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Optimization of the annealed proton exchange method with controlled annealing for multifunctional integrated optical chip production

Fatma Yasemin Aşık,[1,2,3,*] Mutlu Gökkavas,[3] Evren Öztekin,[1,2,3] Ercan Karagöz,[1,2,3] Abdullah Ceylan,[1,2,4] and Ekmel Özbay[3,5,6]

[1]Institute of Science, Hacettepe University, Beytepe, Ankara, Turkey
[2]Department of Nanotechnology and Nanomedicine, Hacettepe University, Beytepe, Ankara, Turkey
[3]Nanotechnology Research Center-NANOTAM, Bilkent University, Ankara, Turkey
[4]Department of Physics Engineering, Hacettepe University, Beytepe, Ankara, Turkey
[5]Department of Physics, Bilkent University, Ankara, Turkey
[6]Department of Electrical and Electronics Engineering, Bilkent University, Ankara, Turkey
*Corresponding author: ykanli@bilkent.edu.tr

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The main objective of our study is to develop a new approach to the annealed proton exchange (APE) method for the fabrication of the multifunctional integrated optical chip (MIOC) used in fiber-optic gyro systems and to eliminate the loss of time and material, especially in mass production applications. In this work, self-polarized waveguides, which are the basic components of a MIOC device, were produced by the APE method and studied. With the developed method, controlled annealing trials have been carried out from a certain region on the $LiNbO_3$ substrate used in waveguide production, and the annealing time specific to the annealing process was determined. By utilizing a special setup for the hot acid process, the proton exchange process was accomplished without a sudden temperature change of the substrate. Using prism coupling measurements of the fabricated waveguides, annealing times were determined to obtain index change values suitable for 45%–50% optical throughput. Mode profiles of devices with high optical throughput that were produced by the proposed method were measured, and it was seen that devices from different proton exchange runs had similar profiles. As a result, many undamaged substrates were fabricated, and their optical quality was found to be within the expected values.

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1. INTRODUCTION

The multifunctional integrated optical device (MIOC) that is used in fiber-optic gyro (FOG) systems has a coupler design to split the light going into the optical fiber coil, a phase modulator to change the speed of the light traveling through the system, and a polarizing waveguide to support only one mode of light into the substrate [1,2]. Even though external polarizers introduce higher polarization, they are not preferred in modern FOG systems due to the increase in the dimensions of the FOG [3,4]. Therefore, studies on a waveguide with a self-polarization feature are emphasized. The waveguide, which is used in the production of MIOC devices, must be formed on a birefringent substrate to improve the polarization. Lithium niobate ($LiNbO_3$) crystal is one of the most frequently preferred substrate materials due to its birefringence and high electro-optical coefficient that make it suitable for waveguide production [5]. Titanium (Ti) diffusion and annealed proton exchange (APE) methods are widely used in waveguide production on $LiNbO_3$ crystals [6,7]. There is only one report in the literature describing the production of polarized waveguides with a polarization extinction ratio (PER) larger than 33 dB via Ti diffusion [8]. A MIOC with 33 dB PER is not sufficient by itself for the error-free operation of the FOG system; therefore an external polarizer must be placed to increase the PER. This increases both the cost and the dimensions of the system. The APE method, which is an industry standard in the production of polarizing waveguides with PER values higher than 60 dB, has two main process steps: proton exchange (PE) with an organic proton source (usually molten acid) and annealing [9–11]. In the literature, it has been stated that strong acids such as cinnamic acid [12], glutaric acid [13], toluic acid [14], stearic acid [15], pyrophosphoric acid [16], and benzoic acid [17] are generally used as a proton source. In this study, benzoic acid has been employed as an organic proton source, since it is a relatively non-toxic acid suitable for working in the range of 150–300°C. In the PE process, the substrate is kept in molten acid for a certain period of time. During this period, lithium (Li) ions in the crystal structure of

