High-Resolution Refractive Index and Micro-Raman Spectroscopy of Planar Waveguides in KGd(WO$_4$)$_2$ Formed by Swift Heavy Ion Irradiation

C. A. Merchant, *Member, IEEE*, P. Scrutton, S. García-Blanco, C. Hnatovsky, R. S. Taylor,
A. García-Navarro, G. García, F. Agulló-López, J. Olivares, A. S. Helmy, *Senior Member, IEEE*, and
J. S. Aitchison, *Senior Member, IEEE*

***Abstract*—We report on the characterization of planar waveguides formed in the Raman-active crystal KGd(WO$_4$)$_2$ using swift carbon, fluorine, and oxygen ion irradiation. The characterization of the waveguiding regions was performed using high-resolution microreflectivity and micro-Raman spectroscopy. The high-resolution microreflectivity measurement fully characterizes the refractive index profile of the barrier formed by amorphization of the crystal and detects other index variations not detected by the m-line technique. Raman spectroscopy measurements reveal details of the Raman properties of the crystal in the waveguiding region in relation to the rest of the sample for the different ion irradiations. Both of these measurement techniques are shown to be important for use of KGd(WO$_4$)$_2$ in integrated Raman-active devices.**

***Index Terms*—Laser crystals, microreflectivity, optical planar waveguides, Raman spectroscopy.**

I. INTRODUCTION

THERE is a growing interest in creating waveguides in crystalline materials for use in integrated optics and waveguide laser devices. Potassium gadolinium tungstate KGd(WO$_4$)$_2$ (or KGW) is a very attractive material for this due to its high third-order nonlinear susceptibility and its suitability as a rare-earth host material [1], [2]. KGW has strong Stokes lines located around 901 cm^{-1} and 768 cm^{-1}, depending on the exciting beam polarization and crystal orientation. These strong Raman modes, coupled with the crystals' short dephasing time T_2 of about 1.5–2.0 ps, make KGW an excellent candidate for high-intensity picosecond stimulated Raman scattering (SRS) laser emission in the telecommunications spectrum [3], [4].

Waveguides have been fabricated in double tungstate crystals using different techniques such as light ion implantation [5], [6], liquid-phase epitaxy [7], and femtosecond laser writing [8]–[10]. Swift heavy ion irradiation (SHI) is becoming increasingly popular as a method of strongly modifying the refractive index of optical crystals in well-defined regions [11]–[14]. Ions with kinetic energies of the order of MeV will lose energy primarily through electronic interactions. This regime is known as the electronic stopping regime (ESR). If the electronic stopping power $S_{\rm e}$ is above a certain threshold $S_{\rm th}$, amorphous (latent) ion tracks of several nanometers in diameter are formed along the ion path [15]–[17]. With a sufficient ion flux, highly amorphous regions can be formed in the crystal at well-controlled depths, using ion fluences that are orders of magnitude lower than light ion implantation techniques operating in the nuclear stopping regime (NSR). It is possible to choose the ion irradiation energy to have the maximum electronic stopping power at a certain depth in the crystal, allowing the formation of amorphous regions away from the surface of the crystal, which is advantageous for fabrication of buried or three-dimensional (3-D) waveguiding structures. Refractive index profiles of KGW irradiated with fluorine, carbon, and oxygen have been reported using a standard m-line technique [18]. However, the nature of the ion beam interactions with the crystal structure in the SHI process can induce defect formation in areas outside of the desired region. It is therefore important to ensure that the crystals' laser host, nonlinear refractive index $\rm n_2$, and Raman characteristics are preserved in the waveguide, ensuring optimum waveguide device performance. This requires careful tailoring of the implantation parameters along with the choice of optimum implantation species. We present in this paper a more complete characterization of the KGW crystals' refractive index and Raman properties in the waveguiding regions, in order to validate the SHI method of waveguide formation of Raman-active integrated devices using KGW. These techniques prove to be important in revealing additional information on the damage

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C. A. Merchant was with with the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON M5S 3G4, Canada. He is now with Brightwell Technologies, Inc., Ottawa, ON K2M 2B2 Canada (e-mail: clark.merchant@utoronto.ca).

P. Scrutton, A. Helmy, and J. S. Aitchison are with the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON M5S 3G4, Canada (e-mail: pscrutton@yahoo.com; a.helmy@utoronto.ca; stewart.aitchison@utoronto.ca).

S. Garcia-Blanco was with the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON M5S 3G4, Canada. She is now with the National Optics Institute (INO), Sainte-Foy, Québec QC G1P 4S4, Canada (e-mail: Sonia.Garcia.Blanco@ino.ca).

