PHOTOBLEACHING FOR THE FORMATION OF NONLINEAR OPTICAL POLYMER WAVEGUIDE DEVICES

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Abstract — A simple but realistic kinetic model has been developed to delineate the refractive index profiles formed by photochemical reaction in nonlinear optical polymers. The effects of the absorption due to the unconverted reactant and the photoproduct are included in the model. The parameters required in the model are obtained from simple transmission experiments. The experimental results are consistent with the model. The refractive index profile is steeper when nonlinear optical polymeric materials are bleached by light with higher absorption. The rate of the bleach depth change becomes slower as the bleaching proceeds. Small absorption of bleaching light due to the photoproduct has significant effect on the resulting refractive index profiles. The photobleaching process is shown to be accelerated by the addition of a photosensitizer. Photobleaching time has been effectively reduced by a factor of 3-5. Linear and nonlinear optical properties of the polymer were little affected by the addition of the photosensitizer. It has also been demonstrated that the photobleaching technique can be utilized to tune the initial state of a directional coupler switch after completing the fabrication of the device. It was shown that successive cross states are passed from the initial cross state by selective bleaching of the gap region. The evolution of the refractive index profiles is successfully applied to predict the evolution of output state.

INTRODUCTION

Electro-optic (EO) poled polymers have attracted large attention during the last decade because of their application potential for information processing [Kim and Lee, 1993; Lytel, 1990; Hornak, 1992]. Organic EO materials offer significant advantages over conventional inorganic materials such as $LiNbO_3$ or semiconductors in several key areas for EO waveguide devices. These include large EO coefficient, low dielectric constant with negligible dispersion from D.C. to optical frequency, fabrication flexibility and simple processibility, which lead to high bandwidth modulators and switches, and high density integration of many functional devices either in monolithic or hybrid forms on a single wafer.

Much research effort has been poured into molecular engineering to improve the material properties such as linear and nonlinear optical properties and thermal stability with great success during the last several years [Eich et al., 1989; Wu et al., 1991; Lee et al., 1993; Hwang et al., 1994]. Potentials of nonlinear optical polymer devices have been demonstrated with test devices such as electro-optic modulators with tens of GHz modulation bandwidth [Girton et al., 1991; Teng, 1992], multilevel modulators [Tumobillo and Ashley, 1993] and switches [Hikita et al., 1993], polarization converters with high extinction ratio [Oh et al., 1995; Hwang et al. 1995; Kim et al., 1995] and hybrid integration of polymer external modulator with semiconductor laser diode [De Dobblaere et al., 1994]. There are, however, still many areas to refine from the device design to process in order to realize EO polymer waveguide devices for the application to information processing.

Various techniques such as poling [Thackara et al., 1988], photobleaching [Aramaki et al., 1990; Rochford et al., 1989; Diemeer et al., 1990; Van Eck et al., 1991; Van Tomme et al., 1991], photolocking [Franke, 1984; Chakravorty, 1993] and reactive ion etching [Hikita et al., 1993] have been employed to form channel waveguide with nonlinear optical polymers. Among them photobleaching technique has been widely used because of its simplicity and controllability [Aramaki et al., 1990; Kim et al., 1994]. The technique exploits the property that many nonlinear optical chromophores are bleached out upon exposure to ultra violet (UV) or visible (VIS) light. The bleaching lowers the refractive index of the material, which in turn can be utilized to guide a light by total reflection, so called waveguide. Many nonlinear optical polymer waveguide devices have been fabricated by this method [Girton et al., 1991; Teng, 1992; Tumobillo, 1993].