the substrate are replaced by hydrogen (H) ions in the acid. This change causes the increase of the extraordinary refractive index of the $LiNbO_3$, while the ordinary refractive index decreases to a negligible extent. As a result, it causes the light to be supported in only the extraordinary mode. These changes in the refractive indices provide the polarization of the light [18]. However, a step index profile is formed in the region where PE occurs, and this structure reduces the electro-optical properties of the crystal. In addition, problems such as instabilities in the refractive index of the waveguide channels and high propagation loss due to scattering are encountered during the PE process. In order to eliminate these problems, a subsequent annealing of the substrate for a certain period after the PE is suggested [19]. The annealing process helps in eliminating negative structural effects by providing a gradual index change in the proton exchanged region. In the annealing process, the annealing time is of great importance in terms of the formation of the index change depth, and even if these times are calculated beforehand, due to the nature of the APE process, it is not guaranteed to have the same effect in every subsequent process. For mass production, it is essential that the annealed substrates meet performance and yield criteria. If the substrate is annealed more than the desired index change depth, it becomes useless, causing unnecessary resource consumption and loss of time. There is a need for a new method that will eliminate these drawbacks and solve the problem.

In this work, a new production technique for a MIOC that will be used in a FOG system is proposed. The proposed production technique will eliminate time and material loss during the waveguide production process. In the technique proposed, the annealing time that is specific to each annealing process was determined by using controlled trials from a certain region on the substrate. Details of the process have been discussed based on PC and optical throughput characterizations results.

2. METHOD

There are two steps of waveguide formation by the APE method on $LiNbO_3$ crystal. The first step, which is the PE in a proton source, is followed by an annealing step. The PE takes place while the substrate is kept in the proton source at high temperatures (150–250°C) for periods of 30 min to 8 h. During the process, an acid resistant Ti metal mask is utilized. In this study, $LiNbO_3$ crystal was coated with Ti metal to form the mask. The Ti-coated $LiNbO_3$ crystal was patterned using photolithographic methods with the designed mask shown in Fig. 1. Large non-patterned rectangular (1 cm × 2 cm) areas (which can be seen as parts A, B, C, or D in Fig. 1) are reserved at the edge of each substrate to allow for diagnostic measurements. The Ti metal on the crystal was etched to form 7 μm wide strip areas. A mixture of H_2O_2: HF: DI water prepared at a ratio of 1:1:30 was used for etching Ti. PE was performed on all areas of the $LiNbO_3$ crystal that are unprotected by the Ti mask.

The PE process was performed in a custom designed system as shown in Fig. 2. A heating stirring mantle is used to keep the acid dissolved in the beaker at a constant temperature for prolonged periods. A custom cover made out of Teflon is employed to prevent the acid vapor escape, and a substrate holder with a manual elevator mechanism is designed to immerse the substrate in the

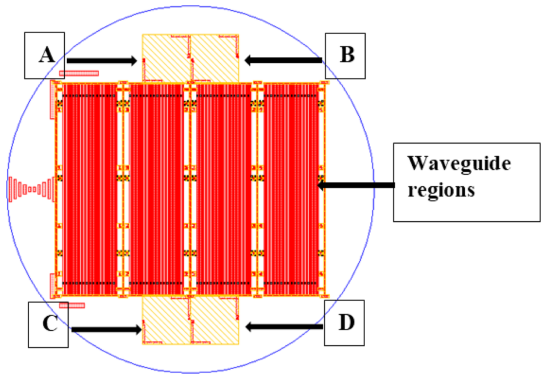

Fig. 1. Waveguide and diagnostic regions on the designed mask.