C. Hnatovsky was with the Institute for Microstructural Sciences, National Research Council, Ottawa, ON K1A 0R6, Canada. He is now with OZ Optics Ltd., Ottawa, ON K0A 1L0 Canada (e-mail: Khnatovsky@ozoptics.com).

R. S. Taylor is with the Institute for Microstructural Sciences, National Research Council, Ottawa, ON K1A 0R6, Canada (e-mail: Rod.Taylor@nrc-cnrc.gc.ca).

A. García-Navarro and F. Agulló-López are with the Centro de Microanálisis de Materiales (CMAM), Universidad Autónoma de Madrid (UAM), Cantoblanco, 28049 Madrid, Spain (e-mail: agar@uam.es; fal@uam.es).

G. García is with the Consortium for the Exploitation of the synchroton Light Laboratory (CELLS), Barcelona, Spain.

J. Olivares is with Instituto de Óptica, CSIC, 28049 Madrid, Spain (e-mail: jose.olivares@uam.es).

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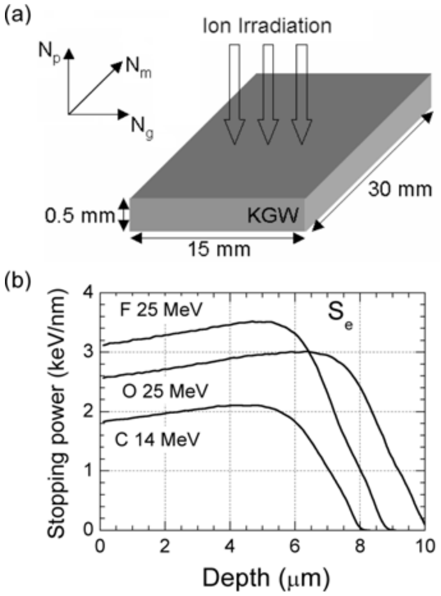

Fig. 1. (a) Crystal orientation of the ion irradiation process and (b) the stopping powers versus depth for the carbon, oxygen, and fluorine ions.

processes of the SHI in terms of showing features through microreflectivity not previously revealed with m-line characterization, as well as showing changes in Raman properties around the waveguiding region.

II. Experiment Setup

A. Ion Irradiation

KGW is a monoclinic crystal with the three refractive indexes $\mathrm{n_m} = 2.041, \mathrm{n_g} = 2.095$, and $\mathrm{n_p} = 2.011$ at 633 nm. In our experiments, irradiations were performed normal to the $\mathrm{n_g}$ and $\mathrm{n_m}$ crystal plane, as illustrated in Fig. 1(a). High-energy irradiations using a 5-MV tandetron accelerator were performed with several ion species and fluences [12], with the electronic stopping power of these ions in KGW calculated using the Stopping and Range of Ions in Matter (SRIM) software and shown in Fig. 1(b). The samples were tilted 8° relative to normal incidence in order to avoid channeling, and the current density was kept below 100 nA/cm^2 to minimize charging and heating. The m-line measurements were carried out using the prism-coupling method with an HeNe laser for the carbon, oxygen, and fluorine irradiations [18]. In our initial m-line characterization, we identified the most promising results for waveguide devices being the 14-MeV carbon ions with fluence of 1×10^{14} atoms/cm^2 and 25 MeV oxygen and fluorine irradiations with fluences of 4×10^{13} and 4×10^{14} atoms/cm^2, respectively. The results were assessed based on the refractive index profiles generated from the m-line measurement, with strong refractive index modification regions detected, useful for formation of highly confined waveguides. Samples were then diced and polished for measuring the properties of the cross-sectional regions influenced by the ion irradiation as a function of depth from the crystal surface.

B. Microreflectivity Setup

The microreflectivity technique uses the principle of Fresnel reflection relating the refractive index change of a material to its change in reflectivity [19]. The microreflectivity measurements were carried out using a setup consisting of a 632.8-nm HeNe laser focused through a 50 × objective with an NA of 0.8, giving an effective spatial resolution of approximately 400 nm and refractive index variation sensitivity of $\Delta\mathrm{n} = 1 \times 10^{-4}$. A combination of a polarizer and waveplate was used to control the probe beam polarization. The sample was placed on an XYZ piezo-scanner, and measurements were taken along the polished facets over a 10- or 15-μm area of the sample with 512×512 sampling points and a scan rate of 3 Hz.

