Fabrication of buried channel waveguides in photosensitive glass using proton beam writing

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(Received 16 December 2005; accepted 27 March 2006; published online 26 April 2006)

We report our results on the fabrication and characterization of buried, channel optical waveguides in photosensitive Foturan™ glass using a high energy proton beam. Waveguides were fabricated with varying fluence, and the propagation loss and refractive index change were measured. Near-field mode data measured at 632.8 nm showed that waveguiding could be achieved for all fluences ranging from 10^{14} to 10^{16} protons/cm^2. The maximum positive refractive index change of 1.6×10^{-3} was measured for the highest fluence. The waveguide propagation losses measured using the scattering technique were estimated to be in the range of 8.3–12.9 dB/cm, increasing with proton fluence. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2198798]

Foturan™ is a photosensitive glass (manufactured by Schott Mikroglas) that can be directly patterned using conventional UV lithography,[1] femtosecond lasers,[2–5] and MeV protons.[6–8] Unlike other glasses that require masking in order to allow for the formation of patterns, irradiated regions of Foturan™ have a 20 times increase in etch rate after heat treatment.[1] The ability to directly form microstructures in this glass along with its resistance to high temperature and corrosion[1] has made it a particularly attractive platform for microfluidic applications such as microelectrochemical reactors.[9–11] Furthermore, its high transparency and the ability to modify the refractive index of this glass make it equally attractive for the fabrication of micro-optical components such as waveguides,[12] gratings,[13] and micromirrors.[3] Although femtosecond lasers are commonly used for waveguide fabrication in many types of glasses,[14] only a limited number of studies have concentrated on the fabrication of waveguides in Foturan™.

An alternative and efficient technique that can be used for waveguide fabrication is proton beam writing.[15] This technique utilizes a focused beam of MeV protons to direct-write structures in a material. The method relies on the fact that energetic ions lose most of their energy when they slow down in the form of collisions with target electrons, and by introducing atomic displacements. The cross section for both these processes increases with decreasing velocity of the incident ion resulting in most of the energy being deposited near the end of range, also known as the Bragg peak. The increased energy deposition at the end of range results in the formation of a region with altered refractive index that can be used for light guiding. Previous studies of proton beam irradiation for waveguide fabrication have concentrated on materials such as polymethylmethacrylate (PMMA),[16,17] fused silica,[18,19] and Er doped phosphate glass.[20] In this study we apply the fabrication technique to Foturan™ because it has the unique property of being a glass that can be both modified by irradiation to form waveguides and patterned to fabricate structures such as microfluidic channels. This makes Foturan™ potentially an important material for microfluidic devices that have integrated optics for biosensing applications.

Commercial Foturan™ samples were cut and mechanochemically polished along the edges to achieve an optical finish. The final polished samples were approximately $15\times7.5\times2$ mm^3 in size with a residual root mean squared surface roughness along the polished edges of approximately 4 nm [measured using an atomic force microscope (AFM)]. The waveguides were fabricated using the proton beam writing facility at the National University of Singapore,[21] where a beam of 2 MeV protons was used to direct-write waveguides over a length of approximately 7.5 mm. Seven waveguides were fabricated with fluences ranging from 1×10^{14} to 1×10^{16} protons/cm^2. During the irradiation, the proton fluence was monitored using an annular surface barrier detector mounted in the backward scattering direction. The SIMNRA software[22] was used to fit the Rutherford backscattering spectra in order to confirm that the desired fluence was delivered to the sample. No postannealing was performed on the sample.

Figure 1(a) shows an optical differential interference contrast (DIC) microscope image of the sample end face. Also shown in Fig. 1(b) is the atomic displacement profile due to nuclear stopping in Foturan™, and the ionization profile due to electronic stopping simulated using the stopping and range of ions in matter (SRIM2003) software.[23] The va-

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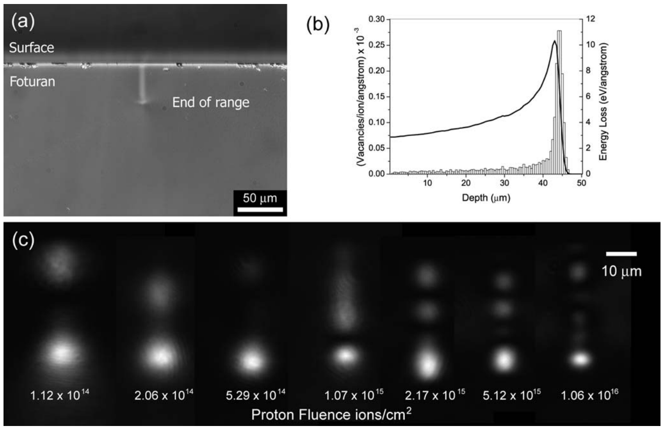

FIG. 1. (a) Optical differential interference contrast image of the Foturan™ sample edge showing a waveguide and the end of range region. (b) SRIM 2003 simulation of 2 MeV protons in Foturan™ (density=2.37 g/cm^3) showing the vacancy profile due to nuclear stopping and the ionization profile due to electronic stopping. (c) Optical images of the waveguide mode profiles for seven different proton beam fluences. The laser used for these measurements was a 17 mW, 632.8 nm helium–neon laser.

