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Embedded photonic structures fabricated in photosensitive glass using proton beam writing

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Abstract

In this paper, we present a method that utilizes a focused beam of mega electron volt (MeV) protons to fabricate embedded photonic structures in Foturan™ glass. Both direct modification via proton beam end of range damage and post irradiation heat treatment are used to make the structures. Microfluidic channels with integrated waveguides and optical diffraction gratings are made using this technique.
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1. Introduction

There has been a trend towards the miniaturization of chemical, medical and biological diagnostic devices into highly integrated microchips often referred to as lab-on-a-chip devices [1]. These devices potentially offer a cost effective method of performing analysis on small samples with a high degree of efficiency and accuracy. For applications that require low cost solutions, disposable polymeric or glass lab-on-a-chip devices are necessary [2]. Lab-on-a-chip devices for biological applications that rely on optical techniques for high sensitivity detection are sometimes known as biophotonic chips [3]. Biophotonic chips typically require embedded optical elements in order to channel a source of excitation, for example a laser to excite florescence and to efficiently collect the emerging light for analysis. Several materials have been used for the fabrication of lab-on-a-chip devices with integrated optics including silicon, glass and polymers [1,4].

A number of recent studies have looked at using Foturan™ glass for lab-on-a-chip applications [5,6]. Foturan™ is a photosensitive glass that can be readily modified using lasers [7,8] or MeV protons [9–11]. Upon heat treatment, the exposed regions of the sample undergo a phase change from glass into a crystalline form of Lithium-metasilicate. Lithium-metasilicate has a 20 times higher etch rate in hydrofluoric acid (HF) than the surrounding glass, hence microstructures can be formed by etching the post heat treated sample [12]. The ability to directly form microstructures in this glass along with its resistance to high temperature and corrosion has made it an attractive material for microfluidic applications such as microelectrochemical reactors [13,14]. In addition to this etch contrast, irradiated and heat treated Foturan™ has also a higher density and refractive index. The ability to selectively modify the refractive index of this material on a micron scale allows for the possibility to fabricate embedded photonic structures such as waveguides [15,16] and gratings [7].

In this study, we use proton beam writing (PBW) to integrate channel waveguides with microfluidic channels and to fabricate embedded gratings in Foturan™. PBW is a new emerging lithographic technique that uses a focused beam

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of MeV protons to scan across a sample allowing for predetermined patterns to be directly fabricated in various materials [17]. Direct proton beam modification as well as heat treatment are investigated as possible methods of producing the embedded photonic structures for biochip applications.

2. Experiment

Foturan™ glass wafers purchased from Schott Mikroglas were cut and edge polished into samples approximately 10×15 mm in size and a thickness of 2 mm. Photonic structures were fabricated in the Foturan™ samples using the proton beam writing facility at the Centre for Ion Beam Applications (CIBA), National University of Singapore.

2.1. Integrated waveguide and microfluidic channel

Two microchannels in a "cross" configuration, 6 mm and 8 mm in length, were fabricated in the Foturan™ sample. A beam of 2 MeV protons focused down to approximately 1–2 μm was magnetically scanned over a width of 40 μm perpendicular to a constant linear motion provided by an EXFO Burleigh inchworm stage. The stage speed used for the scan was 4 μm/s and the proton fluence delivered to the sample was 1.25×10^{12} protons/cm^2 (2 nC/mm^2). This irradiation process leads to the formation of a latent image in the Foturan™ that can be later etched to form microchannels.