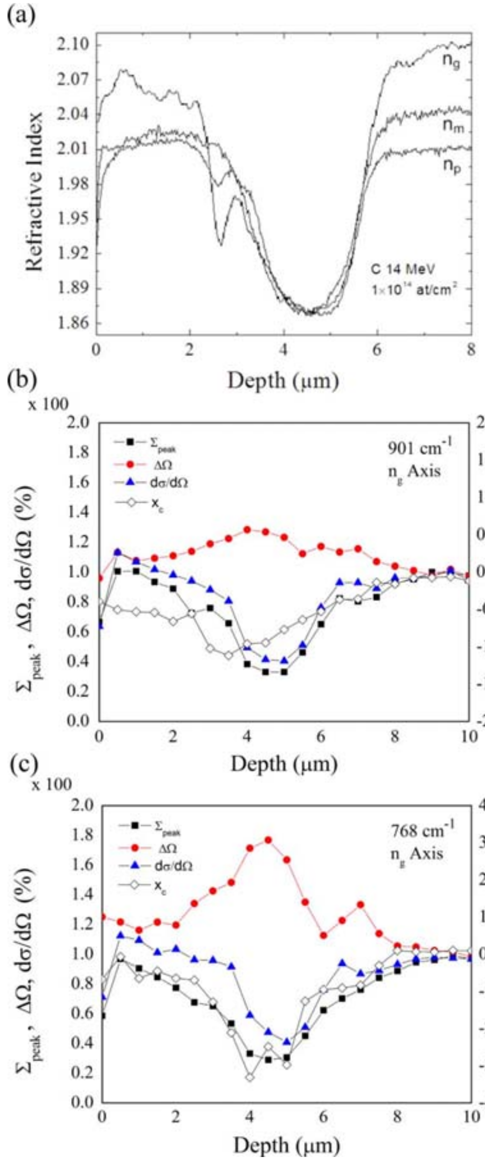

Fig. 2. (a) Refractive index profile and (b) variations in Raman mode parameters across the crystal for a probe beam polarized along the $\mathrm{n_g}$ axis, (c) for the 14-MeV carbon ion irradiated sample.

C. Raman Spectroscopy

Micro-Raman measurements of the facets were taken using a JY Horiba LabRAM micro-Raman instrument in confocal configuration (100-μm confocal aperture, 100-μm slit, 1200-l/mm grating). The excitation source was a doubled Nd:YAG laser at 532 nm, with ~24 mW power, focused through a 100 × objective with a numerical aperture of 0.9. The polarization of the

