti layer and can be approximated by the straggling value given by SRIM as 65 nm. Implantation at a temperature slightly above the recrystallization temperature reduces amorphization of the crystal by the implanted ions [9]. The fluences were based upon previous Ti implantation research which found that a high Ti dose is necessary to replicate the atomic percent concentration of Ti that is produced by Ti indiffusion [3]. The combination of high temperature and O implant stabilizes the position of the Ti implant and prevents the formation of an oxide layer on the surface [8]. Transmission electron

microscopic analysis of stoichiometric implanted LiNbO_3 [10] indicates that the implanted Ti layer remains approximately $70\mu\text{m}$ below the crystal surface, suggesting that the guiding region is completely buried. The implanted crystals were dark after the Ti implantation but were a light gray after the oxygen implant. The color varied with the O fluence, becoming less opaque with increasing O. The crystals were annealed at 1000°C for 4 h in O_2 bubbled through DI water. After annealing, all of the crystals returned to a clear state.

The implanted crystals were tested 632.8 nm with a Metricon 2010 automated prism coupler equipped with a rutile prism to confirm that all were indeed waveguides. All of the crystals had one strong propagating mode and a second 'lossy' mode. Due to the limitations of the prism coupler only TE modes could be investigated. After polishing the end of the crystal a $40\times$ microscope objective was used to couple TE polarized 672 nm laser light into the waveguides. An Electrim 753×244 element, 8-bit, B/W CCD camera was used to capture images of light scattered from the waveguides. The public domain NIH Image program (available on the Internet by anonymous ftp from [zipper.nimh.nih.gov](ftp://zipper.nimh.nih.gov)) for Macintosh computers was used to analyze the digital images from the CCD camera. All of the characterization was done for y-propagating guides. They ranged in length from 5 mm to 1.2 mm.

The side edges of the crystals were blacked out with a permanent marker to reduce scattering from the edges. In addition, a 1 mm wide strip of carbon tape was placed along the input edge of the guide to reduce stray light. As shown in Figure 1, a $20\times$ microscope objective was used to project the output onto a screen or onto a photodetector through a 1 mm slit. A macro lens attached to the CCD camera resulted in each column of $11.5\mu\text{m}$ wide pixels in the digital image corresponding to a distance of $40\mu\text{m}$ along the length of the crystal.

RESULTS AND DISCUSSION

Several techniques are available to measure attenuation on optical waveguides: sliding prism, cutback and scattering are three of the most common. In this work we

report loss measurements made using the scattering technique [11] and the cutback technique.

A surface profile of a light streak from a non-stoichiometric implanted guide is shown in Figure 2. Similar images were obtained for all but one of the implanted crystals. The images were analyzed by integrating one pixel wide cross sections of the scattered light streaks. To keep the background constant a fixed number of pixels were used for each measurement. Figure 3 shows a plot of integrated intensity versus propagation distance after a background subtraction and removal of scattering from dust particles from the waveguide shown in Figure 2. The fit to the data indicates an attenuation of 10.6 dB/cm for this guide, which has a Ti:O ratio of 1:1. A sample implanted with a Ti:O ratio of 1:2 exhibited losses in excess of 20 dB/cm. Such a high loss was unexpected and may be due to an excessive O current that caused the crystal to develop a crack during implantation. In agreement with previous work [12], samples implanted with a Ti:O ratio of 1:3 were found to have losses ranging from 2.3 to 3.1 dB/cm.

A stoichiometric guide which exhibited very low levels of scattering was analyzed exclusively by the cutback method. The waveguide was initially 11.0 mm long and the output was measured after the crystal was cut and polished to lengths of 7.2, 4.4 and 3.2 mm. The same end of the crystal was used for coupling to the three longest lengths to keep the input as constant as possible. The 3.2 mm long crystal was the remnant from the first cutback. For each length the input parameters were optimized for maximum output. Despite the low visible scattering, the loss was found to be 3.1 dB/cm, similar to that found by the scattering method for other stoichiometric crystals. The 1:1 Ti:O crystal was cut once and the loss was found to be 7 dB/cm, in rough agreement with the scattering loss measurement.

CONCLUSION

Stoichiometric Ti and O implantation at 500°C produces waveguides that exhibit losses of less than 3 dB/cm as measured by two different techniques. The losses in non-stoichiometric Ti and O implanted guides were found to be much higher than those

in the stoichiometric guides. The attenuation in the stoichiometric guides is well above the oft-cited goal of 1 dB/cm for practical applications but should be considered as an estimate that may well be reduced with further study.

ACKNOWLEDGMENTS

This research is supported by the Center for Irradiation of Materials at Alabama A&M University, by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and the Alabama Space Grant Consortium.

REFERENCES

- [1] A. Selvarajan and J. E. Midwinter, *Optical and Quantum Electronics* 20 (1989) 1.
- [2] R. R. A. Syms, *Optical and Quantum Electronics*, 20 (1989) 189.
- [3] B.R. Appleton, G.M. Beardsley, G.C. Farlow, W.H. Christie and P.R. Ashley, J. Mater. Res. 1 (1986) 104.
- [4] L. McCaughan in: *Integrated Optics and Optoelectronics*, eds. K. Wong, M. Razeghi (SPIE, Washington, 1993) p. 15
- [5] Ch. Buchal, P. R. Ashley and B. R. Appleton, J. Mater. Res. 2 (1987) 222.
- [6] G. L. Destafanis, J. P. Gailliard, E. L. Ligeon, S. Valette, B. W. Farmery, P. D. Townsend, and A. Perez J. Appl. Phys. 50 (1979) 898.
- [7] D. B. Poker and W. Xia in: *Beam Solid Interactions :Physical Phenomena; Symp. Proc. Vol 157*, eds. J. A. Knapp, P. Borgensen and R. A. Zuhr (Mat. Res. Soc., Pittsburgh, 1990).
- [8] Ziegler, J. F., J. P. Biersack, U. Littmark *The Stopping and Range of Ions in Solids* (Pergamon, New York, 1985).

- [9] D. B. Poker, Solid State Division, Oak Ridge National Laboratory, personal communication.
- [10] Y. Okamura, S. Yoshinaka and S. Yamamoto, *Appl. Opt.*, **22** (1983) 3892.
- [11] E. K. Williams, D. Ila, S. Sarkisov, P. Venkateswarlu and D. B. Poker in *Ion Solid Interactions for Materials Modification and Processing; Symp. Proc. Vol. 396*, D. B. Poker, D. Ila, Y-T Cheng, L.R. Harriott and T. W. Sigmon, Eds. (Mat. Res. Soc., Pittsburgh, 1996).

Figure Captions.

Figure 1. Experimental set-up for scattering and cutback loss measurements.

Figure 2. Light intensity profile from crystal implanted with Ti and O in a 1:3 ratio.

Figure 3. Plot of integrated intensity vs. propagation distance for crystal of Fig. 2.
showing attenuation of 10.6 dB/cm.

Figure 2

Figure 3