acid to start the process when the desired temperature is reached and lift the substrate out of the molten acid to end the PE process. The substrate prepared for the PE process is mounted to the substrate holder and attached to the elevator mechanism that is operated from outside, and the elevator mechanism is mounted on the custom cover and kept in the waiting area. The beaker filled with benzoic acid, which is solid at room temperature, is placed in the heating mantle, and the cover is closed on the beaker. By increasing the temperature, benzoic acid melts and its temperature is increased. Meanwhile, the substrate is kept in the waiting area in the cover [Fig. 2(a)]. In this way, while the environment reaches thermal equilibrium, it is ensured that the substrate is gradually heated up without experiencing a sudden temperature change. After the environment in the beaker reaches thermal equilibrium, the substrate being kept in the waiting area is moved from outside with the help of an elevator mechanism in a closed environment without opening the cover and immersed in the molten acid [Fig. 2(b)]. Upon reaching the desired PE period, the heating mantle temperature of the heating mantle is set to room temperature, and the substrate is pulled back to the waiting area above the acid and kept there until the environment reaches thermal equilibrium. In this way, it is ensured that each substrate is not removed directly from the hot acid to room temperature. The formation of cracks is prevented since the substrate is not exposed to abrupt temperature changes. In addition to that, the release of the acid vapor into the environment is prevented. The substrates are completely removed from the closed environment when the environment temperature reaches room temperature.

A PC measuring device (Metricon company) is used to evaluate the APE process [20]. As a result of the PC measurement made after the annealing phase, the one-dimensional (1D) refractive index profile versus depth (increase in the index on the surface, diffusion depth, etc.) is obtained using the WKB approximation [21,22]. The task of the prism in the prism coupling device is to convert the light from the direction perpendicular to the crystal surface to the direction parallel to the crystal surface. In this way, light is coupled to the waveguide layer via the prism. Normally, when the light reflecting from the prism surface and falling on the detector is matched to the waveguide layer, the amount of light decreases. The effective mode index of the waveguide is calculated using the angle information at which the light is coupled to the waveguide. A depth

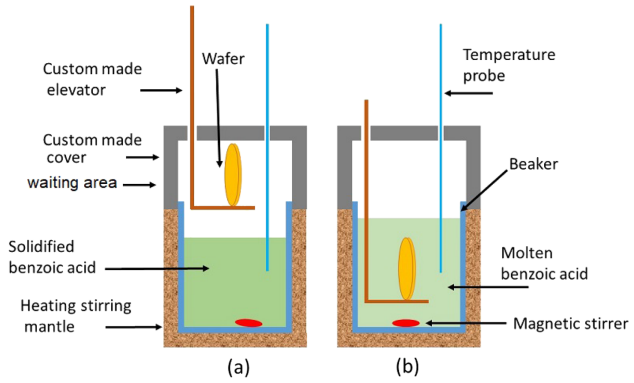

Fig. 2. PE process system. (a) Before the acid was molten and the wafer was lowered. (b) After the acid was molten and the wafer was immersed into the acid.

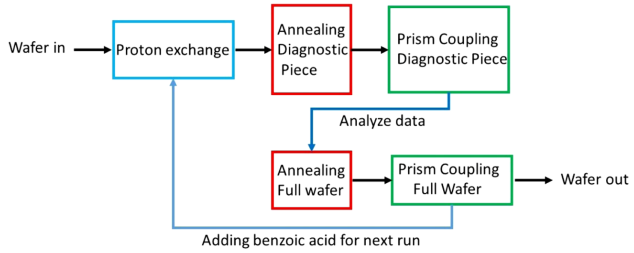

Fig. 3. APE process flow chart.

dependent diffraction index profile is obtained by using the WKB approximation using more than two mode indices.

Figure 3 shows the APE process flow chart. First of all, PE treatment is carried out by keeping the substrate as a whole in acid at certain temperatures for a certain period of time. A, B, C, or D regions cut from the edge of the substrate are annealed at certain temperatures for a certain period of time, and the index change value is obtained by performing PC measurements. Based on our current studies, the index change values should be around 0.025–0.030 for a 7 μm wide waveguide to have the desired optical throughput (low loss). Subsequently, waveguide regions are annealed to obtain this desired index change value, where the annealing time is determined according to the result obtained from the diagnostic piece results. As the next step, optical measurements of the waveguides in the waveguide region are completed.