After irradiation, the sample is slowly heated to 500°C at a rate of 2 °C per minute. The sample is then held at this temperature for 1 h before the temperature is raised to 600 °C at a rate of 2 °C per minute. The sample is then held to 600 °C for a further hour before being allowed to cool to room temperature. The heat treatment process results in the modification of the irradiated region of the sample and the formation of lithium-metasilicate (Li_2SiO_3) crystallites. An optical image of the "cross" region of the sample after heat treatment is shown in Fig. 1(a). To form the microchannel, the heat treated sample was etched in a 12% hydrofluoric acid solution for 5 min and then washed and sonicated in iso-propyl alcohol for a few minutes. Preferential etching of the irradiated regions occurs because the lithium-metasilicate is etched at a rate that is approximately 20 times higher than the SiO_2 in the unirradiated regions. Fig. 1(b) shows an optical image of the microchannel post etching. The increase in surface roughness after etching is detrimental to the fabrication of waveguides by proton irradiation because any induced surface roughness will be transferred to the waveguide thus increasing the scattering loss. To overcome this problem, the surface of the sample was re-polished to achieve an optical finish before the waveguides were fabricated. An image of the sample after polishing is shown in Fig. 1(c). It has been shown by Cheng et al. [18] that the surface roughness can also be reduced by a temperature annealing process. This method will be investigated in future studies.

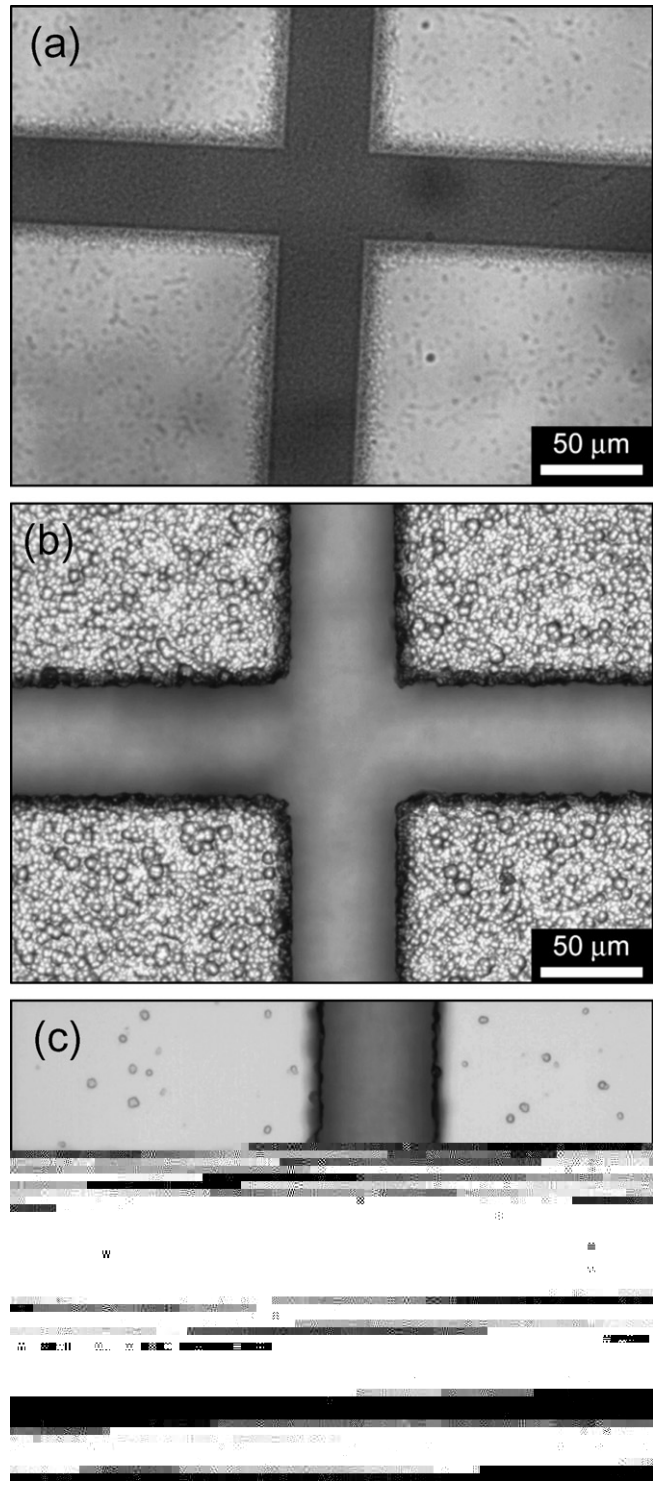

Fig. 1. (a) Optical image of the central region of the microchannel after irradiation and heat treatment. (b) Optical image of central region of microchannel after etching in 12% hydrofluoric acid solution for 5 min. (c) Optical image of the central region of the microchannel after surface polishing to remove the residual roughness.

