

0038-1098(94)00640-7

DAMAGE AND ANNEALING OF POTASSIUM TITANYL PHOSPHATE AFTER Er⁺ AND Yb⁺ IMPLANTATION

Ke-Ming Wang, Bo-Rong Shi, Hong-Ying Zhai and Xiang-Dong Liu

Department of Physics, Shandong University, Jinan 250100, Shandong, People's Republic of China

Yao-Gang Liu

Institute of Crystal Material, Shandong University, Jinan 250100, Shandong, People's Republic of China and

Yi Li and Ju-Sheng Li

Institute of Physics at Chanchun, Chinese Academy of Sciences, People's Republic of China (Received 7 December 1993; in revised form 29 August 1994 by R.H. Silsbee)

200–350 keV Er⁺ and Yb⁺ were implanted into potassium titanyl phosphate (KTP or KTiOPO₄). The dose ranged from 1×10^{13} to 5×10^{13} ions cm⁻². The radiation damage was investigated by RBS (Rutherford backscattering spectrometry)/channeling as a function of dose, energy, ion mass and annealing. The results show that the damage in KTiOPO₄ depends strongly on the dose, energy, ion mass and annealing. After 800°C annealing, some recrystallization was observed, but there remained damage for KTiOPO₄ (100) irradiated by 200 keV Er⁺ at a dose of 2×10^{13} ions cm⁻² which is a little different from the report on the case of 400 keV He ions implanted into the KTP.

Keywords: A: insulators, D: radiation effects, E: helium surface scattering.

1. INTRODUCTION

RECENTLY, there has been considerable interest in the field of rare-earth doped integrated optics because of their potential use in compact and efficient monolithic devices. For example, optical properties of rare-earth ions incorporated in insulators, such as glass hosts are of great interest in opto-electronic technology. Er is of particular interest due to its intra-4f transition with a wavelength of 1.54 μ m [1, 2].

Potassium titanyl phosphate (KTiOP₄) is a relatively new nonlinear optical material. KTiOPO₄ is also a potentially attractive material for certain integrated optical applications [3, 4]. Zhang and co-workers reported the optical waveguide in KTiOPO₄ and second-harmonic generation in KTiOPO₄ waveguide by MeV He implantation [5, 6]. Ion implantation is an accurate method for

modifying the refractive index of surfaces of insulator to form optical waveguide and waveguide lasers. The refractive index changes result either from the radiation damage or from the addition of impurity ions. Addition of doping ions by implantation is tempting in materials which can be annealed to remove the radiation damage and which recrystallize into perfect lattice structures as this allows accurate control of the impurity concentration and more complex doping possibilities. Therefore, ion implantation constitutes an alternative approach to integrated optics. By using ion implantation technique to a laser crystal, a planar optical waveguide can be formed which is capable of supporting laser oscillation. If low waveguide losses can be achieved, very low threshold laser operation could be possible. The process relies on an adequate knowledge of the damage and recovery of characteristics of the implanted layer [7, 8]. Channelling has been shown to be a powerful tool to study the radiation damage created from ion irradiation [9]. To our knowledge, there is no damage study of KTiOPO₄ implanted with rare earth ions. The main purposes of this work are to give the measurement of damage profiles in KTiOPO₄ after Er⁺ and Yb⁺ implantation, secondly to discuss the effect of the dose, ion mass, energy and annealing on the damage in KTiOPO₄ induced by rare earth ions.

2. EXPERIMENTAL

The potassium titanyl phosphate was optically polished and cleaned before irradiation. The quality of KTiOPO₄ crystal was checked using Rutherford Backscattering Spectrometry (RBS). The sample was provided by the Institute of Crystal Materials, Shandong University. In order to ensure uniformity over the implanted area, a two-directional electrostatic scanning system was used. The irradiation was carried out at room temperature. The dose ranged from 1×10^{13} ions cm⁻² to 5×10^{13} ions cm⁻². The KTiOPO₄ was implanted by both Er⁺ and Yb⁺. The energies used are as follows: 200 keV, 300 keV and 350 keV. To investigate the thermal annealing behavior of damage in KTP induced by ion irradiation, two samples were irradiated by 200 keV Er^+ to a dose of 2×10^{13} ions cm⁻². The annealing was carried out at 800°C in N₂ ambient for 10 min.

The KTiOPO₄ samples were analyzed by the RBS/channeling with 2.1 MeV ⁴He²⁺ from 1.7 MV tandem accelerator of Shandong University. The samples were mounted in a three dimensional goniometer with an accuracy of 0.01°. The target rotation was controlled by stepping motors that were controlled by a computerized system, which also determined the alignment corresponding to the minimum of the backscattering particles. The backscattering particles were detected at a scattering angle of 165° with surface barrier detector. The probed area was $1.5 \times 1.5 \,\mathrm{mm}^2$ in size. The He beam current was typically 15 nA. The ion beam was collimated by two slits which can be adjusted in the experiment. The amplified pulses from the detector were transferred to a multichannel analyzer and the spectra were finally stored in a computer for data analysis. The ion implantation was performed at the Institute of Physics at Chanchun, Chinese Academy of Sciences.