Since the performance of a device can be improved by the precise control of refractive index profiles, detailed information on the refractive index profile formed by photobleaching is important for modeling the propagation of light through a polymer thin film waveguide and for the design of a waveguide device. However, less attention has been paid to the refractive index profile in the film formed by the technique. Refractive index profiles formed by photobleaching will be delineated theoretically and experimentally in chapter 2. Some devices are required to bleach out thick films. In chapter 3, we will describe how to accelerate the photobleaching in nonlinear optical polymers to overcome the slowness of the photobleaching process. Up to now photobleaching has been mainly utilized to form channel waveguide. We will demonstrate in chapter 4 that utilities of the photobleaching technique can be extended to improve the device characteristics of an electro-optic directional coupler switch after the completion of the

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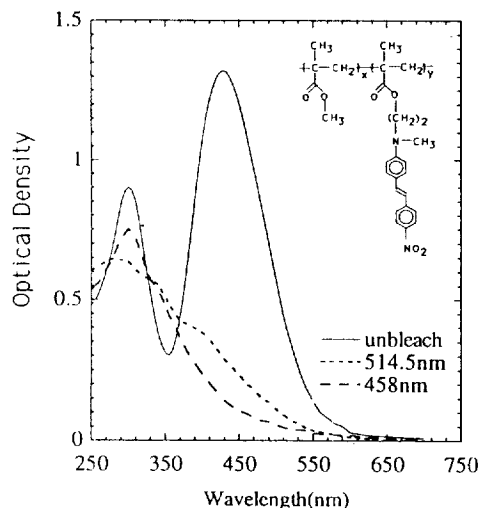[†] To whom all correspondences should be addressed.

Fig. 1. Absorption spectra of the unbleached (solid) and bleached nonlinear optical polymer shown in the inset with 514.5 nm (dotted) and 458 nm (dashed) lights.

device fabrication. Finally some conclusions will be drawn in chapter 5.

DELINEATION OF REFRACTIVE INDEX PROFILES IN NONLINEAR OPTICAL POLYMER THIN FILMS FORMED BY PHOTOBLEACHING

The photobleaching process of isotropic dye molecules in solid matrix was analyzed by many authors [Kaminow et al., 1972; Tomlinson, 1971; Copolla and Lessard, 1991] and similar equations have been used to delineate the refractive index profiles [Horn et al., 1991; Moshrefzadeh et al., 1993]. Most of the analyses were performed for a one photon process based on the assumptions that all the reactant transforms to product by the photoreaction and the photoproduct does not absorb incident bleaching light.

Validity of the assumptions used in the above analyses depends on the material in use and the bleaching light. For instance, the assumptions are not applicable to the poly(methyl methacrylate) based side chain polymer with a stilbene derivative as a pendant group (P2ANS) shown in the inset of Fig. 1. The material is one of the most widely used nonlinear optical polymers for waveguide devices. The absorption spectrum of the material bleached with 514.5 nm light exhibits some indication of the existence of reactant molecules even after the completion of the bleaching. The absorption spectrum of the bleached film by 458 nm light shows no indication of the existence of the reactant molecules, but the absorption due to the product is not negligible.

In this chapter, we present a simple but more general model to delineate the refractive index profile formed in dye attached polymers [Kim et al., 1994; Zyung et al., 1994]. The model considers the incomplete transformation of the reactive chromophores and the absorption due to the photoproduct. Experimental results appear to be consistent with the model. We will also show that the information necessary for modeling the index profile is obtained from a simple transmission experiment.

1. Theory

Consider a slab of thickness L containing photochemically reac-

tive chromophores. At time $t=0$, only species 1 is present and uniformly distributed with number density N_0. During exposure, incident light causes conversion from species 1 into species 2 by following reaction.