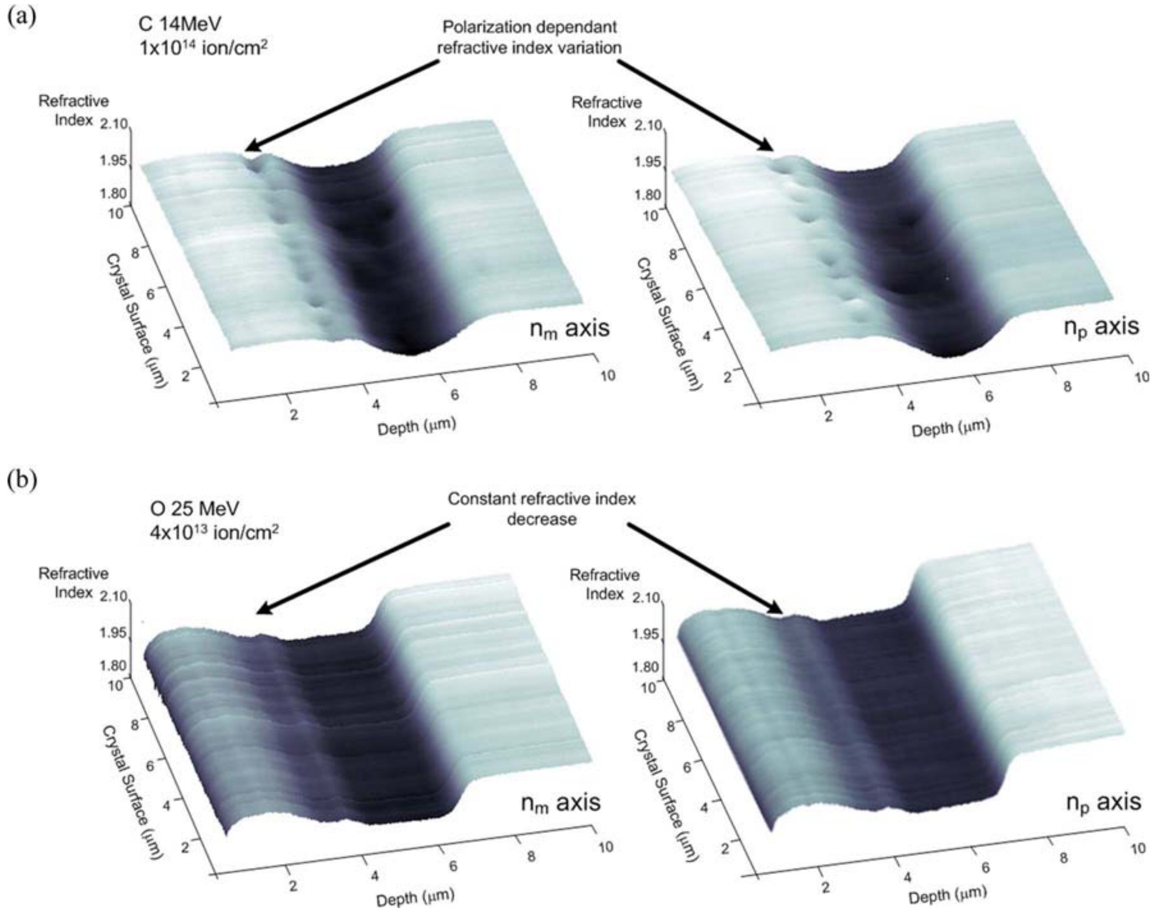

Fig. 3. Refractive index measurements of barriers formed by (a) the 14-MeV carbon ion irradiation and (b) the 25-MeV oxygen ion irradiation for different probe beam polarizations for the facet normal to the $\mathrm{n_g}$-axis.

probe beam was aligned with the crystal's $\mathrm{n_g}$ axis, as it possesses both a strong 901-cm^{-1} and 768-cm^{-1} mode, ideal for SRS applications. The sample was placed on a motorized stage where the cross section of the ion irradiated region was measured over a 20×20 μm area with 0.5-μm step size and a capture time of 1 s per measurement point. The polarized Raman spectrum was measured for each step, and the intensity, width, cross-sectional area, and frequency shift of the Raman modes for this crystal orientation was recorded.

The peak Raman intensity of these modes can be denoted by

$$\Sigma_{\text{peak}} \sim (d\sigma/d\Omega)(\Delta\Omega_R)^{-1} \tag{1}$$

where $d\sigma/d\Omega$ is the cross-sectional area and $\Delta\Omega_R$ is the linewidth of the Raman mode [20]. This peak intensity Σ_{peak} is directly related to the steady-state Raman gain through

$$g_{\text{SS}} = \frac{\lambda_p \lambda_S^2 N}{\hbar c \pi n_S^2} \Sigma_{\text{peak}} \tag{2}$$

where $\lambda_{p,S}$ are the pump and Stokes wavelengths, N is the number of scattering centers, and n_s is the refractive index at the Stokes wavelength. By comparing the intensity of the Raman mode in the irradiated regions of to those of bulk unmodified KGW, (2) can be used to directly predict the expected change in steady-state Raman gain across the waveguiding region. The Raman mode linewidth $\Delta\Omega_R$ is also used to estimate the Raman dephasing time $\mathrm{T_2} \propto 1/\Delta\Omega_R$, which indicates the shortest pump pulse possible for steady-state Raman gain operation [21]. In the transient regime where the pump pulse is much shorter than $\mathrm{T_2}$, the transient Raman gain is proportional to the square root of the cross-sectional area $(d\sigma/d\Omega)^{1/2}$ and not the peak intensity [22]. Thus, changes in the mode area in the waveguiding region alone can be related to a change in the device gain in the transient regime, which is important for ultrafast SRS applications where high transient Raman gain is desired.

The frequency shift of Raman modes as a function of position in a material can be used as a measurement of localized stress on the lattice of the crystal. The prominent Raman modes in KGW arise from the WO_4 group of the crystal, with similar frequency modes arising in other tungstate crystals with the same sublattice arrangement [22]. Stress measurements were performed on $PbWO_4$ crystals, where its similar 902-cm^{-1} mode was found to shift to higher frequencies for compressive stress and lower frequencies for tensile stress [23]. This behavior was also found for channel waveguides fabricated in KGW using femtosecond laser processes that use compressive stress to form core waveguiding regions [10]. In addition, the measurement of Raman frequency shifts in the waveguide core region through either stress or damage will directly indicate the subsequent shifts of the output Stokes wavelength in SRS-based devices.