3. RESULTS AND DISCUSSION

It is difficult to calculate the parameters of damage distribution created by ions in such polyatomic target as KTiOPO₄ which is a four-element target made of O, P, K, and Ti. The He back-

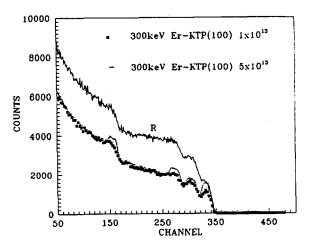


Fig. 1. Channeling spectra in KTiOPO₄ (100) irradiated by 300 keV Er⁺ to doses of 1×10^{13} ions cm⁻² and 5×10^{13} ions cm⁻².

scattering spectra are recorded for the channeling and nonchanneling direction. But the difference in the normalized yield curves (defined as the channeling spectrum divided, point by point, by the nonchanneling spectrum) for pre- and post-implant conditions was taken as a measure of the damage caused by the irradiation. In order to observe the dose dependence of damage in KTP created by both Er⁺ and Yb⁺, 1×10^{13} ions cm⁻², 2×10^{13} ions cm⁻² and $5 \times 10^{13} \, \text{ions cm}^{-2}$ were chosen. Figure 1 shows the typical damage spectra for KTP (100) induced by $300 \,\mathrm{keV} \,\mathrm{Er}^+$ to the doses of $1 \times 10^{13} \,\mathrm{ions}\,\mathrm{cm}^{-2}$ and 5×10^{13} ions cm⁻², respectively. R represents the random spectrum for KTiOPO₄ (100). As shown in Fig. 1, the amount of damage increases with the dose. In order to see the effect of ion energy on the damage, we have used Er+ implanted into KTiOPO₄ (100) at

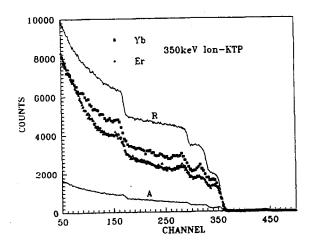


Fig. 2. Channeling spectra in KTiOPO₄ (100) irradiated by 350 keV Er⁺ and Yb⁺ under same dose.

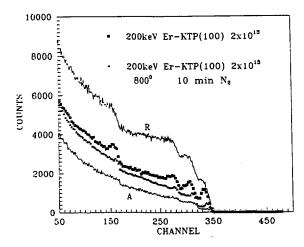


Fig. 3. Channeling spectra in KTiOPO₄ (100) irradiated by 200 keV Er⁺ to a dose of 2×10^{13} ions cm⁻² at room temperature and after 800°C annealing.

different energy. The implanted energies are 200 keV and 350 keV under the dose of 2×10^{13} ions cm⁻², respectively. It is observed that the position of the damage peak created by 350 keV Er⁺ is deeper than one created by 200 keV Er⁺. The effect of ion mass on the damage was investigated by Er⁺ and Yb⁺ at energy of 350 keV under the same dose. Figure 2 represents the RBS/channeling spectra for KTiOPO₄ (100) irradiated by 350 keV Er⁺ and Yb⁺ at the same dose. It is found that the damage is sensitive to the ion mass also. The height of the damage peak created by Yb⁺ is higher than one created by Er⁺. In order to investigate the thermal annealing behavior of the damage in KTiOPO₄, the KTiOPO₄ sample which was implanted with 200 keV Er+ to a dose of 2×10^{13} ions cm⁻² was annealed at 800°C in N₂ ambient for 10 min. Figure 3 indicates the comparison of channeled spectra at room temperature and after annealing. It is seen that with increasing of annealing temperature, the number of displaced atoms is decreased. After 800°C annealing, the aligned spectrum is closer to the one of virgin KTiOPO₄. But there is still remaining damage which is a little different from the case of 400 keV He ions implanted into KTiOPO4 to a dose of $1 \times 10^{16} \, \text{ions cm}^{-2}$ [10]. Where the annealing was

performed at different temperature in oxygen atmosphere. After 5 min annealing at 800°C, the RBS/channeling measurement indicates that the almost perfect recovery in damaged KTiOPO₄ induced by 400 keV He ions is observed.

4. SUMMARY

We have used the RBS/channeling technique to study the damage in KTiOPO₄ created by both Er⁺ and Yb⁺ as a function of the ion mass, dose, energy and annealing. The results show that the damage depends strongly on the ion mass, energy, dose and annealing. After 800°C annealing, some recrystallization is observed, but there is still remaining damage in the case of 200 keV Er⁺ implanted in KTP to a dose of 2×10^{13} ions cm⁻² which is a little different from the report on 400 keV He ions implanted into KTiOPO₄ to a dose of 1×10^{16} ions cm⁻².

Acknowledgements — The authors would like to thank Ji-Tian Liu and Ju-Xin Lu for their help in this work.

REFERENCES

- 1. E. Lallier, Appl. Opt. 31, 5276 (1992).
- A. Polman, A. Lidgard, D.C. Jacobson, P.C. Becker, R.C. Kistler, G.E. Blonder & J.M. Poate, Appl. Phys. Lett. 57, 2859 (1990).
- 3. J.D. Bierlein & C.B. Arweiler, Appl. Phys. Lett. 49, 917 (1986).
- 4. F.C. Zumsteg, J.D. Bierlein & T.E. Gier, J. Appl. Phys. 47, 4980 (1976).
- L. Zhang, P.J. Chandler, P.D. Townsend & P.A. Thomas, Electron. Lett. 28, 650 (1992).
- L. Zhang, P.J. Chandler, P.D. Townsend, Z.T. Alwahabi & A.J. McCaffery, *Electron. Lett.* 28, 1478 (1992).
- 7. P.D. Townsend, *Nucl. Instr. Meth.* **B46**, 184 (1990).
- 8. D.B. Poker & D.K. Thomas, *Nucl. Instr. Meth.* **B39**, 716 (1989).
- 9. L.C. Feldman, J.W. Mayer & S.T. Picraux, Materials Analysis by Ion Channeling. Academic Press, New York (1982).
- Ke-Ming Wang, Bo-Rong Shi, Zhong-Lie Wang, Xiang-Dong Liu, Yao-Gang Liu & Qing-Tai Zhao, Phys. Rev. B (in press).