$$N_1 + \hbar\omega \rightarrow N_2 \tag{1}$$

We postulate that not all of the species 1 transforms into species 2 and the species 2 also absorbs bleaching light. For the one photon process, the number density of species 1 and the light intensity I as a function of time and position are related by

$$\frac{\partial N_1}{\partial t} = -\phi\varepsilon(N_1 - N_1^\infty)I \tag{2}$$

$$\frac{\partial I}{\partial x} = -[(\varepsilon_1 - \varepsilon_2)N_1 + \varepsilon_2 N_0]I \tag{3}$$

where ϕ is the quantum efficiency, ε_i the absorption cross section of species i, N_0 the initial number density of species 1, and the N_1^∞ the number density of species 1 unconvertible by bleaching light. The initial and boundary conditions are, respectively,

$$N_1(x, 0) = N_0 \tag{4}$$

$$I(0, t) = I_0 \tag{5}$$

where I_0 is the incident intensity of bleaching light.
The above equation can be simplified to

$$\frac{\partial f}{\partial x} = (\alpha_1 - \alpha_2)f(1 - f) - \alpha^\infty f \ln(f) \tag{6}$$

with the boundary condition at $x=0$

$$f = \exp(-\beta t) \tag{7}$$

where f is the dimensionless concentration defined as $(N_1 - N_1^\infty)/(N_0 - N_1^\infty)$, and α_i and α^∞ the absorption coefficients defined by $\varepsilon_i N_0$ and $\varepsilon_1 N_1^\infty + \varepsilon_2(N_0 - N_1^\infty)$, respectively, and β the time constant related to quantum efficiency by $\beta = \varepsilon_1 \phi I_0$. Since an independent variable time t appears only in the boundary condition, it can be treated as a parameter. The differential equation can be handled as an ordinary differential equation on x with a parameter t. The intensity profile is obtained from Eq. (3):

$$I = I_0 \exp\{-\int_0^\infty [(\alpha_1 - \alpha^\infty)f + \alpha^\infty]dx'\} \tag{8}$$

and transmittance through the film is

$$T(t) = \exp\{-\int_0^L [(\alpha_1 - \alpha^\infty)f + \alpha^\infty]dx'\}. \tag{9}$$

Assuming that the change of the refractive index is proportional to the change of the number density of species 1, the refractive index profile at exposure time t is

$$n = n_0 - (n_0 - n_f)(1 - f) \tag{10}$$

where n_0 and n_f are the refractive indices of the unbleached film and fully bleached film, respectively. There are three parameters to be determined: α_1, α^∞, and β. α_1 and α^∞ can be determined from a transmission experiment by

$$\alpha_1 = -\frac{1}{L}\ln T(0) \tag{11}$$

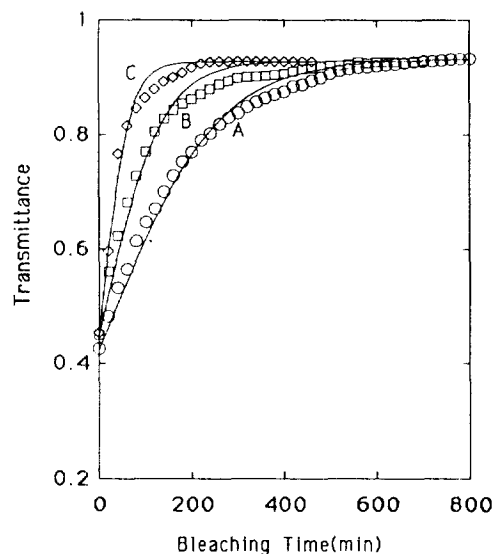$$\alpha^\infty = -\frac{1}{L}\ln T(\infty) \tag{12}$$

Fig. 2. Transmittance through the 0.25 μm thick films vs. time for various power of bleaching light (514.5 nm wavelength): (a) 50, (b) 100, and (c) 200 mW.

Table 1. Parameters deduced from transmission experiments for different bleaching powers of 514.5 nm and 458 nm lights