III. RESULTS AND DISCUSSION

Figs. 2 and 3 illustrate the microreflectivity and micro-Raman spectroscopy measurements of the 14-MeV swift carbon ion irradiation with a fluence of 1×10^{14} ions/cm^2. The microreflectivity measurement for all three polarizations reveals a high-refractive-index guiding region just below the crystal surface followed by a strong reduction of the refractive index, correlating well to the m-line measurements previously performed [18]. However, the microreflectivity technique offers the additional information on the barrier width, where the microreflectivity scans show the refractive index beginning to decrease at a depth of 2 μm from the surface, and recovering at a depth of 6 μm, with an amorphous region formed between 4 and 5 μm from the crystal surface. This characterization of the barrier width is an important parameter in determining waveguide performance, as it determines mode leakage into the substrate of the waveguide. The microreflectivity technique also reveals previously undetected features appearing intermittently in the planar waveguide core. These intermittent changes in the refractive index are found approximately 2–2.5 μm from the crystal surface and are not detected in the previous m-line experiments [18]. The top image in Fig. 3 shows a 3-D profile of the microreflectivity measurement for the carbon irradiation over a 10×10 μm area, illustrating these intermittent changes of refractive index. For the polarization of the microreflectivity probe beam oriented along the $\mathrm{n_m}$-axis direction, both positive and negative refractive index changes are measured. The associated $\Delta\mathrm{n}$ variation was approximately -0.13 to $+0.03$. The polarization of the probe oriented along the $\mathrm{n_p}$ polarization direction showed only reduction in the refractive index. This refractive index variation can have detrimental effects on scattering loss which scales with the square of the change in refractive index. The root cause of this phenomenon is not yet known, however, continuous positive index of refraction values for one polarization have been previously observed in other crystalline insulators after light ion implantation damage events due to ionic motion, bond compaction, or modified bond rearrangements [24].The polarized micro-Raman spectroscopy measurements shown in Fig. 2 for the carbon irradiation illustrate good correlation between the Raman intensity evolution of the 901-cm^{-1} and 768-cm^{-1} modes and the refractive index profile from the microreflectivity measurement, where the barrier reveals a reduced Raman intensity suggesting strong amorphization. The intermittent feature found 2–2.5 μm from the surface shows a reduction in Raman intensity for both modes, suggesting that these positive and negative refractive index changes exist in a region where the crystal has become amorphous in nature. The first two micrometers from the surface of the crystal constituting the guiding region of the planar waveguide shows no lattice damage, having a high intensity count of the Raman modes comparable to nonirradiated KGW. The cross-sectional area is also seen to be recovered in the guiding region, and thus the Raman steady-state and transient gains are expected to be preserved for waveguiding devices using SRS processes. There is a slight negative Raman frequency shift of about -0.5 cm^{-1} observed in the guiding region for the 901-cm^{-1} mode, suggesting a tensile stress following previous results on tungstate crystals [10], [23]. Annealing may help eliminate these stress effects, which will otherwise cause an output wavelength shift in SRS-based devices.

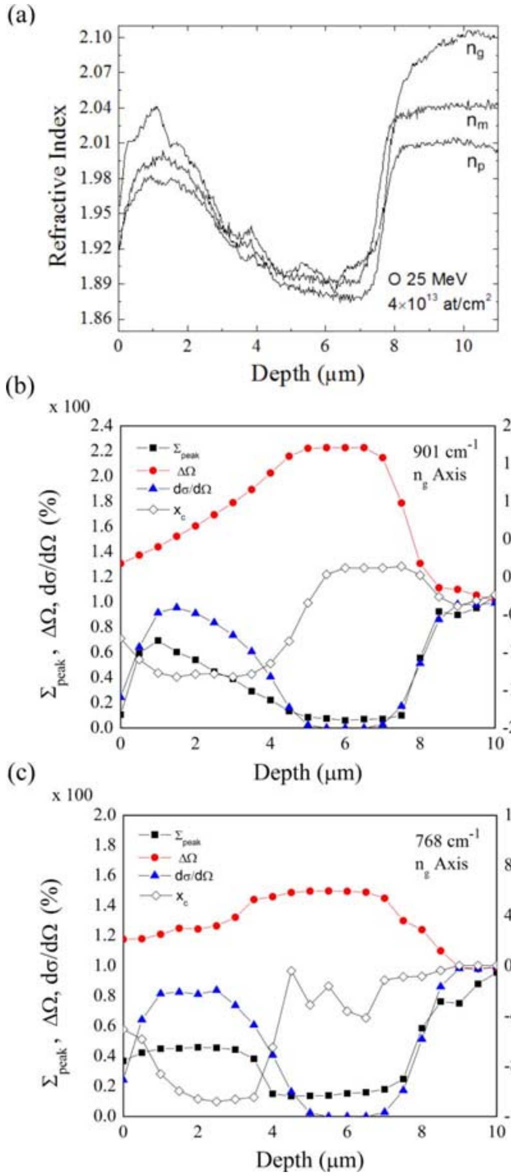

Fig. 4. (a) Refractive index profile and (b), (c) variations in Raman mode parameters across the crystal of 25-MeV oxygen ion irradiated KGW.