Fig. 2(a) shows an overview of the microchannels fabricated in Foturan™. The integrated buried channel wave-

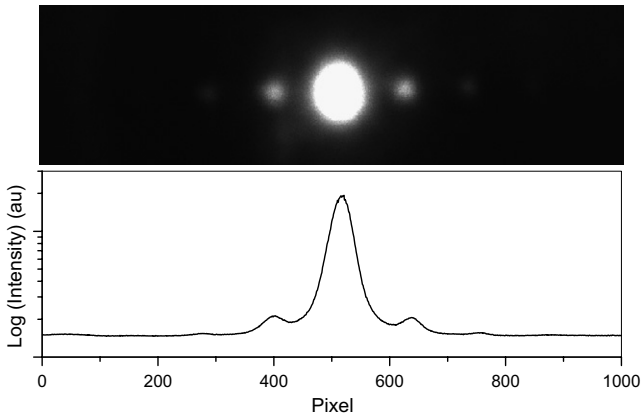

Fig. 6. Transmission diffraction pattern produced by a 473 nm diode pumped solid state laser through the 12 μm period grating fabricated in Foturan™ glass.

Fig. 4 compares the transmission spectrum of the Foturan™ glass before irradiation and after irradiation and heat treatment. The lithium-metasilicate form of the Foturan™ appears brown in color after heat treatment. The strong absorption band appearing in the blue region of the spectrum limits the use of heat treated Foturan™ for waveguiding wavelengths below 500 nm. Using proton beam modification is therefore the preferred method for fabricating integrated waveguides in the glass, especially for short wavelength laser light and fluorescence applications.

For gratings however, strong absorption and refractive index change can allow for the formation of both phase and amplitude gratings. Fig. 5 shows an optical image of a 12 μm period grating (after heat treatment) in: (a) transmission and using (b) dark field imaging. The optical bright field transmission image shows the presence of dark lines corresponding to the irradiated regions of the sample. These lines are wider than the beam spot size used in the irradiation due to the size of the lithium-metasilicate crystallites formed by the heat treatment process. These crystallites can have a diameter between 1 and 10 μm [12]. The dark field image reveals additional structure in the regions adjacent to the dark lines. This structure is not immediately obvious in the bright field transmission image however atomic force microscopy (AFM) reveals a similar structure (see Fig. 5(c)). The height difference between the irradiated region and the non irradiated region is between 15 and 20 nm. The raised region of the grating extends 5 μm either side of the irradiated line. To test the grating structures, a transmission diffraction pattern from a 473 nm diode pumped solid state laser (DPSS) was imaged. Fig. 6 shows the diffraction pattern obtained from the 12 μm period grating showing the zero and first order diffraction peaks. Further work is required to improve the diffraction efficiency and to measure the refractive index variation of these gratings.

4. Conclusion

PBW has been used to fabricate embedded photonic structures in Foturan™ glass. Waveguides and gratings have been produced using two different methods. Refractive index modification by end of range damage is the method of choice for making waveguides because it does not drastically alter the transmission properties of the material. This method also allows one to precisely control the depth of the waveguide by choosing an appropriate beam energy. Post irradiation heat treatment is better suited to fabricating gratings since this method gives a uniform refractive index change throughout the penetration depth of the proton beam. This increase in absorption at the blue end of the visible spectrum allows for the formation of both amplitude and phase type gratings. PBW can also be used to fabricate microstructures like microfluidic channels in Foturan™ by using the etch difference between the irradiated and heat treated regions and the unirradiated regions.

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