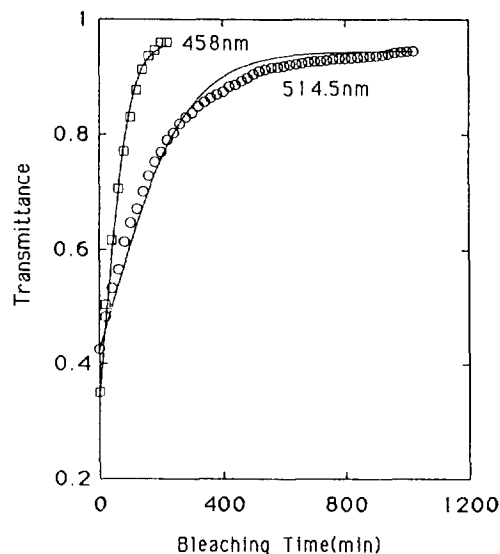| Wavelength (nm)	Incident power (mW)	α_1 (10^4 cm^{-1})	α^∞ (10^3 cm^{-1})	β (10^{-4} s^{-1})	ϕ (10^{-5})
514.5	50	3.4	2.8	1.5	4.3
514.5	100	3.2	3.0	2.7	4.3
514.5	200	3.2	3.0	5.7	4.6
458	50	9.5	3.6	4.4	5.3

Fig. 3. Transmittance through the 0.25 and 0.1 μm thick films vs. bleaching time for two different bleaching lights of 514.5 nm (□) and 458 nm (○) with 50 mW, respectively. The lines are the best fittings using the Eq. (8) with the parameters in Table 1.

where L is the thickness of the polymer film, and T(0) and T(∞) the transmittances of bleaching light through the film at time $t = 0$ and ∞, respectively. The time constant β is determined as the value giving the best fitting with the experimental transmittance.

2. Experimental

Experiments were performed using the P2ANS to confirm the analysis. The material was obtained from Hoechst Celanese Co. and spin coated on quartz substrates. Wavelengths of 458 and 514.5 nm generated from an Ar$^+$ ion laser (Spectra Physics 2020-5) were used as the bleaching lights. The 458 and 514.5 nm are located near the absorption maximum and absorption edge, respectively as shown in Fig. 1. The thicknesses of the films were 0.1 μm and 0.25 μm for the transmission experiment at 458 nm and 514.5 nm and 4 μm for the refractive index measurement, respectively. Transmission of bleaching light through the film was obtained as a function of bleaching time for various incident powers of the light. The incident light was expanded to the diameter of 1 cm and passed through an aperture which was placed in front of the sample. The aperture of about 1mm diameter passed only the central part of the laser beam, in which the intensity of the radiation is considered to be constant. The transmitted light was detected by Si-photodiode. Exposure of the film was periodically interrupted to measure refractive index profiles. The refractive index profiles were obtained using the inverse Wentzel-Kramers-Brilliuin method with measured mode indices by the prism coupling technique [White and Heidrich, 1976]. A wavelength of 830 nm from a laser diode (Larmax Inc., 6491) was used for the refractive index measurement. The refractive indices of the fully bleached and unbleached films were measured separately by the prism coupling method (m-line technique) and were found to be 1.576 and 1.655, respectively.

3. Results and Discussion

The results of the transmission experiments at the wavelength of 514.5 nm are shown in Fig. 2 for various incident powers of the light. The time required to achieve a certain transmittance is inversely proportional to the power of the incident light, suggesting that the photobleaching is a one photon process. The final

transmittances achievable by the photobleaching are almost constant for various incident powers. The best fitting curves to the transmission data using the Eq. (6) are shown in Fig. 2. As seen in the figure, our simple kinetic model describes the experimental transmission data well. The parameters deduced from the transmission experiments are summarized in Table 1. Absorption coefficients of the reactant and the photoproduct, and the quantum efficiency at the wavelength are constant independent of the intensity of the bleaching light within experimental error, demonstrating that the mathematical formalism used in this study is valid.

Transmittance when irradiated with 458 nm is also described by the model very well as shown in Fig. 3 where it is compared with 514.5 nm at the same power of 50 mW. The parameters deduced from the experiment are summarized in Table 1. It is interesting to note that the quantum efficiency at 458 nm is almost the same as at 514.5 nm even though the absorption cross section at 458 nm is almost three times of that at 514.5 nm. This indicates that the conversion probability of the photoabsorbed molecules to photoproduct is almost the same independent of the wavelength of the exciting light.