It has to be noted that this method requires a piece of the wafer to be cut and processed in order to determine the actual annealing time, and this approach adds to the complexity of the process. However, the benefit gained in terms of yield and performance increase is such that this extra step is well justified. It is also possible to implement the described method without cutting the substrates. One way of achieving a similar scenario is to anneal the wafer in two subsequent steps with a PC measurement after the first step and adjust the anneal parameters of the second step according to the result of the first anneal step. Or alternatively, multiple wafers could be processed in a single run with only one of the wafers to be cut and analyzed, whereas the remaining wafers would be processed in a single step.

It was observed that a gradually lowering index change value was obtained when the PE process for subsequent substrates was sequentially carried out with the same parameters using the same beaker. The proton concentration of the acid, which is redissolved and reused each time, affects the PE process and results in lower index change. Using the result obtained from the diagnostic anneal piece, the annealing time for the full substrate is determined and benzoic acid is added to the beaker for the next PE process to keep the hydrogen concentration at the desired level for each PE process.

Figure 4 shows the PC measurement results of diagnostic regions from three different wafers that were proton exchanged at 210°C for 3 h and annealed at 400°C for 40 min in the same proton source. It is seen that the index change gradually decreases from the first run (S1 to the third run S3). In order to prevent this change and to maintain the stability of the PE environment, benzoic acid was added to the beaker at values around 0.5% before each PE process.

3. MEASUREMENTS

In order to have a throughput of 45%–50%, for the 7 μm wide waveguides proton exchanged at 210°C for 3 h, the index change values should be between 0.025 and 0.030. To obtain index change values in this range, the index change values

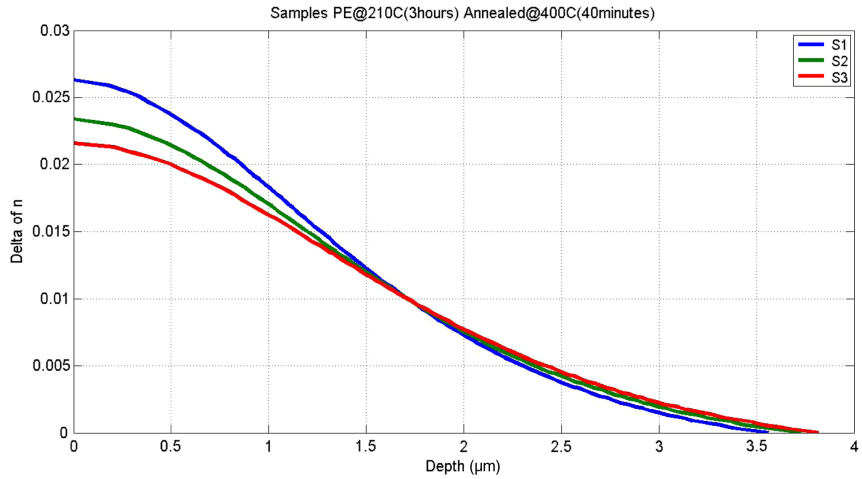

Fig. 4. Sequential PE operations. First, sample S1 was processed, followed by S2, and finally by S3.

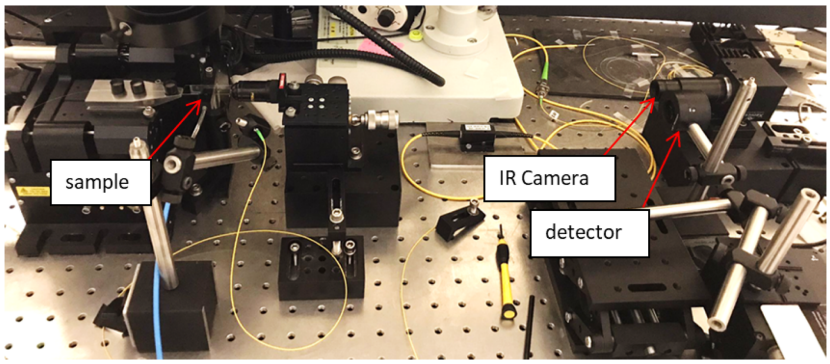

Fig. 5. Optical throughput and mode profile measuring setup.