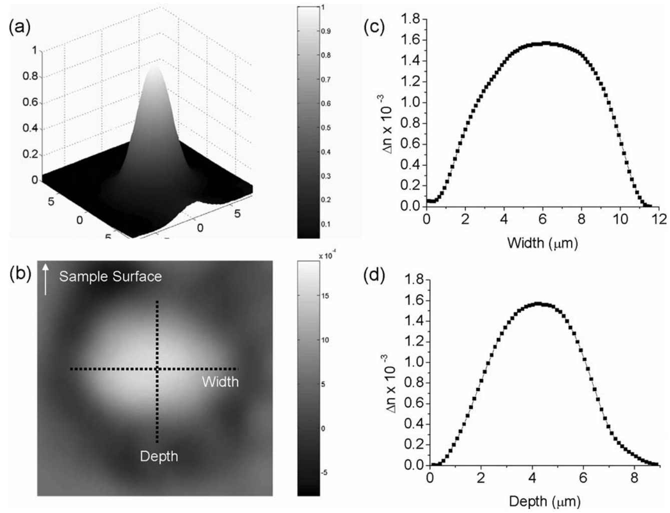

FIG. 2. (a) Near-field mode profile measured from the waveguide fabricated with the highest fluence (1×10^{16} protons/cm^2). (b) Recovered two-dimensional 2D refractive index profile and line profiles through the center of the mode along the (c) width and (d) depth of the sample.

cancy profile shows that the damage is fairly constant for the first 40 μm and increases sharply at the end of range. For a proton fluence of 1×10^{16} protons/cm^2, this corresponds to a vacancy concentration of 10^{20} cm^{-3} located approximately 45 μm below the top surface. This is at least an order of magnitude higher than the number of vacancies created in the first 40 μm of the sample. The electronic stopping profile peaks slightly before the end of range at about 42 μm below the surface. The whole proton range appears lighter in the DIC image indicating that there is some modification in the refractive index over the whole proton beam path into the sample. The work of Presby and Brown[24] on the irradiation of fused silica showed that both the nuclear and electronic stopping of protons can modify the refractive index. Based on this work, if we assume that the mechanism for refractive index change in Foturan™ is similar to that of fused silica, the refractive index change will be dominated by nuclear stopping for the range of fluences used in our study. The size of the waveguide core region that can be fabricated using proton beam writing is primarily determined by the scan size and the damage profile. It can be seen from the simulation that a majority of the atomic displacements that occur at the end of range are confined to a region approximately 5 μm in depth.

Optical characterization of the waveguides was performed using a 17 mW He–Ne (632.8 nm) laser that was launched into a single mode fiber (3M FS-SN3224). The fiber was buttcoupled to the end face of the waveguides, and the emerging light was imaged using a 50 times long working distance objective and a 12 bit cooled charge coupled device (CCD) camera. The sample was placed on a three-axis stage, while input and output stages had six axes for precise control over the light coupling. Appropriate neutral density filters were used to attenuate the input light for obtaining unsaturated near-field images with the CCD camera. Figure 1(c) shows a composite image of the mode profiles for the seven waveguides that were fabricated. Also shown is the proton fluence measured during the irradiation. This image shows that the increase in refractive index at the end of range with fluence results in a decrease in the mode field diameter. The mode field diameter for the waveguide produced with a fluence of 1×10^{16} protons/cm^2 was measured to be 8.1 μm in the vertical and 8.6 μm in the horizontal ($1/e^2$). This difference in the vertical and horizontal mode field diameter is due to the way in which the waveguide is fabricated. The horizontal profile can be controlled by changing the proton beam scan size, while the vertical profile is largely dependent on the way in which the protons interact with the sample via the nuclear and electronic stopping powers.