Refractive index profiles resulting from bleaching with an incident optical power of 50 mW for both wavelengths are obtained using the parameters in Table 1 and are compared with the experimental ones in Fig. 4. The figure demonstrates that the model
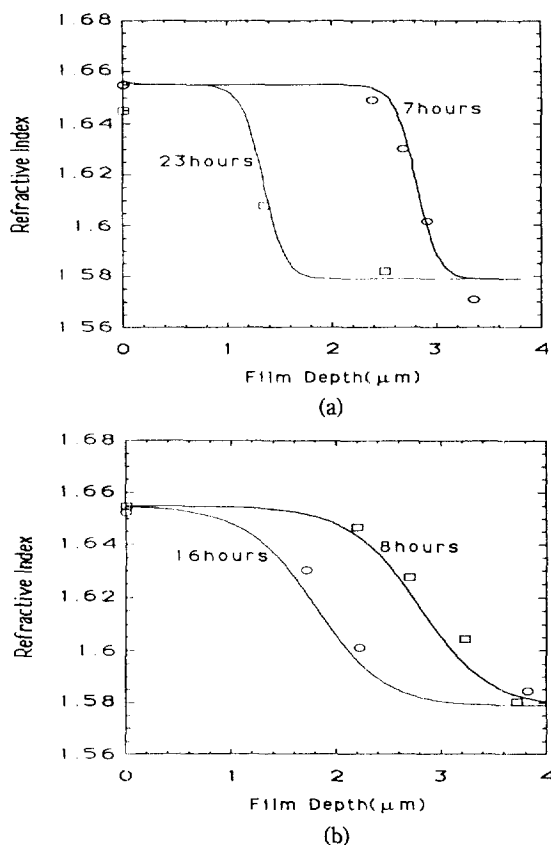
Fig. 4. Refractive index profiles formed by photobleaching with 50 mW of (a) 514.5 nm and (b) 458 nm for various times are compared with theory (solid lines).

delineates the refractive index profiles very well. Graded index profiles are obtained as expected and they are well delineated by the model. The profiles obtained from the bleaching with 514.5 nm are more graded with position than those bleached with light of 458 nm. The steepness of the profiles is expected to depend on the wavelength of bleaching light. Since the light intensity with higher absorption will drop more rapidly than that with lower absorption as the lights propagate through the film, the refractive index profiles formed by the bleaching light with higher absorption are expected to be steeper than those with lower absorption.

One important feature of the solution is that the bleaching rate becomes slower as the bleaching proceeds, as shown in Fig. 5. This can be attributed to the absorption of the bleaching lights by the photoproduct and/or the unconvertible reactant. The absorption coefficients of the fully bleached films are only one tenth or one thirtieth of the initial absorption coefficients at the bleaching wavelengths of 514.5 nm and 458 nm, respectively. The analysis suggests, therefore, that even the small absorption due to the photoproduct should not be neglected to delineate the refractive index profiles. The degree of the retardation of bleaching rate is higher for 458 nm bleaching light than 514.5 nm due to the higher final absorption by the photoproduct and/or unconvertible reactant at 458 nm.

ACCELERATED PHOTOBLEACHING OF NONLINEAR OPTICAL POLYMERS

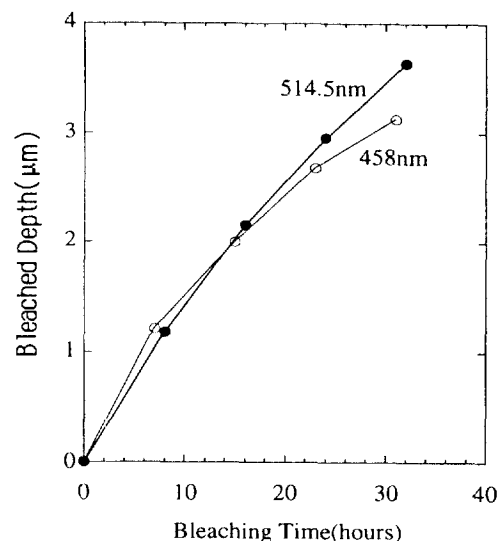Photobleaching offers simple processibility and precise control-