Table 1. Annealing Periods of the Substrates

Substrate	Δn of Diagnostic Regions after 40 min Annealing	Annealing Periods of Substrate (min)	Δn after Annealing
S1	0.0265	38	0.028
S2	0.0235	35	0.028
S3	0.0215	30	0.027
S4	0.0350	53	0.029
S5	0.0325	48	0.026
S6	0.0300	45	0.027
S7	0.0285	40	0.028

measured for the fabricated substrates and the annealing periods determined for these substrates are listed in Table 1. It is observed that all of the substrates S1–S7 had surface Δn values in the desired range.

In order to make optical throughput measurements, the input–output surfaces of the devices must be of a certain optical quality. For this purpose, the device groups cut with a dicer were stacked, and the input and output surfaces were lapped and polished. Both optical throughput measurements and mode profile measurements have been carried out (in the same setup shown in Fig. 5) with the help of a six-axis stage. For both the optical throughput measurements and mode profile measurements, an amplified spontaneous emission (ASE) light source operating around 1550 nm center wavelength with a 30 nm bandwidth was used. The optical throughput measurements of the devices with a 7 μm wide waveguide were carried out, and it was observed that for all the substrates studied, similar optical throughput values ranging from 45% to 50% were obtained. The mode profile measurements were performed with the same ASE light source described above that is used in FOG systems. The IR camera used in the mode profile measurements is sensitive in the same wavelength region of interest. The infrared image showing the mode profile of a waveguide with a width of 7 μm and an index change value of 0.027 is presented in Fig. 6.

Processing the images taken from the measurements made with this method and using the MATLAB program, the mode shapes of the S4, S6, and S7 samples were shown in Fig. 7 (top left), (top right), and (bottom left), respectively, where the contour plots correspond to the power values at the 90%, 80%, 70%, 60%, 50%, and 40% of the maximum amplitude.

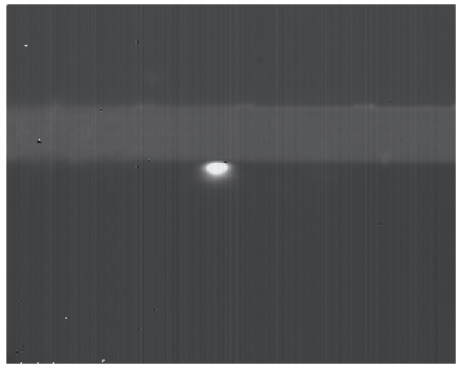

Fig. 6. Mode profile image of 7 μm wide waveguide.

The comparative graph in Fig. 7 (bottom right) shows the contour plots corresponding to 90% and 40% of the maximum for the three devices depicted in Fig. 7 (top left), (top right), and (bottom left). The annealing times for these substrates are 53 min, 45 min, and 40 min, respectively. Although the annealing parameters are different, the produced waveguides appear to have reasonably similar mode profiles.

4. CONCLUSION

In order to increase the performance of fiber-optic gyroscope systems, MIOC devices with self-polarizing waveguides are preferred. In this study, a controlled APE method used for the production of self-polarized waveguides is proposed. With the proposed controlled annealing and PC measurement method for the two-step APE process, it is possible to compensate the variations occurring in the first step of the process before proceeding to the next step. This will decrease the production cost and time while improving the quality of the output. In order to avoid variations occurring in the first step of the process, 0.5% benzoic acid was added before each PE step to keep the proton concentration constant. In the method proposed, the annealing time of the substrate is determined using controlled annealing trials from a certain region on the substrate used in the production of the waveguide. In addition, the stability of the proton source is preserved, and the efficient use of both materials and time, which is critical especially in the multiproduction environment, is ensured. We achieved the aimed index change values (0.025–0.030 for 7 μm wide waveguide) that are necessary for