Figs. 3 and 4 show the irradiation of the KGW crystal by swift oxygen ions at 25 MeV and a fluence of 4×10^{13} ions/cm^2, revealing the formation of a 3-μm-wide amorphous region located approximately 4 μm from the surface of the crystal. The refractive index profile measured with the microreflectivity technique also shows an unexpected refractive index feature located about 3 μm from the surface. As seen in the bottom image of Fig. 3, the refractive index measurement for the oxygen ion irradiation does not show the intermittent positive and negative index variation found in the carbon irradiation, instead exhibiting a constant refractive index decrease for both polarizations. The micro-Raman spectroscopy measurements reveal the guiding region near the surface to have more lattice damage than was seen with the carbon ion irradiation, with the peak intensity of the 901-cm^{-1} mode in the core region less than half of the intensity of undamaged KGW. The 768-cm^{-1} mode shows better

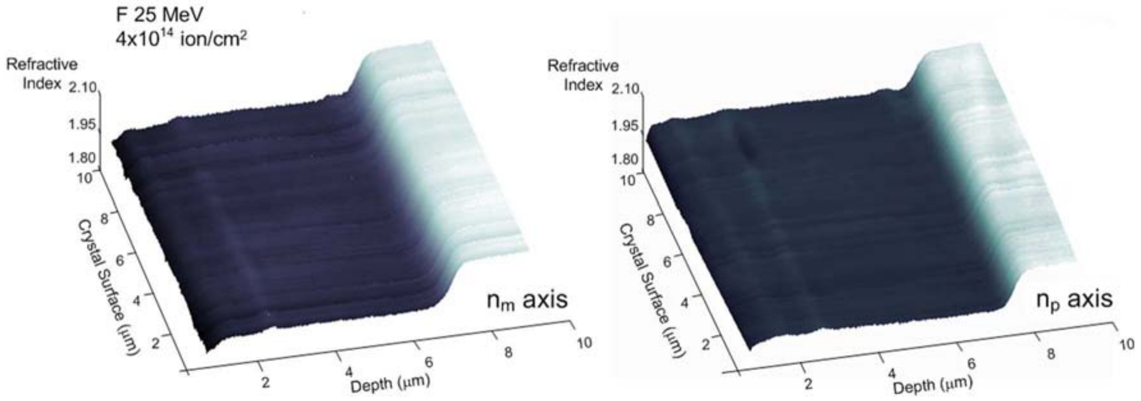

Fig. 5. Refractive index measurements of barrier formed by the 25-MeV fluorine ion irradiation, showing a highly amorphous region extending from the surface.

preservation of the peak intensity, measured to be approximately 80% in the center of the guiding region. Using (2), it can then be inferred that the steady-state Raman gain will be reduced by the same percentage as measured by the intensity profiles of each mode in Fig. 4. The cross-sectional area of the mode is also reduced slightly, suggesting the transient Raman gain will also be affected, but to a lesser degree than the steady-state Raman gain. The shifts of the Raman modes reveals some interesting characteristics, where the 901-cm^{-1} mode is found to shift by -1 cm^{-1} in the guiding region, while the 768-cm^{-1} mode shifts by approximately -10 cm^{-1}. This suggests this 768-cm^{-1} mode to be a sensitive stress indicator for KGW, as was also reported in [10]; however, here the presence of lattice damage can also be a factor in producing frequency shifts in addition to stresses. This strong Raman shift in the waveguide core region can have an impact on the output wavelength of SRS-based devices. This was found previously in Raman gain experiments performed on waveguides fabricated by compressive stress through femtosecond laser writing, where a similarly shifted 768-cm^{-1} mode in these waveguide cores produced SRS output at wavelengths which correlated strongly with these measured Raman mode shifts [10]. Overall, although strong waveguides are formed using oxygen irradiations as seen by the refractive index profiles, the Raman performance in the guiding regions is significantly reduced, suggesting that damage to the crystal lattice and thus device performance is expected to decrease for Raman conversion applications.

For samples irradiated with 25-MeV fluorine ions at a fluence of 4×10^{14} ions/cm^2, previous m-line measurements reported a predominantly amorphous region extending from the crystal surface. The microreflectivity measurements of the fluorine-irradiated sample shown in Fig. 5 reveal a more detailed profile of the damaged crystal, where the microreflectivity measurement shows two distinct weakly guiding regions, separated by a small dip in refractive index around 2 μm. Micro-Raman spectroscopy measurements revealed no Raman signal in the damage region, indicating the region to be amorphous in nature.

For each of these ion irradiation species, the use of annealing could improve the Raman performance of the devices. Routes forward based on this work are to explore various annealing techniques on both eliminating the irregular refractive index features found in the carbon and oxygen irradiated samples, as well as potentially improving the Raman performance in the guiding region of the oxygen irradiated sample.