Fig. 5. Change of bleached depth with time when polymer thin films are bleached with 50 mW of 514.5 nm (●) and 458 nm (○) lights. The bleached depth is defined as the distance from the surface to the point where the change of the refractive index is half of the total change.
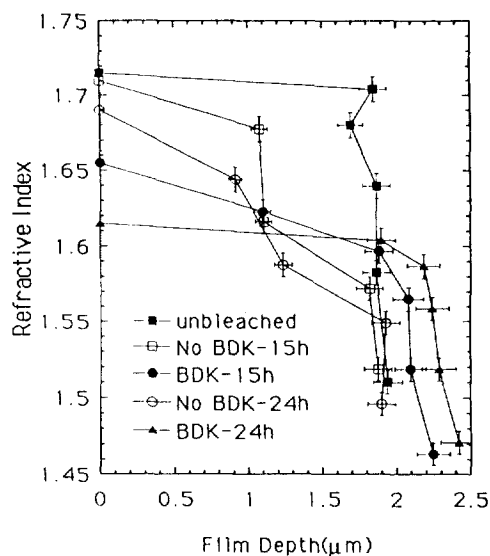

Fig. 6. Evolutions of refractive index profiles of copolymer thin film treated with photosensitizer and pure P2ANS thin film. Film depth is the distance from the film surface and the films were exposed from the back side.

lability as mentioned before. However, one drawback of the technique is the slowness of the photoreaction to bleach out the film. It takes long time, typically on the order of tens to hundreds of hours to bleach out a few μm thick side-chain polymer thin films using a photo mask aligner with typical intensity of 10 mW/cm² of 366 nm UV light. For some devices it is required to bleach out all the way through a few μm thick films. Therefore it is desirable to find a way of reducing the bleaching time. We have achieved it by adding a photosensitizer into the NLO polymer thin films without any significant deterioration on the nonlinear optical properties of the film and on the waveguiding proper-

ties.

The photo-sensitizer we used is BDK (Aldrich Co.). The material is sensitive in UV region, within which the spectral region of a mask aligner (Karl-Suss MJB3) falls. The material was added into P2ANS solution. The molar ratio of BDK to stilbene group was 1 : 1. The solution was spin coated on quartz substrates and photobleached under the mask aligner with the intensity of 10 mW/cm^2 in the UV region. The photobleached samples were thermally annealed at 100°C for 48 hr to evaporate the residual BDK. BDK in the unilluminated region can be removed by thermal annealing with little effect on nonlinear optical moieties. It was confirmed by infrared (IR) spectra, refractive index measurements and electro-optic coefficient measurements, which show little difference between the annealed samples without photobleaching and pure P2ANS. Details of the experiments were reported in ref. 34.

The evolution of the refractive index profiles of P2ANS thin films with and without BDK upon photobleaching is shown in Fig. 6. The sample containing BDK almost bleached out all the way through the 2 μm thick film after 15 hr bleaching. On the contrary, the sample containing no BDK was bleached up to a little bit less than 1 μm even after 24 hr exposure under the UV light. It takes about 60 hr to bleach out 2 μm thick films of the P2ANS. Therefore addition of BDK into the film is estimated to accelerate the photobleaching about 3-5 times.

Channel waveguides were fabricated using the P2ANS/BDK material system on Si substrates. PMMA was used as both upper and lower buffer layers. The thicknesses of the layers are 2 μm and that of the guiding layer is 0.9 μm, respectively. Even without optimization of the channel waveguide dimension, good propagation of light through the guide and good single mode patterns were obtained, demonstrating the potential of the technique used in this study [Zyung et al., 1994].