A minimum of 20 near-field images were recorded for each waveguide allowing us to average the output profiles for refractive index reconstruction. The refractive index profile was determined for the highest proton fluence (1×10^{16} protons/cm^2) using the propagation mode near-field technique.[19] This method allows one to determine the change in refractive index from the mode profile and a known refractive index for the bulk unirradiated material. We measured the bulk refractive index of our sample to be 1.511 at 632.8 nm using a Metricon prism coupling system. Figure 2 shows the extracted refractive index profile from the near-field data. The change in refractive index (Δn) determined using this method was estimated to be $(1.6\pm0.4)\times10^{-3}$ by using several series of images and repeating the acquisition several times. In order to confirm the accuracy of our method, the same system was used to recover the refractive index of a single mode fiber (3M FS-SN3224). The results obtained were in good agreement with the manufacturer data.[25]

Propagation loss measurements were performed using the scattering technique.[26] Laser light was launched into the waveguides using the same single mode fiber, and the scattered light was imaged using a stereo zoom microscope mounted perpendicular to the waveguide. The loss values were obtained by fitting the data to the equation $I=I_0e^{-\alpha l}$, where l is the length of the sample, and α being the propagation loss coefficient. The loss values retrieved from the best fits were in the 8–13 dB/cm range, increasing with proton fluence. Figure 3 shows an image of the light scattered from the waveguides, fabricated using the lowest and highest fluences, and a graph showing the extracted loss results. For these two waveguides we obtained loss values of 8.3 and

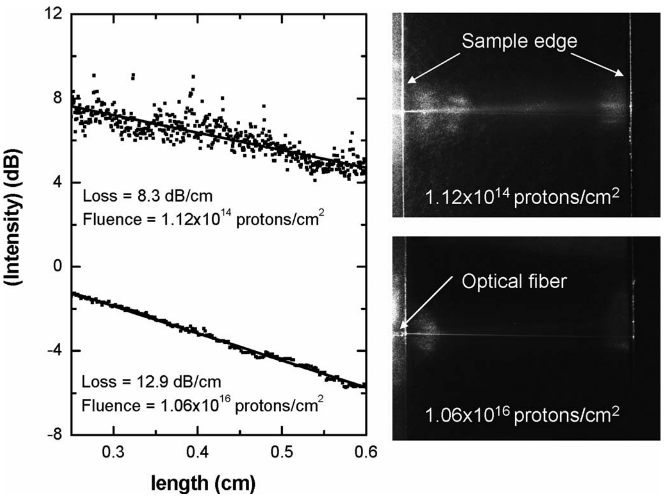

FIG. 3. Loss data measured from the waveguides fabricated with a fluence of 1×10^{14} protons/cm^2 and 1×10^{16} protons/cm^2. The loss values we obtained ranged from 8.3 to 12.9 dB/cm.

12.9 dB/cm for the lowest and highest fluences, respectively.

In the recent work of Bhardwaj *et al.*,[12] a maximum refractive index increase of 1.5×10^{-3} was measured for waveguides fabricated using femtosecond laser modification at 800 nm. It was also noted that a positive index change could be induced by a femtosecond laser without heat treatment. It is known that if a two step heat treatment is applied to Foturan™, Ag nanoparticles begin to aggregate in the exposed regions, and then a crystalline phase of Li-metasilicate is formed.[1] Since no heat treatment is applied to our samples it is unlikely that the aggregation of Ag nanoparticles in the irradiated area is responsible for the refractive index change. This result is consistent with the findings of Bhardwaj *et al.* who also tried various Foturan™ samples containing a reduced amount of silica and others that contained no Ag or Ce. All of these samples showed an increase in refractive index. It is more likely that the mechanism for the increase in refractive index at the end of range for proton beam irradiated Foturan™ is compaction. This compaction is a result of the breaking and reordering of the Si–O tetrahedra network[27] into a denser form of silica and can be directly measured using an AFM.[28] In our AFM studies of the surface of Foturan™ waveguides we observed no compaction for fluences up to 1×10^{16} protons/cm^2. The root mean squared surface roughness we measured was between 2–3 nm. Further studies are therefore required to fully understand the mechanism for the refractive index change in Foturan™.

Typically the propagation loss of proton beam irradiated waveguides in fused silica before annealing is about 3 dB/cm for fluences of the order of 1×10^{15} protons/cm^2, dropping below 1 dB/cm after thermal annealing.[18] Waveguides fabricated in fused silica using femtosecond lasers also yield a loss of just over 1 dB/cm.[29] Our results are higher, the minimum loss we achieved for unannealed Foturan™ was 8.3 dB/cm for a fluence of 1×10^{14} protons/cm^2. Future annealing studies should enable us to lower the loss of our samples as well.

In conclusion we have successfully fabricated buried, channel waveguides in Foturan™ photosensitive glass using a high energy proton beam. Seven waveguides with fluences ranging from 1×10^{14} to 1×10^{16} protons/cm^2 were obtained, all of which were able to guide light. Optical characterization results at 632.8 nm yielded a propagation loss ranging from 8.3 dB/cm for a fluence of 1×10^{14} protons/cm^2 to 12.9 dB/cm for a fluence of 1×10^{16} protons/cm^2. The propagation mode near-field technique was used to measure a positive refractive index change of 1.6×10^{-3} for a proton fluence of 1×10^{16} protons/cm^2.

The authors acknowledge the financial support from the Agency for Science, Technology, and Research (A-STAR) (Singapore) and the Ministry of Education (MOE) Academic Research Fund.

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