IV. CONCLUSION

This paper has shown features of planar waveguides formed by SHI in KGW crystal that are not revealed through standard m-line measurement techniques. The carbon and oxygen ion irradiation results show strong potential for the SHI technique for use in fabricating planar waveguides in KGW. The carbon ion irradiation shows better preservation of the crystal Raman characteristics in the guiding region, although exhibiting irregularities in the refractive index profile. The oxygen ion irradiation showed crystal damage in the guiding region of the crystal, although annealing the crystal may help restore the crystal structure. The fluorine ion irradiation creates an amorphous layer up to 7–8 μm from the crystal surface, and, when used in conjunction with a carbon or oxygen irradiation and masking, shows good promise for the creation of 2-D waveguide structures in KGW. The measurement techniques used in this paper prove to be valuable in revealing important information such as Raman gain characteristics and small refractive index fluctuations, which are important for device fabrication based on the KGW crystal. As well, these characterization techniques can be useful for more completely exploring the damage formation mechanisms of the SHI process in KGW. Overall, the SHI process of waveguide formation in KGW shows excellent potential for use in rapid fabrication of Raman and Kerr-active planar waveguide arrays. Future work lies in developing multiple SHI irradiation processes with masking and annealing towards fabricating 2-D or 3-D integrated, Raman-active waveguide structures.

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Clark A. Merchant (M’07) received the B.Sc. and B.Eng. degrees from Dalhousie University, Halifax, NS, Canada, in 1996 and 1999, respectively, and the M.A.Sc. degree from Carleton University, Ottawa, ON, Canada, in 2003, and the Ph.D. degree in electrical and computer engineering from the University of Toronto, Toronto, ON, Canada, in 2008. His dissertation research focused on fabrication and characterization of waveguides in nonlinear crystals.

He was with the Exploratory Research Department at JDS Uniphase from 1999 to 2003. He has over ten years of both industrial and academic experience with photonic devices and is currently with Brightwell Technologies Inc., Ottawa, where he is involved with the field of flow microscopy.

Dr. Merchant is a Registered Professional Engineer in the Province of Ontario.

Philip Scrutton received the B.Sc. and M.A.Sc. degrees from the University of Toronto, Toronto, ON, Canada, in 2004 and 2006, respectively.

His research interests encompass optical spectroscopy of compound semiconductor nanostructures.

Sonia García-Blanco received the Ingeniero Superior degree from the Escuela Superior de Ingenieros de Telecomunicación from the Universidad Politécnica de Madrid, Madrid, Spain, in 1999 and the Ph.D. degree in electrical engineering from the University of Glasgow, Glasgow, U.K., in 2003. Her dissertation focused on electron-beam irradiation of silica for integrated optics.

From May 2003 to February 2005, she was a Postdoctoral Fellow with the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON, Canada, where her research interests included the study of the photosensitivity of various glass materials by different irradiation sources for the fabrication of optical biosensors and the integration of optical waveguides and microfluidics circuits in polymer materials for the realization of lab-on-a-chip devices. She is currently a Researcher with the MOEMS group of INO, Québec, QC, Canada, where she works on the development of miniaturized optical systems, microassembly techniques, and wafer-level vacuum hermetic packaging technologies.

Cyril Hnatovsky received the Ph.D. degree in optical physics from the University of Ottawa, Ottawa, ON, Canada, in 2005.

In 1999, he joined the National Research Council of Canada (NRC), where he worked on the development of probes for near-field scanning optical microscopy (NSOM) and for manipulation, heating, and interrogation of microscopic quantities of material in liquids. He also carried out application-oriented research in the field of fiber optics, waveguide optics, and optical microscopy. His latest research interests concentrate on using focused femtosecond laser radiation for the fabrication of microfluidic components and rewritable highly robust optical data storage in glass. Since 2008, he has been a Senior Scientist with OZ Optics Ltd., Ottawa, where he is involved with the development of the first commercial fiber optic products manufactured using femtosecond laser technology.

Rod S. Taylor received the Ph.D. degree in physics from McMaster University, Hamilton, ON, Canada, in 1977.

That same year, he joined the National Research Council of Canada (NRC), Ottawa, ON, where he worked on the development and applications of self-sustained discharge excimer lasers. In 1987, he was part of a surgical team which performed the world’s first excimer laser recanalization of a human coronary artery. In 1995, he switched fields into near-field scanning optical microscopy (NSOM) where he concentrated his efforts into developing high-throughput bent fiber probes so that commercial AFMs could be adapted to include NSOM. His research interests now focus on the development of specialized NSOM probes to investigate cellular function. He is also considered one of the technology pioneers of femtosecond laser induced self-organized planar nanocracks inside transparent dielectric materials.

Ana García-Navarro received the Physics degree from the Universidad de Sevilla, Seville, Spain in 2002 and the Ph.D. degree in material physics from Universidad Autónoma de Madrid (UAM), Madrid, Spain, in 2007. Her dissertation focused on photonic applications with high energy ions, particularly leading the topic of novel and efficient optical waveguide fabrication with swift heavy ions.