POST-PHOTOBLEACHING TO TUNE DIRECTIONAL COUPLER TYPE SWITCH

Directional coupler switch is one of the most important integrated optical devices for optical communications, whose device structure is schematically shown in Fig. 7. The polymer waveguide device consists of triple layer stack. The middle layer guides light wave and possesses electro-optic property. Light coupled in one arm at one side can be switched to bar state (A in Fig. 7) or cross state (B in Fig. 7) depending on the voltage applied to the electrode on top of waveguides. Coupling of light can be expressed by the coupling constant of the directional coupler switch. In order to have a good switching extinction, the initial output state must be in cross state. To satisfy the condition, the coupler length L should be equal to [Yariv and Yeh, 1984]:

$$L = (1 + 2m)L_c = (1 + 2m)\frac{\pi}{2c} \tag{13}$$

where m is an integer, L_c the coupling length, and c the coupling constant. The coupling constant depends on the many waveguide parameters such as propagation constants, damping constants, and gap width, etc.. In real fabrication processes, it is almost impossible to control all the parameters exactly to satisfy Eq. (13). By the reason, it is valuable to find a method which can control the coupling length independently with the fabrication process.

We will propose and demonstrate a method for the first time
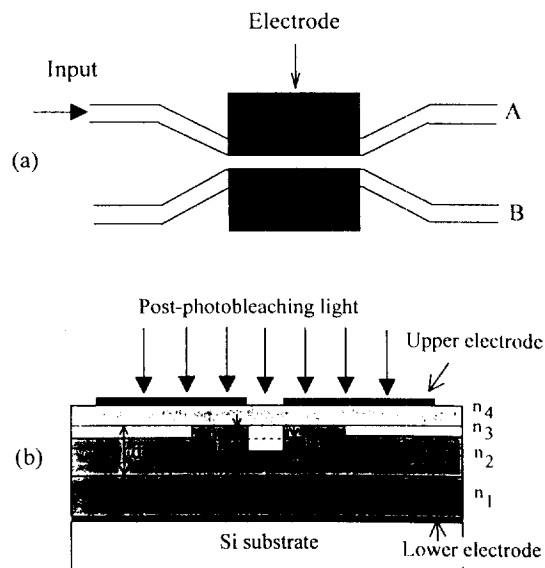

Fig. 7. Schematic diagrams of a directional coupler switch: (a) top view and (b) cross sectional view. In (b) post photobleaching process is schematically shown.

to the authors' knowledge, so called the post-photobleaching technique, which can control the initial output state of an EO polymer directional coupler by controlling the coupling constant after finishing the device fabrication process. The idea comes from the fact that many EO polymers can be bleached out upon irradiation of UV or VIS light, resulting in lowering of refractive index as described before. Since the coupling constant varies with the effective refractive index of the planar waveguide in the gap region, it can be controlled by the selective photobleaching of the gap region after completion of the device fabrication. If the photobleaching is performed while monitoring the outputs from the two waveguides, the output state can be tuned exactly to the cross state by the method.

The post-photobleaching process are schematically shown in Fig. 7(b). The patterned switching electrodes are located on both waveguide channels, so that the gap region is open to the post-photobleaching light. As the bleaching proceeds in the gap region, the refractive index of the region becomes smaller and the coupling constant becomes smaller too. Since the initial output powers of the cross and the bar state in a symmetric directional coupler can be expressed by

$$p_{cross}/P_{in} = \sin^2(cL) \tag{14}$$

$$P_{bar}/P_{in} = 1 - \sin^2(cL), \tag{15}$$

initial output states with no bias can be tuned by controlling the post-photobleaching depth of the gap region. Details of the mathematical description of the method has been described elsewhere [Hwang et al., 1995].