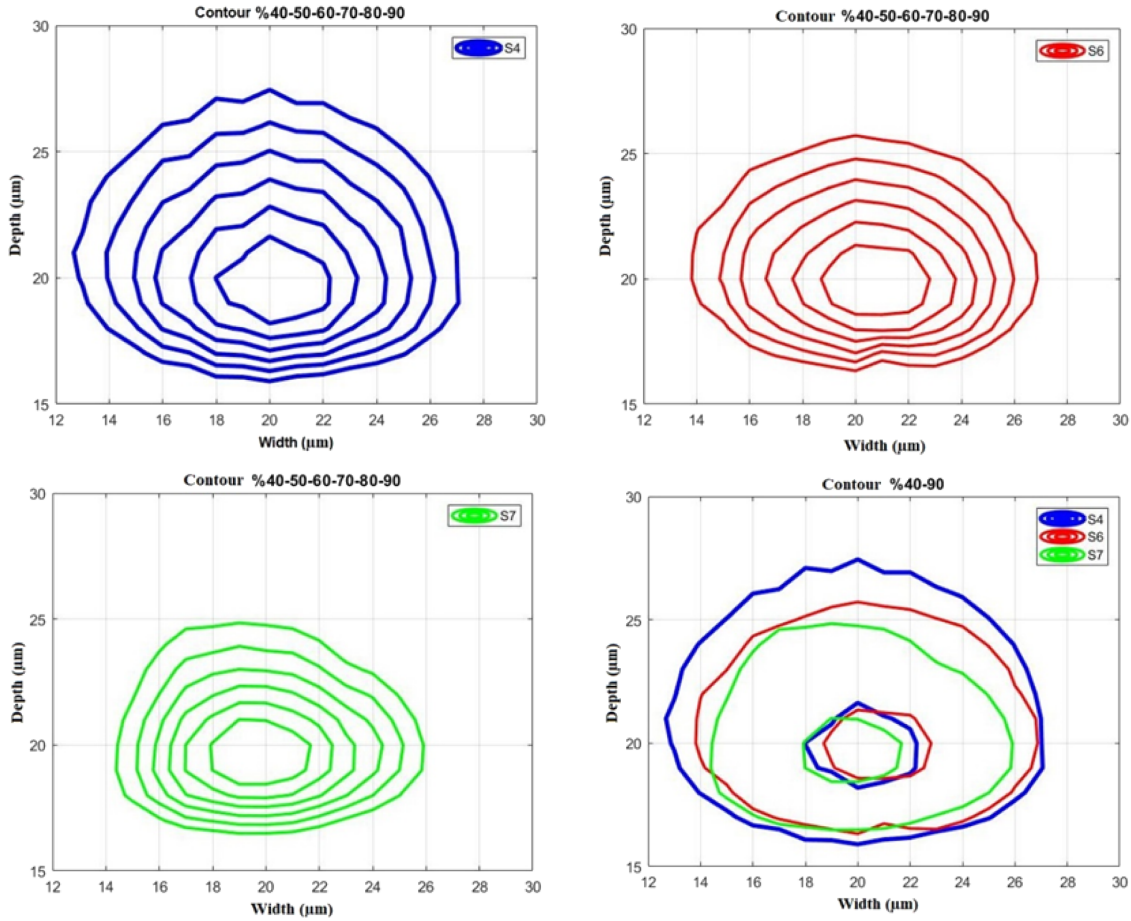

Fig. 7. (Top left) Mode profile of S4. (Top right) Mode profile of S6. (Bottom left) Mode profile of S7. (Bottom right) Comparative mode profiles of S4, S6, and S7.

high optical throughput from the waveguide. We measured the mode profiles of the fabricated waveguides and found that they had similar mode profiles although the annealing parameters were different.

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Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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