Gaston García received the Ph.D. degree from Universidad Autónoma de Madrid (UAM), Madrid, Spain, in 2000. His dissertation focused on experimental high-energy physics.

From 2000 to 2004, he was with the Centro de Microanalisis de Materiales (CMAM) of UAM as a Chief Enginieer, and he was also a Scientist working on the topic of ion-solid interaction and material modification. He is currently an Assistant Director in the construction of the Alba Synchrotron Ligh Facility, Barcelona, Spain.

Fernando Agulló-López is an Emeritus Professor with the Department of Physics of Materials, Universidad Autónoma de Madrid (UAM), Madrid, Spain. He is currently associated with the Center for Microanalysis of Materials (CMAM) as one of its promoters. His scientific interests include defects in solids, optical spectroscopy, nonlinear and photorefractive optics, and ion–beam–matter interactions. He is the author of more than 325 publications and several books and monographies. He has organized a number of schools, workshops and scientific conferences in his fields of activity.

José Olivares received the Ph.D. degree in physics from Universidad Autónoma de Madrid (UAM), Madrid, Spain, in 1994. His dissertation focused on the topic of proton-exchanged waveguides in lithium niobate.

From 1994 to 1996, he was a Postdoctoral Fellow with the University of Sussex, Sussex, U.K., where he was involved with the topics of ion-implanted waveguides and laser damage and ablation for film deposition and micro structuring. From 1997 to 1998, he was with the Universidad de Oviedo, Spain, where he was involved with integrated optics in lithium niobate. From to 1999 to 2001, he was with the Instituto de Optica, CSIC, Madrid, working in the field of third-order optical nonlinearities of metallic nanocomposites. In 2001, he became a Tenured Scientist of CSIC. Since 2003, he has been working in collaboration with the Center of Microanalisis de Materiales (CMAM) of UAM in the field of photonic applications with high-energy ions, particularly leading the topic of novel and efficient optical waveguide fabrication with swift heavy ions.

Amr S. Helmy (M'99–SM'06) received the B.Sc. degree in electronics and telecommunications engineering from Cairo University, Cairo, Egypt, in 1993, and the M.Sc. and Ph.D. degrees with a focus on photonic fabrication technologies from the University of Glasgow, Glasgow, U.K., in 1994 and 1999, respectively.

Prior to his academic career, he held a position at Agilent Technologies Photonic Devices, R&D Division, in the U.K. At Agilent, his responsibilities included developing distributed feedback lasers, monolithically integrated lasers, modulators, and amplifiers in InP-based semiconductors. He also developed high-powered submarine-class 980-nm InGaAs pump lasers. He is currently an Assistant Professor with the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON, Canada. His research interests include photonic device physics and characterization techniques, with emphasis on nonlinear optics in III-V semiconductors; applied optical spectroscopy in IIIV optoelectronic devices and materials; and III-V fabrication and monolithic integration techniques.

Dr. Helmy is a member of the Optical Society of America.

J. Stewart Aitchison (M'96–SM'00) received the B.Sc. (with first class honors) and Ph.D. degrees from the Physics Department, Heriot-Watt University, Edinburgh, U.K., in 1984 and 1987, respectively. His dissertation research was on optical bistability in semiconductor waveguides.

From 1988 to 1990, he was a Postdoctoral Member of Technical Staff with Bellcore, Red Bank, NJ. His research interests were in high-nonlinearity glasses and spatial optical solitons. He then joined the Department of Electronics and Electrical Engineering, University of Glasgow, Glasgow, U.K., in 1990 and was promoted to a personal chair as Professor of Photonics in 1999. His research was focused on the use of the half-bandgap nonlinearity of III-V semiconductors for the realization of all-optical switching devices and the study of spatial soliton effects. He also worked on the development of quasi-phase-matching techniques in III-V semiconductors, monolithic integration, optical rectification, and planar silica technology. His research group developed novel optical biosensors, waveguide lasers, and photosensitive direct writing processes based around the use of flame hydrolysis-deposited (FHD) silica. In 1996, he was the holder of a Royal Society of Edinburgh Personal Fellowship and carried out research on spatial solitons as a visiting researcher at CREOL, University of Central Florida. Since 2001, he has held the Nortel Chair in Emerging Technology in the Department of Electrical and Computer Engineering, University of Toronto, Toronto, ON, Canada. His research interests cover all-optical switching and signal processing, optoelectronic integration, and optical biosensors. His research has resulted in seven patents, approximately 185 journal publications, and 200 conference publications. From 2004 to 2007, he was the Director of the Emerging Communications Technology Institute at the University of Toronto. Since 2007, he has been vice Dean Research, for the Faculty of Applied Science and Engineering, University of Toronto.

Dr. Aitchison is a Fellow of the Optical Society of America and a Fellow of the Institute of Physics London.