P2ANS was used as the electro-optic polymer again, i.e., the guiding layer. The lower cladding layer was the same polymer as the guiding layer but with smaller dye concentration and the upper cladding layer was UV curable epoxy (NOA 61). After successive spin coating of the lower and the guiding layers with the thicknesses of 2.5 μm and 3 μm on gold coated Si wafer, respectively, a directional coupler type channel waveguide was formed
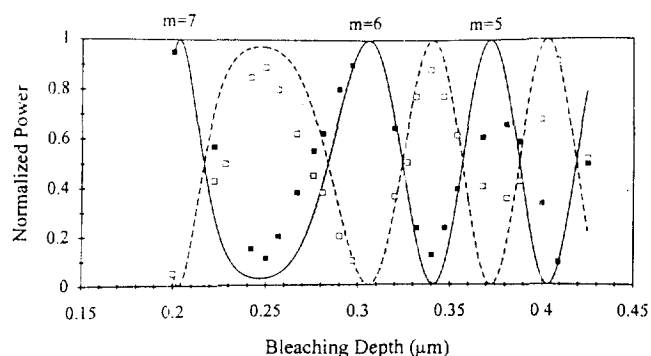
Fig. 8. The experimental output power evolutions of the cross (■) and the bar state (□) as a function of total bleaching depth.

by photobleaching using the mask aligner followed by spin coating of the upper cladding layer. A gold poling electrode was deposited by thermal evaporation. The waveguide was poled at 140℃ for 10 min with the poling field of about 2 MV/cm over the whole region including the input and output stages. Switching electrode was formed using the poling electrode by etching process. Initial bleaching depth was 0.2 μm, which is estimated by comparing the observed mode size and the calculated one. The coupler length L is 18 mm. The waveguide width and the gap width are 4 μm and 2 μm, respectively. The completed devices was cleaved for test. 1.3 μm light from a laser diode was coupled to one arm in the input side by end fire coupling and measured the output powers from the both arms of output by a vidicon camera (Electro-Physics). Selective post-photobleaching was performed while monitoring the output states using 458 nm light from Ar⁻ laser with the light intensity of 8 mW/cm².

Fig. 8 shows the experimental output power evolutions of the cross and the bar states as a function of the total bleaching depth. The calculated power evolutions are also shown with the solid line and the dotted line. The total post-photobleaching time in the experiment was about 8.2 hr and was converted to the bleaching depth by the data shown in Fig. 4 to compare the result with the calculated one. The initial output is almost in the cross state and the two successive cross states appear as the post-photobleaching proceeds. The result agrees with the calculated one very well and clearly demonstrates that the proposed post-photobleaching technique can be utilized to tune the output state and the cross-over coupling order of the device as well. The poor extinction after long post-photobleaching may be due to the slight misalignment of the switching electrodes with the channel waveguides. Since the switching electrodes were used as the mask for the post-photobleaching, some part of the channels can be exposed unsymmetrically to the post-photobleaching light as well as the gap region due to the electrode misalignment, resulting in the phase mismatch of the propagation constants. In fact, the electrode misalignment was observed by optical microscope.

CONCLUSIONS

In conclusion, we have demonstrated that a simple photochemical kinetic model considering the effects of the absorption due to the unconverted reactant and the photoproduct well describes the refractive index profiles formed by photochemical reaction. The parameters required in the model were obtained from simple transmission experiments. The experimental results are consist-

ent with the model. The refractive index profile is steeper when nonlinear optical polymeric materials are bleached by light with higher absorption. The rate of the bleach depth change becomes slower as the bleaching proceeds. Small absorption of bleaching light due to the photoproduct has significant effect on the resulting refractive index profiles.

The photobleaching process is shown to be accelerated by the addition of a photosensitizer. Photobleaching time has been effectively reduced by a factor of 3-5. Linear and nonlinear optical properties of the polymer were little affected by the addition of the photosensitizer.

We have also demonstrated that the photobleaching technique can be utilized to tune the initial state of a directional coupler switch after completing the fabrication of the device. It was shown that successive cross states are passed from the initial cross state by selectively bleaching the gap region. The evolution of the refractive index profiles is successfully applied to predict the evolution of output state.

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